

## **Multi-scale laboratory investigation on black cotton soils stabilized with calcium carbide residue and fly ash**

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### **ABSTRACT**

Calcium carbide residue and fly ash are industrial by-products from acetylene gas industry and thermal power stations. Pavement construction has been recognized as a platform to utilize huge quantities of these waste materials for soil stabilization. An attempt has been made, in this paper, to evaluate the potential of a binder that consists of calcium carbide residue and fly ash to improve the engineering behavior of an expansive soil. The role of curing period and different percentages of these binder contents on Atterberg limits, compaction characteristics, strength, and mineralogical and morphological behavior has been examined. The index properties such as Atterberg limits and compaction characteristics have improved considerably. Significant improvement in the strength properties such as unconfined compressive strength and California bearing ratio has been observed with the addition of calcium carbide residue up to 8%. The binder content prepared with 10% calcium carbide residue and 6% fly ash obtained 18-fold enhancement in California Bearing Ratio (CBR) value and 16-fold enhancement in unconfined compressive strength (UCS). This is attributed to the formation of calcium based minerals formed as a result of a chemical reaction between the soil and binder, which were evidenced from mineralogical and morphological studies. From this experimental investigation, it can be concluded that the binder prepared with calcium carbide residue and fly ash is well suited for stabilization of expansive soils.

**Keywords:** Calcium carbide residue, CBR, Fly ash, SEM, XRD.

### **INTRODUCTION**

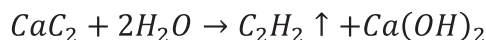
Expansive soils pose challenges to civil engineers worldwide due to their exhibition of high swelling and shrinking behavior. Black cotton soils of India are well known for their expansive behavior. In India, black cotton soil covers around 0.7 million square kilometers (i.e., 2025- % land area approximately). They are highly fertile for agricultural purposes, but pose severe problems to pavements, embankments, and light to medium loaded residential buildings due to cyclic volumetric changes caused by moisture fluctuation.

The addition of chemical stabilizers is a widely used technique to improve the engineering properties of black cotton soil. It includes traditional stabilizers like cement, lime, and fly ash. Effect of stabilization depends upon the calcium exchange and pozzolanic reactions between soil and stabilizers.

Cement stabilization is one of the extensively used techniques to rectify the deficiencies in engineering properties of expansive soils, especially for pavement applications. An advantage of

cement stabilization is that the required strength can be attained in a shorter period. The effect of moisture content, replacement ratio, compaction effort, curing period, and cement content on the engineering characteristics and microstructure of cement aided soils is widely being researched (Tatsuoka & Kobayashi, 1983; Nagaraj *et al.*, 1997; Horpibulsuk & Miura, 2001; Horpibulsuk *et al.*, 2010b; Horpibulsuk *et al.*, 2011b; Deng *et al.*, 2012). In order to restrict the stabilization cost, replacement of cement by the industrial by-products like fly ash, pond ash, and granulated blast furnace slag has been widely adopted in practice. Many researchers studied the behavior of fly ash stabilized soils (Sharma & Reddy, 2004; Edil *et al.*, 2006), and it was noticed that the addition of fly ash leads to increasing reactive surface of soil-cement clusters for the pozzolanic reactions (Horpibulsuk *et al.*, 2009). The curing period also plays a vital role in enhancing the strength of black cotton soil stabilized with lime and fly ash (Zha *et al.*, 2008). A micro-level investigation of clayey soil stabilized with fly ash and lime reported 20% FA and 8.5% lime as the optimum percentages (Sharma *et al.*, 2012).

One of the by-products from the acetylene gas industries is calcium carbide residue (CCR), and it is formed due to the reaction between calcium carbide and water. In developing countries like India, there are many acetylene gas production units and PVC chemical plants, which produce CCR in huge amounts (Sharma & Reddy, 2004; Phetchuay *et al.*, 2014). It is usually dumped in the landfills and causes environmental problems to landfills due to its alkalinity. Formation of CCR can be demonstrated by the following chemical equation:



Jiang *et al.* (2015) performed multi-scale investigation on clayey soils stabilized with CCR and lime and reported the optimum dosages of lime and CCR as 6% and 8 %, respectively, for silty clay stabilization. However, CCR stabilization was found to be more effective than lime due to its superior mechanical performance. The existence of ettringite and non-crystalline phase calcium silicate hydrate (C-S-H) after 7 days of curing was identified when the effect of CCR and biomass ash binder on soft Bangkok clay was investigated (Vichan & Rachan, 2013).

