Evaluation of the Free Swell and Physio-chemical Properties of Black Cotton Soil Treated with Bacillus Coagulans

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Abstract. The effect of Bacillus coagulans (B. coagulans) on the free swell and physio-chemical properties of black cotton soil (or expansive tropical black clay) was investigated using the Microbial Induced Calcite Precipitation (MICP) technique. Black cotton soil was treated with different mix ratios of B. coagulans suspension density (B) and cementation reagent (C) for the volume obtained as product of the liquid limit and a specific weight of soil sample. The mix ratios considered were 25B-75C, 50B-50C and 75B-25C for the treated specimens, while the control specimen was mixed with 100C only. The results obtained show that for treatment of soil with 25B-75C mix ratio, free swell value decreased with increased B. coagulans suspension density from 70.0 % and 50.0 % for the natural and control specimens, respectively, to minimum value of 40.0 % at B. coagulans suspension density of $2.4 \times 10^9$ cells/ml. The cation exchange capacity (CEC) value for the natural and control specimens were 49.7 and 29.6 Cmol/Kg, respectively, while for the treated specimens the values generally decreased a minimum value of 29.0 Cmol/Kg at B. coagulans suspension density of $2.4 \times 10^9$ cells/ml. The pH test results initially decreased from 7.4 for the control specimen to a minimum at of 7.37 at B. coagulans treatment suspension density of $1.5 \times 10^8$ cells/ml and thereafter gradually increased to 7.51 at peak B. coagulans suspension density of $2.4 \times 10^9$ cells/ml. Tests results indicate that treatment of black cotton soil with 25B-75C mix ratio at $2.4 \times 10^9$ cells/ml B. coagulans suspension density significantly reduced the free swell value and makes it useable for engineering application.

Keywords: Bacillus coagulans, Black cotton soil, Free swell, physio-chemical properties, cation exchange capacity, pH.

1. Introduction

Black cotton soils (BCS) are problem tropical clay deposits with high swelling and shrinkage potentials. Hence, they are generally unsuitable for use as construction materials. They are classified under the smectite group of clay minerals which are known to contain the highly expansive clay mineral, montmorillonite [1]. BCS are formed mainly in semi-arid tropics with pronounced varying dry and wet seasons [2]. They can be found, especially in the North Eastern region of Nigeria, and in other countries around the world like Australia, India, and Asia [3].

In Nigeria, BCS are formed through the disintegration of basaltic rocks and clayey and shaley sediments. The soil has excessive montmorillonite content with subsequent exhibition of expansive tendencies. In the North Eastern region of Nigeria where the soil is predominantly found, it spans an area of about 104,000 km$^2$. Major roads connecting this region with bordering West African countries such as Chad Republic, Cameroun and Niger traverse through this soil [4].

The chemical composition of BCS varies depending on factors such as parent rock, generic characteristics of soil (transported or residual), degree of weathering, etc. However, BCS are generally
rich in silica, lime, iron, magnesia and alumina. In addition, it contains titanium oxide in small concentrations, which is responsible for the characteristic black color to the soil. The organic matter contents of black cotton soils are low [5].

Generally, BCS exhibits properties that make it a candidate for improvement before use in any engineering construction. Stabilization of soil becomes necessary when the soil does not have sufficient strength to support the designed structural load. Also, stabilization can also be used to improve or regulate the shrink-swell characteristics of the soil as well as decrease compressibility and permeability of a mass of soil in earth structures [6].

The use of chemical admixtures to enhance the properties of BCS is widespread however, the method is neither eco-friendly nor economical [7]. This prompted the search for novel soil improvement methods that resulted in the use of the Microbial Induced Calcite Precipitation (MICP) technique that entails biological processes, nutrients, microorganisms that are environmentally sustainable solution to soil improvement [8-10]. This research was conducted to evaluate the effect of B. coagulans - induced calcite precipitate on the free swell and physio-chemical properties of black cotton soil using MICP technique. The specific objective was to ascertain the changes in the free swell and physio-chemical properties of BCS when treated with stepped B. coagulans suspension density.

