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Preliminary Laboratory Evaluation of By-Product Gypsum as Pavement Base Material

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ABSTRACT

This paper presents the results from a preliminary laboratory evaluation on recycled blended calcium sulfate (BCS) material conducted at Louisiana Transportation Research Center. The results indicate that BCS material will obtain various unconfined compressive strengths depending on the initial moisture content and relative humidity of curing conditions. The bonds between gypsum crystal particles are the interfacial surface energy and cementing agent precipitated from the solution around the particles. Since both bonds are highly sensitive to moisture conditions, the presence and absence of moisture content in BCS material would decrease or increase its strength.

The addition of cementitious materials such as cement, fly ash, and ground granulated blast-furnace slag (GGBS) can increase the strength and water resistance of the BCS specimens. The extent of this improvement is various for different cementitious materials. This improvement can be attributed to pozzolanic reaction and cementitious hydration. The newly formed hydrates would whole or partially cover the gypsum crystal particles and bond them together. Of cementitious materials used in this study, GGBS is the most effective agent to stabilize BCS. However, the long-term volumetric stability of stabilized BCS is not fully understood at this time. More systematic laboratory and field tests should be conducted for the optimum use of the BCS material in highway construction.

KEY WORDS: Blended calcium sulfate, Gypsum, Pavement base course, Stabilized gypsum, X-ray diffractometry (XRD), Scanning electron microscopy (SEM)

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INTRODUCTION

Currently, over 150 million tons of the waste byproduct fluorogypsum (FG) is stockpiled in the United States with about 20 millions tons being produced annually. FG is a byproduct of the production of hydrogen fluoride (HF). In the manufacturing process, fluorospar (calcium fluoride) reacts with condensed sulfuric acid (H_2SO_4) to form HF gas and calcium sulfate or gypsum ($CaSO_4 \cdot 2H_2O$), i.e., fluorogypsum. One possible application for the use of FG is in highway construction.

As a low-cost recycled material, the Louisiana Department of Transportation and Development (LA DOTD) has been using this waste material as the base course material in pavements over the last 15 years. The fluorogypsum is blended with lime or limestone to meet pH requirement and is commonly referred to as Blended Calcium Sulfate (BCS). While the use of this material has performed satisfactorily on many projects, moisture sensitivity of the material has been a concern for engineers in LA DOTD.

The Louisiana Transportation Research Center (LTRC) has been asked to evaluate the continued use of BCS as a base course. A select number of projects previously constructed with BCS base course were evaluated with the Falling Weight Deflectometer (FWD), Dynaflect, and the Dynamic Cone Penetrometer (DCP). Table 1 presents the results of this evaluation. A single cement stabilized base course on LA 16 was also evaluated for comparison. The average values reported in Table 1 for BCS corresponded well with the modulus values of 3,260 MPa (473 ksi) from FWD and 1.4 mm/blow from the DCP for the soil cement base course. It should be noted that a single site reading taken on Jefferson Highway over a broken joint resulted in a DCP reading of 50 mm/blow. This measurement could be an indication of water infiltration.

In 2002, LTRC monitored two BCS base course construction projects: Relocated LA 1 in Lafourche Parish and Louisiana and Evangeline Thruway in Lafayette Parish. The Relocated LA 1 base course was constructed on a free draining sand embankment. The Evangeline Thruway base course was constructed in a depressed location that did not have positive drainage. The material was subjected to severe ponding of surface rainwater. Figure 1 presents results of DCP tests conducted at six locations taken periodically after compaction of the base course. After initially gaining strength with time and curing, half of the test locations began to dramatically lose strength. This loss of strength was attributed to water infiltration. The remainder of the project was randomly tested with DCP and FWD. The results averaged 7.8 mm/blow for the DCP test with a corresponding modulus of 407 MPa (59 ksi) from FWD tests. These values are substantially less than previous results obtained from older projects. The average values for the Relocated LA 1 project were 3.1 mm/blow and 86 ksi for the DCP and FWD respectively. While these values are better than the Lafayette project, they too are lower than the previous readings taken on existing projects.