It is reported in the literature that stabilization of silty sand with CCR and FA resulted in improved stiffness and strength of the soil (Consoli *et al.*, 2001). The presence of fly ash (FA) further improved the material behavior due to the occurrence of time-dependent pozzolanic reactions. Addition of CCR is found to be effective for a highway embankment filling material and resulted in an increase in UCS, CBR, and water soaking durability (Duet *et al.*, 2011). Studies regarding the effect of CCR and FA on the behavior of expansive soils are less reported in the literature. In the present study multiscale laboratory analyses are carried out on black cotton soil stabilized with CCR and FA. A series of tests were carried out to determine Atterberg limits, California Bearing Ratio (CBR), and unconfined compressive strength (UCS) of black cotton soils stabilized with CCR and FA. Mineralogical and micro structural studies were conducted by using XRD and SEM analysis in order to understand the stabilization mechanism.

## EXPERIMENTAL PROGRAMME

### 2.1 Materials used

The black cotton (BC) soil was collected from a construction site in Warangal, India. The soil sample was dried and stored in containers, and the required amount was pulverized and used for different experiments. Fly ash was procured from NTPC thermal power plant at Ramagundam where, as CCR, it was obtained from a local welding industry at Warangal.

Table 1 shows the index properties of BC soil and FA obtained from laboratory experiments carried out as per IS 2720 part 5. Table 2 shows the chemical composition of BC soil, FA, and CCR obtained using X-ray diffraction (XRD) analysis. As per IS classification system, the BC soil can be classified as High Plasticity Clay (CH). Fly ash can be classified as class C or class F as per ASTM C618. Class C was assigned to ashes that generally have a high amount of calcium oxide (CaO). The fly ash used was class F fly ash as it contains low amount of calcium oxide content.

**Table 1.** Physical and engineering properties of BC soil and FA.

S.NO	Properties	BC Soil	FA
1	Specific gravity	2.65	2.32
2	Grain size analysis		
	Gravel (%)	4	0
	Sand (%)	29	6
	Silt (%)	40	94
	Clay (%)	27	
3	Liquid Limit (LL)	59	Non Plastic
4	Plastic Limit (PL)	18	Non Plastic
5	Plasticity Index (PI)	41	Non Plastic
6	IS Soil classification	CH	CH

**Table 2.** Elemental chemical composition of BC soil and FA.

Constituent	BC	FA	CCR
Silica (SiO <sub>2</sub> )	79.93%	63.0%	40.7%
Alumina (Al <sub>2</sub> O <sub>3</sub> )	10.59%	28.3%	10.17%
Ferrous (Fe <sub>2</sub> O <sub>3</sub> )	5.07%	3.8%	8.04%
Calcium (CaO)	1.05%	2.1%	40%
Titanium (TiO <sub>2</sub> )	0.54%	0.4%	0.65%
Magnesia (MgO)	2.11%	1.4%	0.44%
Sodium (Na)	0.6%	-	-
Potassium (K)	1.11%	-	-

## **Methodology**

The experimental program can be divided into two phases. In the first phase, the effect of CCR on BC soil was studied. Variation in Atterberg limits, UCS values, and CBR of BC soil when mixed with different percentages (by dry weight) of CCR was studied. An optimum percentage of CCR was obtained based on the results obtained from the first phase. In the second phase, improvement in the properties of CCR stabilized BC soil with the further addition of FA was studied. In this phase optimum percentage of CCR (from the first phase) was added to the BC soil, and the variation of its properties with different FA percentages was studied.

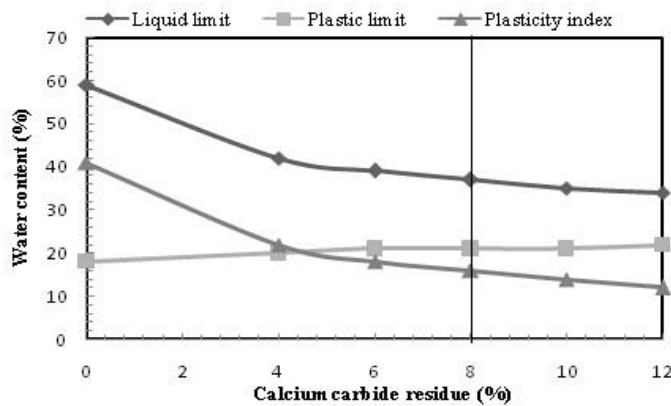
The procured BC soil was dried and pulverized before using for the laboratory experiments. All the experiments were carried out using soil fraction finer than 4.75mm. For finding the optimum moisture content and maximum dry density for the binder-amended soil, the standard Proctor test was conducted. These tests were conducted as per IS: 2720 Part 7 (1980) on various mixes, which are prepared on the basis of dry weight. The mould with standard volume of 1000cc is used, and the material was compacted by 25 blows in three layers. Standard hammer of 2.45kg weight falling from a height of 30 cm is used for compaction, and the test is repeated with an increase in water content. Dry density is calculated at all water contents so as to obtain the compaction curve. The water content at maximum dry density was considered as optimum moisture content. Experiments to determine the Atterberg limits were carried out on air dried soil passing through a 0.425 mm sieve. The soil sample was mixed with the required amount of CCR and FA and Atterberg limits were determined as per IS: 2720 Part 5 (1985). Soil was mixed thoroughly with the required amount of CCR and FA and wrapped in vinyl bags for curing at 250 C. UCS tests and CBR tests were carried out on these cured samples after 7, 14, and 28 days. UCS tests were performed as per IS 2720 Part 10 (1991). Specimens used in this study were having a diameter of 38mm and height of 76mm and were prepared at maximum dry density and optimum moisture content. CBR tests were carried out as per IS: 2720 Part 16. A surcharge of weight 25N was used throughout the testing. The rate of loading 1.25mm/min was applied to the specimen up to the failure of the specimen. A metal plunger of 50 mm in diameter and 100 mm in height is used.