2. Materials and methods

2.1 Materials

2.1.1 Black cotton soil. The black cotton soil samples utilised in this work was acquired along Gombe-Biu road in Yamaltu Deba Local Government Area (latitude 10° 190N and longitude 11° 300E) in Gombe state, Nigeria.

2.1.2 Microorganism. The species of bacteria used as the soil improvement agent is B. coagulans. It is classified as ATCC 7050 in the American Type Culture Collection (ATCC). It is Gram-positive rod-shaped bacteria. Five different bacteria suspension densities on the McFarland turbidity scale, MFS (i.e., 0.5, 2.0, 4.0, 6.0 and 8.0 with equivalents 1.5×10^8 cells/ml, 6 × 10^8 cells/ml, 1.2 × 10^9 cells/ml, 1.8 × 10^9 cells/ml and 2.4 × 10^9 cells/ml, respectively) were used in this study. The control soil was referenced as 0 cells/ml (0 MFS).

2.1.3 Cementation reagent. Cementation reagent was used to activate the urea hydrolysis process. The constituents of the cementation reagent used in this study comprised of 2.8 g of calcium chloride (CaCl₂), 3g nutrient broth, 10 g ammonium chloride (NH₄Cl), 20 g of urea (CO (NH₂)₂) and 2.12 g sodium bicarbonate (NaHCO₃) per litre of de-ionized water [11-14].

2.2 Methods

2.2.1 Free swell. The test was conducted according to the methods outlined in the United States Bureau of Reclamation (USBR) [15]. About 10 g of soil passing BS No.40 sieve (425 μm aperture) was oven-dried and allowed to cool in a desiccator. The sample was slowly poured into a 100 cm³ measuring cylinder and the cylinder was then filled with water. Before the free swell; the initial volume of soil in the measuring cylinder was recorded. The cylinder was then agitated in order to obtain a homogenous mixture of water and soil after which it was left for 24 hours to settle, Free swell was calculated using:

\[
F(\%) = \frac{V_1 - V_2}{V_1} \times 100
\]  

Where;  
F = Free Swell (%)  
V₁ = Initial Volume (cm³)  
V₂ = Final Volume (cm³)

This procedure was repeated for the various bacteria suspension densities.
2.2.2 Cation exchange capacity.

This test was conducted in accordance to the procedures outlined by ISRIC [16]. 10 g of soil sample passing through BS Sieve No.10 (2.00 mm aperture) was poured inside a 100 cm$^3$ beaker. 40ml of Ammonium acetate (1N pH7.0) was poured into the beaker. With the aid of a glass rod, the mixture was stirred and left for 24 hours. The soil was filtered with a light suction using a 55 mm Bucher funnel, the soil was leached so that it could fit in a funnel with Ammonium acetate of a volume of 250 cm$^3$. The filtered product was tested to ascertain the absence of calcium. White turbidity or precipitate indicated calcium presence. With 150-200 ml of isopropyl alcohol, the electrolyte was washed out. With (0.1N AgNO$_3$), chloride content was tested in the leachate until the leachate became negligible. A volume of 250ml of the leached soil was acidified, after the soil was verified to drain thoroughly. 50ml of boric acid and mixed indicator, a few drops of it, was poured into a 250 ml conical flask. The acidified soil was turned inside a 500 ml flask, with the flask still connected to the steel, anti-bump and 10 ml of 1N NaOH was poured inside the flask. The content in the flask was then distilled over the boric acid and 150ml distilled sample was collected. A standard acid of 0.1N HCl was used to titrate the NH$_4$-borate. The CEC was calculated using:

$$\text{CEC (Cmol/kg) } = \frac{(\text{Titre} - \text{B}) \times \text{NA}}{\text{Weight of soil}} \times 100$$  
(2)

Where:
B = Blank
NA = Normality of acid
This process was repeated for both the untreated and treated samples.

2.2.3 pH.