In a dry condition, BCS bases are very hard and can have high strength and stiffness, but problems occur when they absorb moisture. In a wet environment, BCS can have trouble gaining the necessary strength to maintain traffic or reach the required design value. The base course of a pavement is designed to and must work in a dry and wet cycle environment

with a minimum strength requirement. These issues have questioned the strength and stability of BCS base courses as a viable alternate. The purpose of this test program was to establish the foundation for a full-scale research program needed to determine its proper use as highway construction material. This paper describes the preliminary findings from this test program.

ACADEMIC BACKGROUND

The poor performance of gypsum materials concerning resistance to water has been noted earlier (2), and it is expected that they may lose up to half their dry strength when they absorb moisture (3). However, the underlying mechanism has not been fully understood. Laboratory experiments have shown (4) that at 40°C under dry conditions gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) slowly dehydrates to hemihydrate ($\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$) or anhydrite (CaSO_4). Under damp conditions decomposition is slower, and hemihydrate previously formed may rehydrate to gypsum. When conditions favor alternative dehydration and rehydration, weakening and powdering is likely to ensue. This may account for the failure of gypsum panels after alternating wet and dry cycles.

Research work (5) suggested both gypsum and hemihydrate are soluble, with the solubility of 2.6 g/l and 8.7 g/l, respectively. There may be some direct relationship between hardness and solubility. The hypothesis was developed that partial dissolution of the material near the contact points of the crystals is the origin of poorer mechanical properties, or conversely, that drying the samples restores the bonds between the crystals by re-precipitation of solute at the interface. Scanning electron microscopy (SEM) observation (6) indicated that when soaking in water, the gypsum crystal needles evolve towards their equilibrium form. During this slow process, they shorten and thicken leading to the breakdown of the solid. A recent theory (7) is based the surface energy theory. Bonds between the crystals in contact result in the mechanical strength of gypsum. Water molecules infiltrating between the crystals to shield the bonds may drastically weaken these bonds, which leads to an important decrease of the mechanical resistance of gypsum. Conversely, gypsum material recovers its mechanical strength as soon as water leaves the interfaces, allowing the bonds to set up again in the dry solid.

Even though the mechanism of strength loss is an unsettled issue, some research works have been conducted to improve gypsum's water resistance and strength in wet conditions. Clay minerals (8) and fly ash (9) have been added and mixed with gypsum to improve the water resistance. It is believed that these materials may fill in the voids between gypsum crystals and decrease the permeability of the material, or that these materials may produce hydrated products to coat the gypsum crystals and maintain its integrity under wet conditions.

BASIC PROPERTIES

BCS material, with the composition shown in Table 2, was tested for its basic mechanical properties. The particle size distribution of the BCS was determined using sieve analysis and hydrometer analysis according to ASTM D 422. Figure 2 shows the gradation of the BCS. It indicates a well-graded distribution with the uniformity coefficient $C_u = 34.78$ and the coefficient of gradation $C_c = 1.76$. Approximately 4 percent of its total weight consists of particles less than 0.05-mm.

The standard Proctor compaction (ASTM D 2166) was used to determine the moisture-density relationship of BCS. The BCS material that passed the number 4 Sieve (4.75 mm) was used for this test as required by the standard procedure. Figure 3 shows its moisture-density curve that indicates a maximum dry density of 17.1 kN/m^3 (109.0 lb/ft^3) with the optimum moisture content of 9.8 percent.

UNCONFINED COMPRESSIVE STRENGTH

Unconfined Compressive Strength (UCS) tests were conducted on BCS specimens with the United Compression Model SFM-30E load frame. The procedure for specimen preparation was similar to that used for the compaction tests. To better understand the correlation between the strength of a BCS specimen and its dry density and moisture content, the specimens were compacted at various initial moisture contents and/or cured in different relative humidity (RH). Specimen cylinder numbers with different moisture content and curing condition preparation are listed in Table 3.

The first group of specimens with cylinders numbered I-1 through I-5 shown in Table 3 for UCS tests was cured at a RH of 50 percent for 28 days. The results of UCS tests in Figure 4-a indicate that the UCSs of BCS material are strongly moisture-dependent as expected, but the maximum strength, 1.4 MPa (203 psi), was achieved at a moisture content of about 7 percent, not the optimum of 9.8 percent for compaction. This is same with regular soils. The explanation for this phenomenon could be as follows: the strength of BCS specimens increased with the increase of moisture content up to about 7 percent due to the increase of dry density, just like what will occur in soils. However, according to the surface energy theory (7), the increased moisture content in BCS could weaken the bonds between gypsum crystal particles. The balance point of these two factors in this case is at 7 percent of moisture content.