X-ray diffraction (XRD) analysis and scanning electron microscopy (SEM) of the mixture of BC soil and CCR and FA were carried out to study the mineralogical and micro structural alterations occurring in the soil due to the stabilization with additives. The results were compared with the PCPDF database. X-ray diffraction technique was carried out with the help of PAN analytical X-ray diffractometer with Cu-K $\alpha$  radiation in steps of 0.02 at a rate of 1° (2 $\theta$ ) per minute, swept from 6° to 70° (2 $\theta$ ), according to the diffraction powder method. The XRD results were analyzed with an X-Pert high score plus software based on database provided by Joint Committee of Powder Diffraction Data Service (PCPDFWIN, 1999). Morphological studies on soil samples were carried out using TESCAN VEGA 3LMU microscope with conventional tungsten heated cathode having live stereoscopic imaging using 3D beam technology. The samples were coated with gold using a sputter coater prior to scanning.

## RESULTS

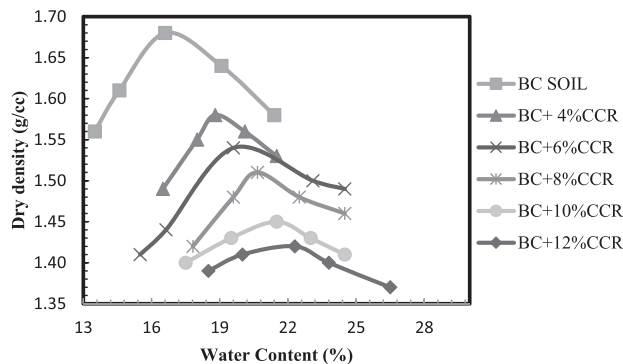
### Behavior of CCR-stabilized black cotton soil

Fig. 1 presents the variation of Atterberg limits with CCR. It can be seen that liquid limit (LL) and plasticity index (PI) increase, whereas plastic limit (PL) decreases. Addition of CCR to BC soil resulted in an increase in the coarser particle content and flocculation of clay particle due to the inclusion of  $\text{Ca}^{2+}$  from the cation exchange (Horpibulsuk *et al.*, 2011). These reasons caused a reduction in LL and PI and an increase in PL. It can be observed that, with the addition of more than 8% CCR, there is only nominal reduction in PI. It shows that, at 8% CCR, BC soil can absorb max amount of  $\text{Ca}^{2+}$  ions. This point is known as CCR fixation point.



**Figure 1.** Variations of LL, PL, and PI with the addition of CCR to BC soil.

The standard Proctor tests were conducted by adding different percentages of CCR to the BC soil. BC soil is mixed with CCR by varying its percentages from 4% to 12% in increments of 2%. From figure 2, it can be observed that there is an increase in the optimum moisture content and a decrease in the maximum dry density when CCR is added to BC Soil. The reduction in maximum dry density of soil with the addition of CCR is mainly due to its lesser specific gravity, and an increase in optimum moisture content can be attributed to flocculation soil particles (Horpibulsuk *et al.*, 2013).



**Figure 2.** Showing changes in OMC and MDD with the addition of CCR to BC soil.

Samples for UCS tests were prepared at the maximum dry density and optimum moisture content obtained from the compaction tests. The variation of UCS values for different CCR content corresponding to 7-, 14-, and 28-day period of curing is obtained and is shown in Figure 3. It can be observed that, with the addition of CCR to the BC soil, it is able to sustain higher stresses than the virgin BC soil. With an increase in the curing time, an increase in the UCS value is observed for the stabilized BC soil. The increase in UCS values of CCR stabilized soil is credited to the pozzolanic reaction between silica and alumina of BC soil and lime of CCR to form various types of cementing agents (Kinuthia *et al.*, 1999). The reaction products can be evidenced from XRD results. The UCS of CCR stabilized soil is found to increase up to 8% CCR content and decrease with a further increase in the CCR content. The reason for this behaviour is due to the fact that complete consumption of  $\text{Ca(OH)}_2$  by the natural pozzolonic material in the soil happens at 8% CCR. More CCR content leads to precipitation of free lime, which leads to a reduction in strength. It can be observed that the maximum UCS is coinciding with the CCR fixation point obtained from the Atterberg limits.