This test was conducted according to the procedures stated in BS 1377-3: 1990 [17] using electrometric method with a pH meter (PHS-25; Technel & Techmel, USA). A soil to water ratio of 1:2 (10 g of soil to 20 ml of distilled water) was adopted for this test. 10 g of air-dried soil sieved through BS Sieve No.10 (2 mm aperture) was poured into a 50 ml plastic beaker and distilled water of 20 ml volume was poured inside the beaker. The mix was stimulated repeatedly for about 30 minutes and was left undisturbed for another 30 minutes. Using pH buffer of 4, 7, and 9, the pH meter was regulated. An electrode was inserted into the soil but was not allowed to touch the bottom of the beaker and the pH read after 30 seconds. These steps were repeated using 0.01M CaCl$_2$ and 1N KCl solutions.

2.2.4 Soil sample preparation and treatment for test procedures.

400 g of Soil sample of passing through BS Sieve No.40 (425 μm aperture) was utilised for this process. For the control specimen, only cementation reagent which volume was obtained from the total volume stated in equation (3) was utilized for the mixing process of the soil sample. For the treated specimens, the soil was mixed with each B. coagulans suspension density and cementation reagent added to the soil. Three trial mixes were adopted for the calculation of the volume of each of the bacterial suspension density (1.5x10$^8$, 6 x 10$^8$, 1.2 × 10$^9$, 1.8 × 10$^9$ and 2.4 × 10$^9$ cells/ml) and cementation reagent added to the soil. The mixes are:

i. 25B-75C: In this mix, the soil was mixed with 25% bacterial suspension (25B) and 75% cementation reagent (75C) of the total volume of soil sample used which was obtained from the equations;

$$\text{Total Volume} = \frac{\text{Liquid Limit of Natural Sample}}{100} \times \text{Weight of Soil Sample}$$  
(3)
\[ \text{Volume of Bacteria} = \frac{25}{100} \times \text{Total Volume} \] (4)

\[ \text{Volume of Cementation Reagent} = \frac{75}{100} \times \text{Total Volume} \] (5)

ii. 50B-50C: In this mix, the soil was mixed with 50% bacterial suspension (50B) and 50% cementation reagent (50C) of the total volume which was obtained from the equations;

\[ \text{Volume of Bacteria} = \frac{50}{100} \times \text{Total Volume} \] (6)

\[ \text{Volume of Cementation Reagent} = \frac{50}{100} \times \text{Total Volume} \] (7)

iii. 75B-25C: In this mix, the soil was mixed with 75% bacterial suspension (75B) and 25% cementation reagent (25C) of the total volume which was obtained from the equations;

\[ \text{Volume of Bacteria} = \frac{75}{100} \times \text{Total Volume} \] (8)

\[ \text{Volume of Cementation Reagent} = \frac{25}{100} \times \text{Total Volume} \] (9)

After mixing the soil sample with the three trial mixes for the various bacterial suspensions, the treated samples were left to air-dry before being pulverized and sieved with BS Sieve No. 40 (425 μm aperture) for free swell, CEC and pH test procedures. However, for the CEC and pH test procedures, only the most suitable mix ratio obtained from the free swell test for the various mix ratios was utilized.

3. Results and discussion

3.1 Natural soil

The natural black cotton soil had a high natural moisture content of 15.1 %. Its high moisture content was probably influenced by the period in which the samples were collected. The index properties of the natural soil are shown in Table 1, while its oxide composition is presented in Table 2. The sample is greyish-black in colour and has a free swell value of 70.0 %. Its liquid limit, plastic limit and plasticity index values are 53%, 26.7% and 26.3 % respectively. It has 78.7 % fines passing BS sieve No.200. The soil was classified as A-7-6(23) soil group according to American Association of State Highway and Transportation Officials [18] as well as medium and high plasticity clay (CH) according to the Unified Soil Classification System, USCS [19].
Table 1. Basic properties of the black cotton soil used in the study.