The second group of specimens shown in Table 3 explored the effect of curing conditions on UCS. It indicates that some of the specimens were also submerged into water for four hours prior to the test. As showing in Figure 4-b, with the decrease of relative humidity from 100 percent to 25 percent, the strength of the specimens increases from 0.44 MPa (63.8 psi) to 1.35 MPa (195.8 psi). It is interesting to notice that the strength of the specimen reaches 1.85 MPa (268.3 psi) when dried for four hours at 40°C prior to test, while the specimen soaked in water collapsed and exhibited no strength.

The third group of specimens was prepared at the optimum moisture content with its maximum dry densities. Unconfined compression tests were conducted on these specimens at different cure times. Half of them were tested to indicate strength gain with time. The other half were tested at the same frequency as the first half, but were subjected to a three-hour soak immediately prior to load testing. The results are shown in Figure 5. Figure 6-a is the photo taken when a BCS specimen was tested dry while Figure 6-b is the photo taken after a BCS specimen was soaked for three hours.

STRENGTH OF STABILIZED BCS

Test results discussed previously indicate a strong moisture dependence of UCS for BCS material. Therefore, a preliminary design of chemical additives to improve the moisture sensitivity of BCS was conducted. The results are shown in Table 4 with their curing conditions. Portland cement, fly ash, and ground granulated blast-furnace slag (GGBS) were used as chemical additive. The relative chemical and physical properties of the additives are given in Table 2. The Class C fly ash, complying with ASTM C-618, was obtained from Bayou Ash Co., Erwinville, Louisiana. Lone Star Industries, Inc. supplied the GGBS and the Type I Portland cement. The cement was a low alkali cement containing 51.4 percent tricalcium silicate (C_3S) and 5.7 percent tricalcium aluminate (C_3A).

The same procedure was used for the preparation of both the stabilized specimens and non-stabilized specimens. The BCS was thoroughly mixed with the cementitious materials before water was added. UCS tests were conducted after a 28-day cure period. Half of the specimens were tested dry with the other half submerged into water for four hours prior to the test. The results are given in Figures 7-a and 7-b.

It is obvious that the addition of cementitious materials to BCS material increased the strength and water resistance of the FG specimens. When soaked in water for four hours prior to test, the BCS specimens with cementitious materials added can remain at approximately half of their strengths when tested in a dry condition. Of the cementitious materials used in this evaluation, GGBS was the most effective. Even though soaked in water for four hours, BCS specimen S-2 stabilized with 8.18 percent GGBS retained an UCS of 1.86 MPa (270 psi). As indicated in Figure 7-b, higher amounts of GGBS in the stabilized SBC specimens increased their UCS values.

MINERALOGICAL AND MICROSTRUCTURAL ANALYSES

Mineralogical and microstructural analytical methods including X-ray diffractometry (XRD) and scanning electron microscopy (SEM) were used to characterize the BCS before and after cementitious stabilization. XRD analysis was carried out on a Siemens diffractometer D5000, using a $Cu\ K\alpha$ radiation at 40 kV and 30 mA. The scanning speed is 0.02° , two second counting at each step, and the angle scanned is $5-70^\circ\ 2\theta$. SEM analysis was carried out using Jeol (JSM-840). For SEM observation, each particle sample was first dried through freezing and coated with a film of gold.

The mineralogical analysis on pure BCS material indicated that its main phase compositions were gypsum and hemihydrates, as shown in Figure 8-a. This figure shows the X-ray diffractometry (XRD) pattern of BCS when it was in its natural dry state. When the BCS material was mixed with water, compacted to form specimen cylinders, and cured for 28 days, the phase of hemihydrates vanished, as shown in Figure 8-b. However, the same figure shows no new phases can be traced when cementitious materials were added.