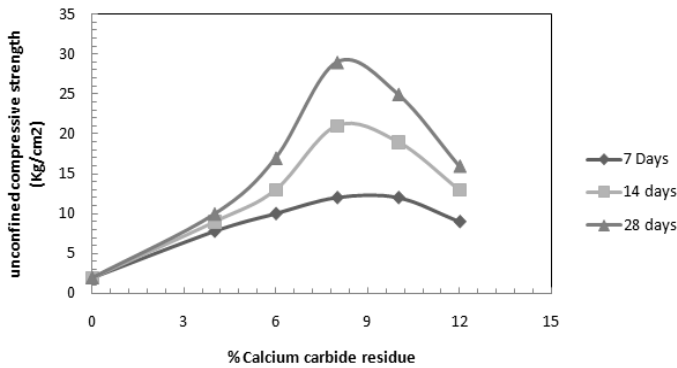


Figure 3. Variation in UCS of BC soil treated with CCR for curing periods of 7, 14, and 28 days.

CBR tests were performed for BC soil stabilized with varying percentages of CCR. All the samples were compacted at maximum dry density and with water content equal to OMC. CBR values obtained from different CCR contents are shown in Figure 4. From the figure, it can be noticed that the CBR value of soil-CCR mix increased significantly with an increase in the CCR content. Similar to the case of UCS results, a reduction in the CBR values was observed after the optimum CCR content of 8%, the reason for which can be attributed to the presence of free lime.

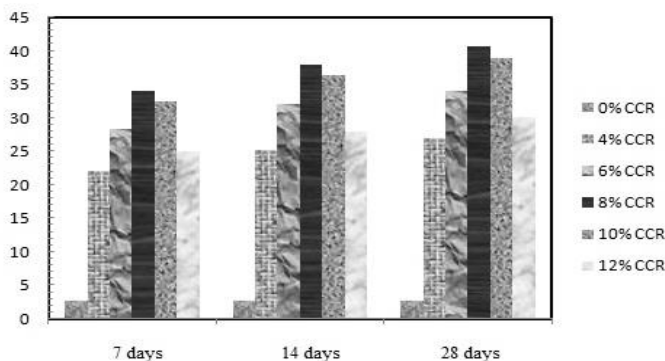
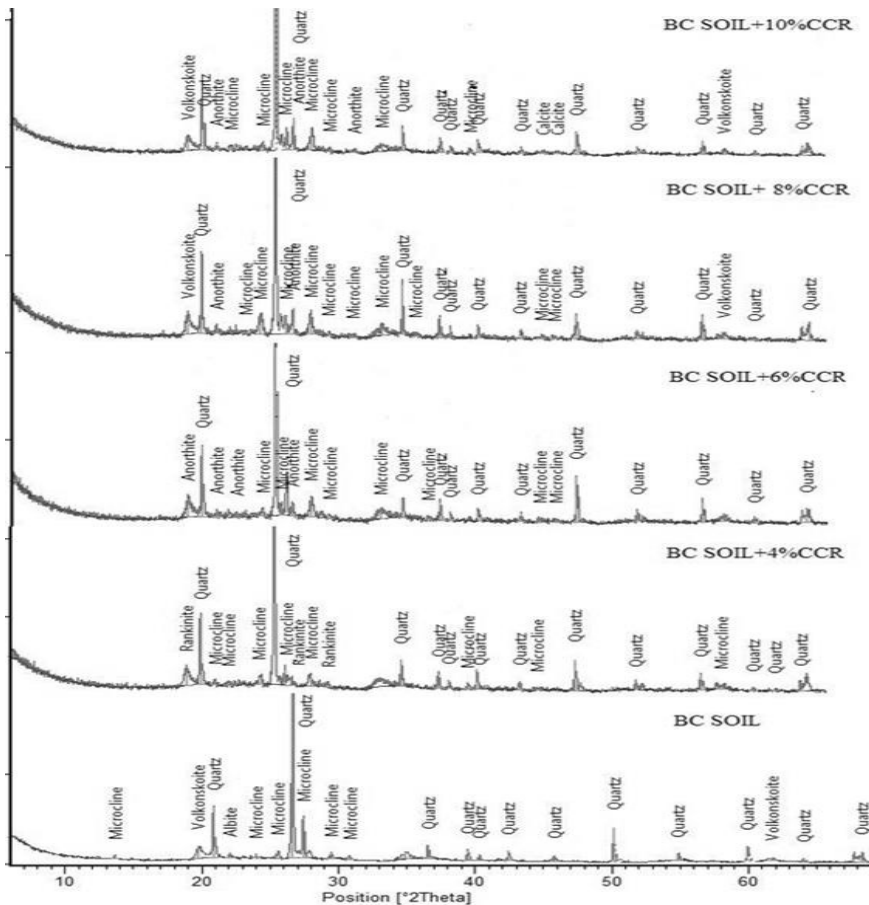


Figure 4. CBR of BC soil treated with CCR for curing periods of 7 days, 14 days, and 28 days.