| Properties                        | Quantity |
|-----------------------------------|----------|
| Percentage passing 0.075mm sieve  | 78.7     |
| Natural moisture content, %       | 15.1     |
| Free Swell, %                     | 70.0     |
| Specific gravity                  | 2.42     |
| Liquid limit, %                   | 53.0     |
| Plastic limit %                   | 26.7     |
| Plasticity index, %               | 26.3     |
| Linear shrinkage, %               | 11.5     |
| AASHTO classification             | A-7-6(23)|
| USCS                              | CH       |
| Maximum dry density, Mg/m³        | 1.65     |
| Optimum moisture content, %       | 17.5     |
| Colour                            | Greyish - Black |

Table 2. Oxide compositions of black cotton soil.

| Oxide                              | Concentration (%) |
|------------------------------------|-------------------|
| Sodium Oxide (Na₂O)                | 0.064             |
| Magnesium Oxide (MgO)              | 0.572             |
| Alumina (Al₂O₃)                    | 20.601            |
| Silica (SiO₂)                      | 63.208            |
| Potassium oxide (P₂O₅)             | 0.141             |
| Sulphur oxide (SO₃)                | 0.337             |
| Chloride (Cl)                      | 0.026             |
| Potassium Oxide (K₂O)              | 2.63              |
| Lime (CaO)                         | 2.017             |
| Titanium Dioxide (TiO₂)            | 1.736             |
| Chromium (III) Oxide (Cr₂O₃)       | 0.011             |
| Manganese Oxide (Mn₃O₅)            | 0.091             |
| Iron oxide (Fe₂O₃)                 | 8.522             |
| Zinc oxide (ZnO)                   | 0.009             |
| Strontium Oxide (SrO)              | 0.034             |
| Loss on Ignition (LOI)             | 0.001             |

3.2 Mechanism of microbial induced calcite precipitate (MICP) process

The concept of utilizing microbes in bio-geotechnical engineering purposes was first initiated in 1992 [20]. The concept involves the catalysis of microbes to generate deposits that can cement soil particles. This new technique, where calcite precipitate is induced is termed Microbial induced calcite precipitation (MICP).

MICP is a soil stabilization method, which involves hydrolyzing urea by using bacteria, to generate carbonate ions, that react with a solution rich in calcium (i.e., calcium chloride); to create calcium carbonate (calcite) which results in an improved soil strength and stiffness by increasing the bond between the particles of the soil [21,22]. The technique can be applied in diverse means such as permeability reduction, soil strength improvement, surface erosion control, and seismic remediation [11,8,23,24].
### 3.3 Influence of B. coagulans induced calcite precipitate on free swell and physio-chemical properties of black cotton soil

#### 3.3.1 Free swell

The variation of free swell of black cotton soil with B. coagulans suspension density is presented in Figure 1.

![Figure 1. Variation of free swell of black cotton soil with B. coagulans suspension density.](image)

Generally, free swell (FS) decreased from 70 % for the natural BCS with increase in B. coagulans suspension density for the mixes considered, while the free swell value obtained for the control specimen was 50.0 %. The lowest FS values of 30.0 %, 40.0 % and 49.0 % were recorded for 25B – 75C, 50B – 50C and 75B – 25C mix ratios, respectively, at B. coagulans suspension density of $2.4 \times 10^9$ cells/ml.

The observed decrease in FS with increase in B. coagulans suspension density can be attributed to the reduction in the clay sized particles and increase in coarser fraction because of the precipitation of calcite from the reaction between B. coagulans and cementation reagent [25,26]. The decrease in the fines fraction was probably due to flocculation and agglomeration of the particles in the treated soil samples which resulted in the formation of larger-sized particles from the clay fractions [27]. In addition, it was observed from Figure 1 that the lowest FS values were obtained for 25B-75C mix when compared with 50B-50C and 75B-25C mixes. This could probably be due to the volume of cementation reagent being insufficient to activate the desired reaction with the microbe colony in each suspension density.

The amount of cementation reagent and B. coagulans suspension density are two of the numerous factors that influence the rate of calcite precipitation [25,26].