The SEM micrograph of the BCS specimen I-4, which was compacted at a moisture content of 9.10 percent and cured at 50 percent relative humidity for 28 days, demonstrates the visual presence of minerals in the sample. It shows a crystalline microstructure with stacked crystal particle size of about 5 – 50 μm , but a certain number of void spaces also exists between crystal particles, as shown in Figure 9-a.

The SEM micrographs of the specimens, which were added with cementitious materials and also cured for 28 days, were given in Figure 9 from b through d, respectively. When cement was added to the specimen, ettringite crystals and other possible hydrates were observed to be present around or between gypsum crystal particles of the specimen C-1, as shown in b of Figure 9. Small contact or bond can also be traced between gypsum crystal particles of the specimen F-1 when fly ash was added as shown in c of Figure 9. Significant change in microstructure of BCS specimen was exhibited when GGBS was added. As shown in d of Figure 9, gypsum crystal particles were covered with cementitious hydrates and developed into a whole mass.

DISCUSSION OF RESULTS

The sensitivity of BCS material to moisture content was demonstrated previously when BCS specimens were kept in different curing conditions. With the decrease of relative humidity for curing conditions, the strength of FG specimens increased. It is believed that in dry conditions, free water may be easily vaporized from the specimens and dissolved sulfate may be re-precipitated, resulting in extra bonds among the crystal particles. This is in agreement with another SEM observation (6). Considering the two extreme cases when drying the specimen prior to testing increased the strength significantly and soaking the specimen before testing lead to its collapse, it can be deduced that the strength of BCS material is developed with the bonds of interfacial free energy of gypsum crystals and cemented material deposited from the solution. However, both bonds are moisture dependent. When the initial moisture content is high or water penetrates to the specimen later on, the interfacial free energy bond is shielded and the cemented bond disappears due to dissolution. Once drying, the strength will recover as both bonds will recover, be maintained, and increased. The results from the mineralogical and microstructure analyses also gave support to the above discussion as shown in Figures 8-a and 8-b.

Cementitious materials have been incorporated in phosphogypsum for construction of road bases (10). Cementitious materials have various types and properties, but all depend on pozzolanic reaction and/or cementitious hydration reaction to produce hydrates and develop

strength. This study also indicates that with the addition of cementitious materials, the strength and water resistance of BCS specimens are both increased significantly. Among cementitious materials, cement can hydrate to form a network and serve as the “glue” that provides structure and strength of the BCS specimen. It can also react with gypsum to form ettringite. However, the properties and behavior of ettringite in cement–gypsum system have not been fully understood (11), but they have been proved by Louisiana’s experience to be detrimental. BCS base course stabilized with Portland cement can have serious expansion problems.

Fly ash is a form of pozzolan that needs the presence of high calcium to produce its cementing property. GGBS is a cementitious material that reacts more slowly with water than Portland cement does, but it can be activated chemically (12). Activators can be either alkaline activators such as lime or sulfate activators such as gypsum. Both activators are abundant in BCS material. In this study, it is believed that BCS material activated the latent hydraulic activity of GGBS and led to high and stable strength development.

The SEM observations on stabilized BCS revealed some changes in microstructure. Although the SEM technique is subjective in terms of quantifying the mineral, it is evidence that certain morphology is predominant. Comparing Figure 9-a to Figures 9-b, -c, and -d, the addition of cementitious materials produced varying degrees of bonds between gypsum crystal particles. These bonds are responsible for the increase of strength and water resistance of the BCS specimens.

SUMMARY AND CONCLUSIONS

BCS is a byproduct of the Louisiana chemical industry. As a low-cost recycled material, it is highly competitive as an alternate to pavement base material in south Louisiana. While the use of this material has performed satisfactorily on many projects, unresolved issues have caused a continued evaluation and re-evaluation. A limited laboratory evaluation on BCS was conducted in this study and some preliminary findings can be summarized as follows.

The unconfined compressive strengths of the untreated BCS at 28 curing days varied depending on the initial moisture content and relative humidity of curing conditions. Drying the specimens could increase its strength significantly, whereas soaking the specimens would lead to its collapse. Results from this evaluation suggest that the bonds between gypsum crystal particles are interfacial surface energy and cementing agent precipitated from the solution around the particles, which is consistent with previous studies (6,7). Since both bonds are highly sensitive to moisture conditions, the penetration of water into the matrix of the BCS material would shield interfacial surface energy and dissolve cementing agent, resulting in the decrease or loss of its strength. Drying the specimens would recover both bonds and regain the strength.