PAN analytical X-pert powder diffractometer is used to study the mineralogical behavior of soil samples. Specimens were scanned from  $6^\circ$  ( $2\theta$ ) and  $70^\circ$  ( $2\theta$ ) angles using copper K-alpha radiation at a scanning rate of  $2^\circ/\text{min}$ . X-pert high score plus software is used to find out minerals presented in the soil using the obtained data from XRD test. XRD pattern of soil with respect to water shows that the natural BC soil contains volkonskoite (peaks at 4.49, 2.56, and  $1.50 \text{ \AA}$ ) along with Quartz (peaks at 3.34, 2.45, 1.81, and  $1.67 \text{ \AA}$ ) and microcline (peaks at 3.24, 2.90, and  $1.98 \text{ \AA}$ ) as their major minerals. The BC soil treated with 4% CCR showed peaks pertaining to rankinite (peaks at 3.20, 3.03, and  $2.13 \text{ \AA}$ ), a new mineral, which is a calcium silicate mineral. BC soils treated with 6% CCR and 8% CCR showed peaks pertaining to anorthite (peaks at 3.47, 3.18, and  $2.90 \text{ \AA}$ ), which is a calcium aluminum silicate mineral, and the soil treated with 10% CCR showed peaks to calcite (calcium carbonate) at  $3.03, 2.28, \text{ and } 1.87 \text{ \AA}$  in addition to anorthite. The pozzolanic reactions between the silica and alumina of BC soil and the lime of CCR are clearly evident from the X-ray diffraction results confirming the reason for increase in UCS and CBR values with the addition of CCR.



**Figure 5.** X-ray diffraction patterns of BC soil and BC soil treated with different percentages of CCR.

Scanning electron microscope images of the BC soil and soil treated with CCR are as shown in Figure 6. The soil-CCR mix after curing for 14 days was scanned using electron microscope under



different magnifications. The scanning electron micrographs, which show the microstructure of BC clay mixed with CCR, are given in Figure 6.

Natural BC soil exhibits a film type of microstructure. It can be seen from Figure 6(a-b) that the stabilized soil exhibits flocculated structure as compared to that of natural soil. The precipitation of binders around the particles can also be clearly evidenced from the SEM micrographs of stabilized soils.

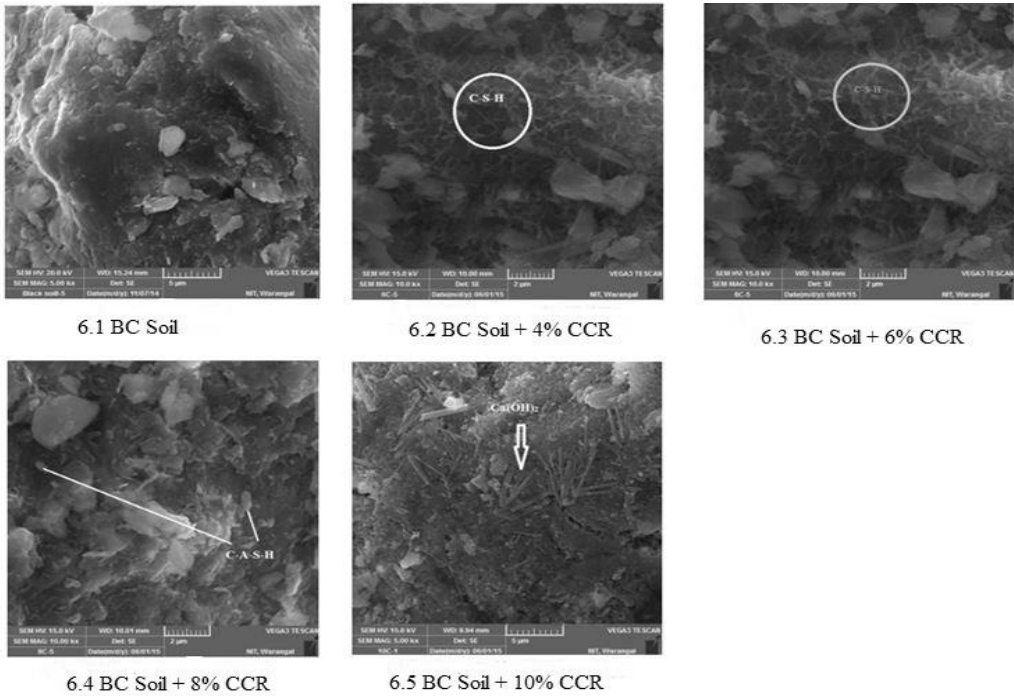


Figure 6. SEM images of BC soil and BC soil stabilized with CCR.

The optimum content of CCR obtained from experimental results is 8%, and the addition of optimum CCR is found to improve the BC soil, which is poor according to AASHTO classification of sub-grade soil to excellent.