#### 3.3.2 Cation exchange capacity (CEC)

Cation exchange capacity (CEC) can be utilized to ascertain a soil specimen’s mineral composition. A high CEC value is often indicative of a high expansiveness rate [28]. Some clay experience replacement of cation of one kind by another while retaining the same crystal structure in a process known as isomorphous substitution. This substitution, which occurs along with the dissociation of hydroxyl ions, often leads to a residual negative charge on the clay mineral’s particle surface. As a result, positively charged ions, known as cations, are adsorbed on the clay mineral’s surface. The bonds between the ions are weak and can be replaced by other ions present in water. This phenomenon is termed as cation exchange. The amount of exchangeable cation available in a soil is designated as the exchange capacity [29,30].

The variation of the CEC of black cotton soil with B. coagulans suspension density is presented in Figure 2. A general decrease of CEC with increase in B. coagulans suspension density was recorded.
The CEC value for the natural and control sample were 49.7 and 29.6 Cmol/Kg. For the treated samples, the CEC values initially increased from the value obtained for the control sample to a peak value of 31.4 Cmol/Kg at B. coagulans suspension density of $1.5 \times 10^8$ cells/ml and thereafter gradually decreased to a value of 29.0 Cmol/Kg at B. coagulans suspension density of $2.4 \times 10^9$ cells/ml. This decrease could be attributed to the changes in the soil’s granular matrix and mineralogical composition due to the flocculation of the treated soil’s particles resulting in decreased specific surface area and the clay sized fractions within the soil [31,27].

The changes in the soil’s granular matrix was due to the biogeochemical reaction between the carbonate ions from the urea hydrolysis process and the calcium ions present in the cementation reagent, which causes the formation of calcite within the soil mass [25].

3.3.3 pH.

Under proper conditions, urease positive bacteria are capable of hydrolyzing urea into carbonate ions and ammonium ions, forming an alkaline micro-environment around the microorganism cells and increasing the pH of the environment in the process. The carbonate ions in the presence of calcium ions react to produce calcium carbonate. The precipitated calcium carbonate bound soil grains together to enhance the strength and reduce the expansive characteristics of clayey soils [26,28].

The variation of pH of black cotton soil with B. coagulans suspension density is presented in Figure 3. The pH value for the control specimens initially decreased to a minimum before increasing with increase in B. coagulans suspension density.

Figure 2. Variation of cation exchange capacity of black cotton soil with B. coagulans suspension density.

Figure 3. Variation of pH of black cotton soil with B. coagulans suspension density.
The pH value for the control specimens of 7.4 decreased to a minimum value of 7.35 at 6.0 x 10^6 cells/ml and then gradually increased to a peak value of 7.51 at B. coagulans suspension density of 2.4 x 10^9 cells/ml. This trend correlates with the results obtained for the free swell values for the control and treated samples. The increase in pH was probably due to the breakdown of urea by the urease enzyme that formed ammonia as a by-product and eventually resulting in the precipitation of calcite [32]. Therefore, the gradual increase in pH is an indication that more urease activities occurred at higher bacteria suspensions leading to a larger amount of precipitated calcite at 2.4 x 10^9 cells/ml bacteria suspension density.

4. Conclusion

The black cotton soil classified as A–7–6 (23) and medium to high plasticity clay (CH) in AASHTO and USCS, respectively, was treated with stepped B. coagulans suspension density (B) of 0, 1.5 x 10^8, 6.0 x 10^8, 1.2 x 10^9, 1.8 x 10^9 and 2.4 x 10^9 cells/ml and cementation reagent (C). Three mix ratios 25B-75C, 50B-50C and 75B-25C were adopted for the study, while treatment with cementation reagent only was used as control specimen. Results indicated that the free swell improved with higher B. coagulans suspension density with the best results recorded for the 25B-75C mix ratio. Also, the pH and CEC test results indicate increase in urease activity and calcite formation with increase in B. coagulans suspension density. From the results obtained it can be concluded that B. coagulans can be effectively used to reduce the shrink-swell properties of black cotton soil in engineering construction.

5. References

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