The addition of cementitious materials such as cement, fly ash, and GGBS can increase the strength and water resistance of the BCS specimens. The extent of this improvement is different for different cementitious materials. When soaked in water prior to

test, the strengths could reach approximately half of their original values. This improvement can be attributed to pozzolanic reaction and cementitious hydration. Incorporation of GGBS is an effective method to improve the properties of BCS material. It not only increases the strength of BCS, but also retains a high strength after soaked in water for four hours. The latent hydraulic activity of GGBS is activated by the presence of gypsum, and the hydrates released will cover and bond the gypsum crystal particles and convert the weak bonded particle stack of BCS material into a strong integral mass. Fly ash is not an effective stabilizer to the BCS material due to the lack of calcium oxide in fly ash-gypsum systems and the weak pozzolanic activity.

The long-term volumetric stability of stabilized BCS is not fully understood at this time. Field reports from Louisiana indicated the expansion damage due to the cement stabilization of BCS. Further research is needed in this area.

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Table 1. Summary of BCS Base Course Testing Sections

Calcium Sulfate			
Site		FWD	DCP
LA 16 - Watson to Wiess		342 ksi	3.4 mm/blow
LA 3002		91 ksi	5.3 mm/blow
LA 1034		746 ksi	2.0 mm/blow
LA 16 Denham Springs		243 ksi	2.2 mm/blow
LA 73 - Jefferson Highway		NA	3.5 mm/blow
Average		358 ksi	3.3 mm/blow
Soil Cement			
LA 16		473 ksi	1.4 mm/blow

Table 2. Compositions and Properties of Materials Used

Composition (%)	FG	Type I cement	Fly Ash	GGBS
SiO ₂	0.5	22.4	47.5	34.5
Al ₂ O ₃	0.1	4.1	20.6	9.5
Fe ₂ O ₃	0.2	3.9	5.2	1.3
CaO	29.0	65.1	16.2	39.6
MgO	-	1.2	2.5	10.9
K ₂ O	-	0.15	0.7	1.26
Na ₂ O	-	0.11	0.3	0.48
SO ₄	54.0	-	-	-
CO ₃	3.0	-	-	-
Moisture	5-30	-	-	-
Specific Surface Area (m ² /kg)	-	386	350	380

Table 3. Specimen Cylinders with Different Moistures at Molding and Curing Conditions

No.	Dry FG (weight %)	Water (weight %)	Curing condition (RH)	Curing time (days)
I-1	97.56	2.44	50%	28
I-2	95.24	4.76	50%	28
I-3	93.11	6.98	50%	28
I-4	90.9	9.10	50%	28
I-5	88.89	11.11	50%	28
E-1	90.9	9.10	25%	28
E-2	90.9	9.10	50%	28
E-3	90.9	9.10	100%	28
E-4	90.9	9.10	50%*	28
E-5	90.9	9.10	50%**	28

* soaked in water for 4 hours prior to test; ** dried at 40°C for 4 hours prior to test

Table 4. Specimen Cylinders Stabilized with Cementitious Materials

No.	Dry FG (weight %)	Cement (weight %)	Fly ash (weight %)	GGBS (weight %)	Water (weight %)	Curing Condition (RH)	Curing time (day)
C-1	86.0	4.09	-	-	9.10	50%	28
C-2*	86.0	4.09	-	-	9.10	50%	28
F-1	82.7	-	8.18	-	9.10	50%	28
F-2*	82.7	-	8.18	-	9.10	50%	28
S-1	86.0	-	-	8.18	9.10	50%	28
S-2*	82.7	-	-	8.18	9.10	50%	28
S-3	82.7	-	-	4.09	9.10	50%	28
S-4	74.5	-	-	16.36	9.10	50%	28