### Behaviour of BC soil stabilized with CCR and FA

The optimum content of CCR obtained from experimental results in phase 1 is 8%. For stabilization using both CCR and FA, the percentage of CCR was fixed at 10% so that the free lime from CCR will enable pozzolanic reactions with silica and alumina of FA. Table 3 shows a variation of LL, PL, and PI with the addition of fly ash to the CCR stabilized black cotton soil. LL and PI are decreasing, while the PL is increasing, but to a lesser extent.



**Table 3.** Variations of LL, PL, and PI with the addition of 10% CCR and different percentages of FA to BC soil.

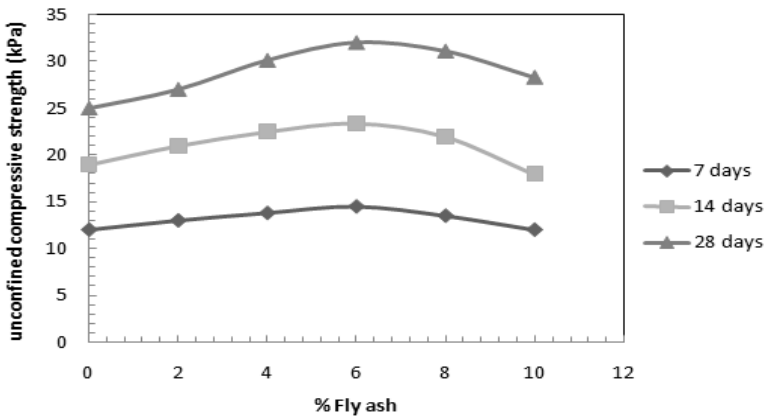
Composition	LL	PL	PI
BC+10 %CCR+2%FA	32	21	11
BC+10 %CCR+4%FA	31	21	10
BC+10 %CCR+6%FA	30	21	9
BC+10 %CCR+8%FA	30	22	8
BC+10 %CCR+10%FA	30	22	8

Table 4 represents the compaction characteristics of CCR+FA stabilized soil. The maximum dry density of CCR+FA blended BC soil showed slightly higher values than blended CCR-stabilized BC soil in the initial FA increments up to 6%. It is because CCR has more specific gravity compared to FA leading to an increase in specific gravity of CCR+FA binder. Due to the spherical nature of FA, soil and CCR particles slip over each other and into a densely packed state (Horpibulsuk *et al.*, 2011). Similar compaction pattern as in the case of addition of CCR alone is observed in the case of CCR+FA stabilized BC soils also, due to lower specific gravity and the spherical nature of fly ash particles. The increase in optimum moisture content is due to the fineness or more surface area of fly ash particles compared to the BC soil.

**Table 4.** Variation of optimum moisture content and maximum dry density with the addition of 10% CCR and different percentages of FA to black cotton soil.

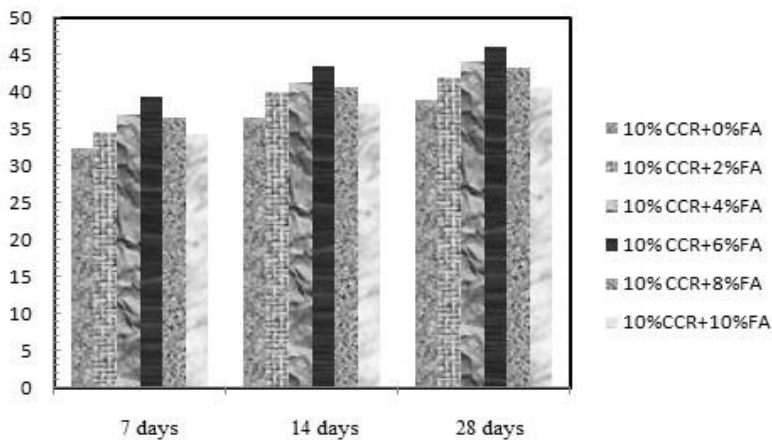
Composition	Optimum moisture content (%)	Maximum dry density (g/cc)
BC+10 %CCR+2%FA	22.28	1.5
BC+10 %CCR+4%FA	22.67	1.49
BC+10 %CCR+6%FA	23.44	1.47
BC+10 %CCR+8%FA	24.2	1.44
BC+10 %CCR+10%FA	24.5	1.4

Further, the UCS tests were carried out on 10%CCR+FA stabilized BC soil. It is observed from Figure 7 that the strength increases as the FA content increases up to 6% and then decreases. The maximum UCS obtained for 10% CCR is about 25kg/cm<sup>2</sup> while the BC soil stabilized with 10% CCR and 6% FA blender is 32 Kg/cm<sup>2</sup>.



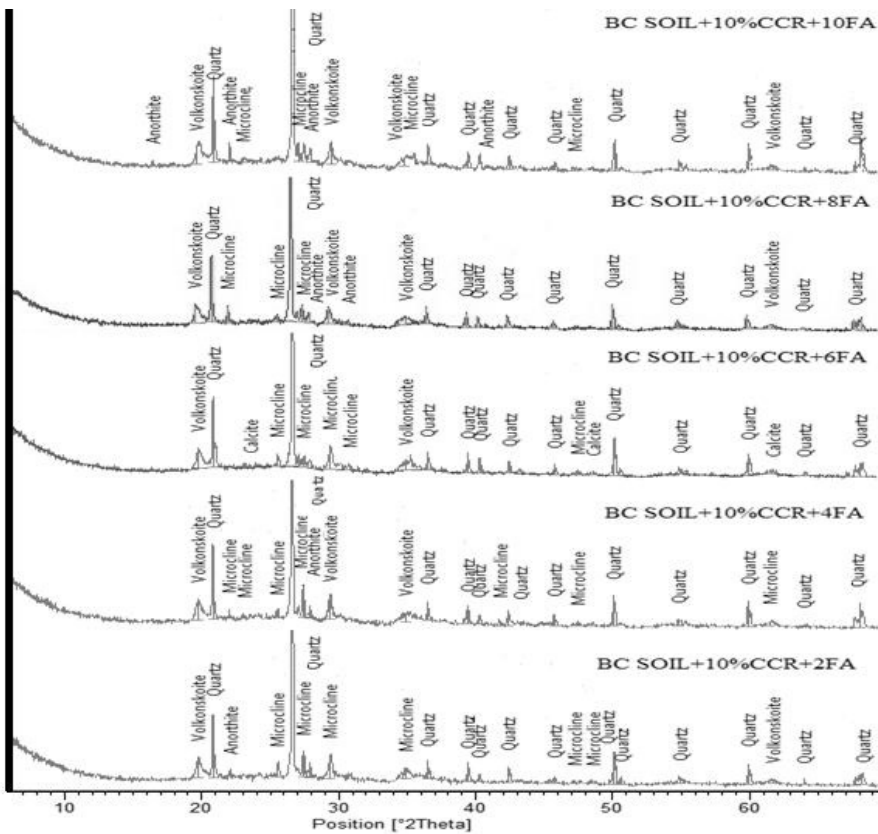
**Figure 7.** Variation in UCS of BC soil treated with 10%CCR+ FA for curing periods of 7,14 and 28 days.

CBR tests were carried out on BC soil stabilized with 10% CCR at different percentages of FA, and the results are shown in Figure 8. From Figure 8, it can be observed that CBR value is increasing up to 6% FA content and thereafter, it is decreasing. The improved CBR may be attributed to the presence of the free lime content in CCR, which will react with silica and alumina of fly ash to cause pozzolanic reactions. With further increase in fly ash, CBR is decreasing because there is no free lime content to react with fly ash.



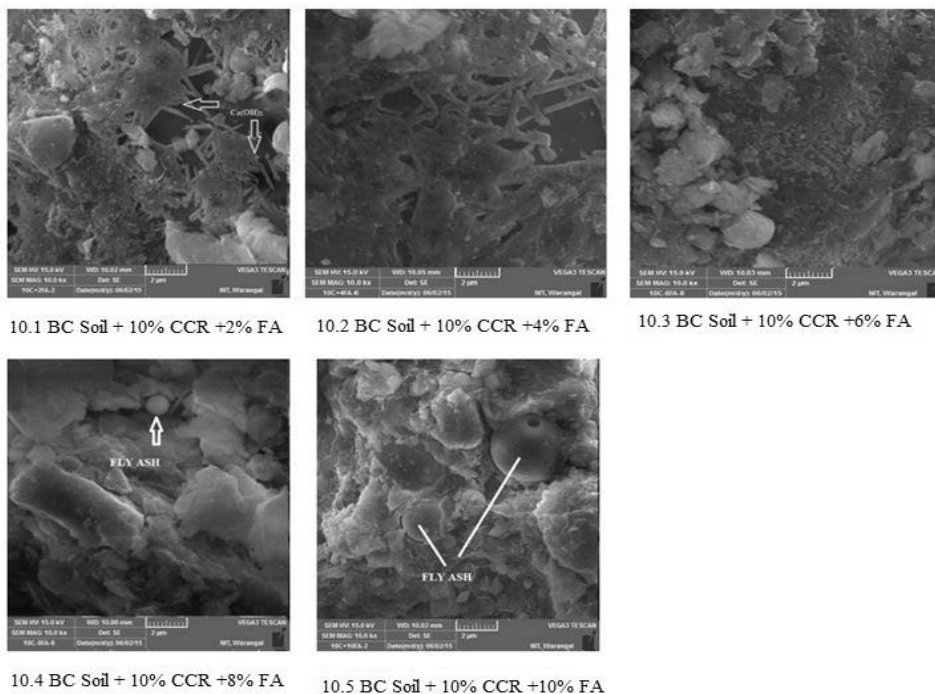
**Figure 8.** CBR of BC soil treated with 10% CCR+ FA for curing periods 7, 14, and 28 days.

It can be observed from the X-ray diffraction patterns given in Figure 9 that the BC soil treated with 10% CCR and FA samples showed anorthite (peaks at 3.47, 3.18, and 2.90 [Å]) in addition to minerals present in the natural soil.



**Figure 9.** X-ray diffraction patterns of BC soil treated with 10% CCR and FA of different percentages.

Scanning electron microscope images of the BC soil and soil treated with CCR+FA are shown in Figure 10. The SEM images of soil treated with CCR+FA mix showed significant changes in morphology. At the lower fly ash contents the formation of needle shaped particles indicates the unreacted CCR content, which reacts with the FA content. From SEM images of soil treated with 10% CCR+6% FA, the absence of needle-like structure can be observed, which confirms the complete utilization of CCR and FA. Thereafter, the presence of spherical shaped particles highlights the excess amount of fly ash, which leads to a decrease in strength. It can be observed from the experimental results that, by the addition of 10% CCR and 6% FA to BC soil, it can be improved from a poor sub-grade material to excellent material according to the AASHTO classification of sub-grade.



**Figure 10.** SEM images of and BC soil stabilized with CCR+FA blender.

## CONCLUSIONS

This paper deals with physical, mechanical, mineralogical, and morphological studies on CCR and CCR+FA stabilized BC soil, and the following conclusions can be drawn.

- 1) The index properties of BC soil such as atterberg limits and compaction characteristics showed drastic changes with the addition of CCR and CCR+FA. The reduction in plasticity of soil is evident from these tests.
- 2) Utilization of calcium carbide residue for stabilization of black cotton soil resulted in significant improvement in the strength properties, which is observed from the unconfined compressive strength and California bearing ratio test results. Stabilization with calcium carbide residue enhanced the unconfined compressive strength and California bearing ratio by 1014.5- times when compared to the natural soil. The BC soil stabilized with both calcium carbide residue and fly ash obtained 13.5–18-fold strength enhancement in CBR tests compared to the natural soil.
- 3) The formation of cementing agents such as calcium silicate hydrates and calcium aluminum silicates liable for improved strength was evidenced from mineralogical studies using XRD. The formation of pozzolanic compounds and flocculation is evident from the SEM images also.
- 4) The above results affirm that industrial waste products like calcium carbide residue and fly ash can be used for stabilizing expansive clays for different geotechnical applications like pavements and foundations for light weight structures.

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*Submitted:* 23/7/2016

*Revised* : 2/11/2016

*Accepted* : 11/3/2016



## التحليل المختبرية متعددة الأغراض على رواسب القطن السوداء المثبتة بمخلفات كربيد الكالسيوم والرماد المتطاير

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### الخلاصة

تعتبر مخلفات كربيد الكالسيوم والرماد المتطاير منتجات ثانوية ناتجة عن صناعة غاز الأسيتيلين ومحطات الطاقة الحرارية. تم الاعتراف برصف الطرق كمنصة للاستفادة من الكميات الضخمة من تلك المخلفات لتثبيت التربة. في هذا البحث، تم إجراء محاولة لتقييم قدرة الرابط الأسفلتي الذي يتكون من مخلفات كربيد الكالسيوم والرماد المتطاير لتحسين المعالجة الهندسية للتربة التمددية. وتمت دراسة دور فترة إنضاج الأسمنت بالترطيب ونسب مختلفة من محتويات الرابط الأسمنتي على حدود اتربرج (Atterberg)، وكذلك خصائص الضغط، والقوة، والمعالجة المعدنية والمورفولوجية. لقد تحسنت الخصائص الدليلية مثل حدود اتربرج وخصائص الضغط بشكل ملحوظ. وقد لوحظ تحسن كبير في خصائص القوة مثل مقاومة الانضغاط اللامحصورة ونسبة تحميل كاليفورنيا عند إضافة مخلفات كربيد الكالسيوم بنسبة تصل إلى 8%. وحصل محتوى الرابط الأسفلتي المُجهز بنسبة 10% من مخلفات كربيد الكالسيوم و6% من الرماد المتطاير على تعزيز بنسبة 18 ضعف من قيمة نسبة تحميل كاليفورنيا (CBR) و16 ضعف في مقاومة الانضغاط اللامحصورة (UCS). ويعزى ذلك إلى تكون المعادن المرتكزة على الكالسيوم والتي تشكلت نتيجة لحدوث تفاعل كيميائي بين التربة والرابط الأسفلتي الذي اتضح من الدراسات المعدنية والمورفولوجية. ومن هذا البحث التجريبي، يمكن استنتاج أن الرابط الأسفلتي المُجهز بمخلفات كربيد الكالسيوم والرماد المتطاير مناسب تماماً للعمل على استقرار التربة التمددية.