* soaked in water for 4 hours prior to test

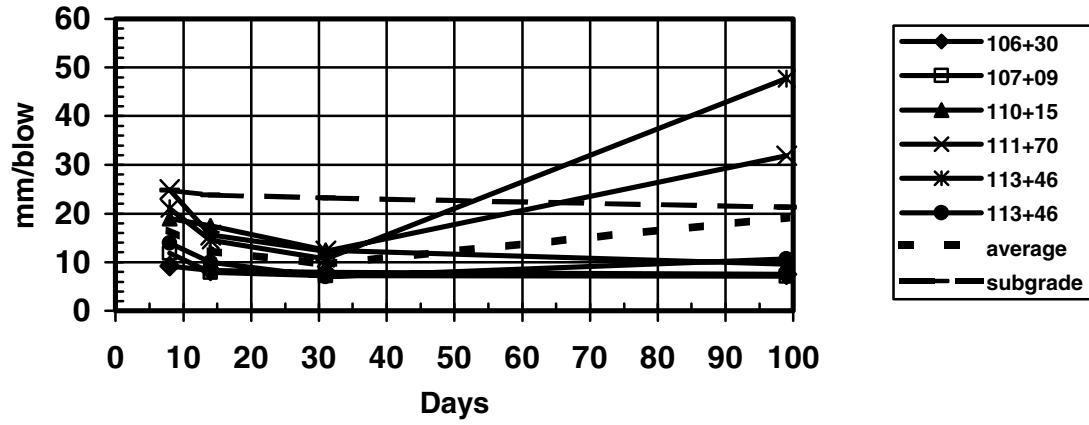


Figure 1. DCP Results - Evangaline Thruway, Louisiana

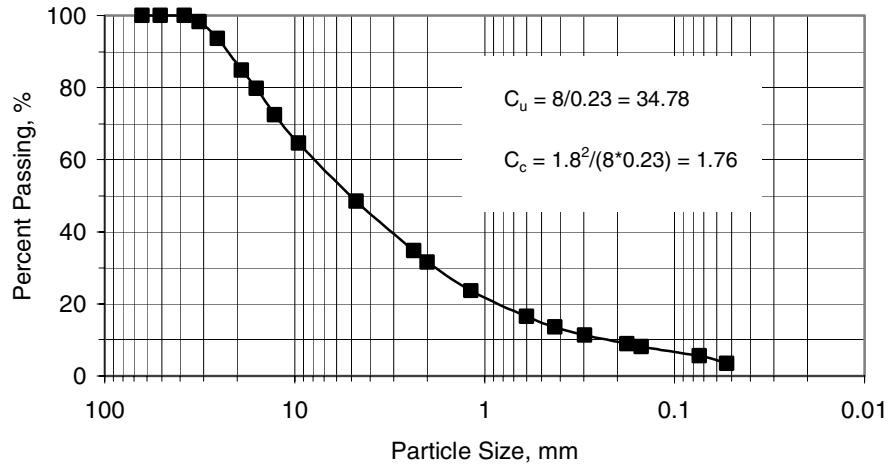


Figure 2. Particle Size Distribution of Blended Calcium Sulfate

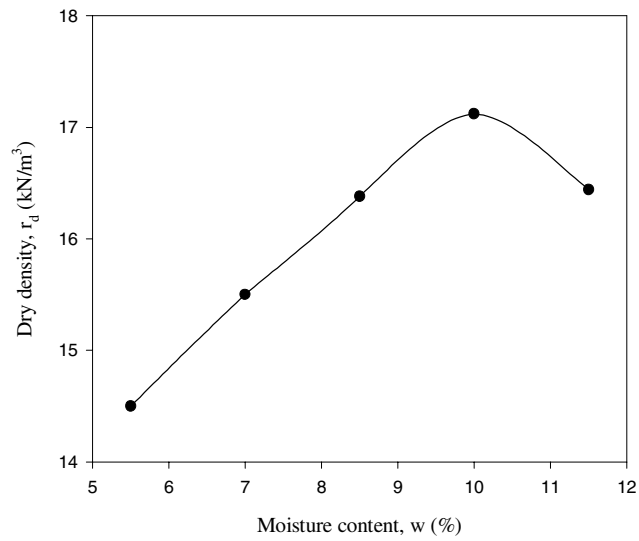


Figure 3. Standard Proctor Test Results for BCS Material

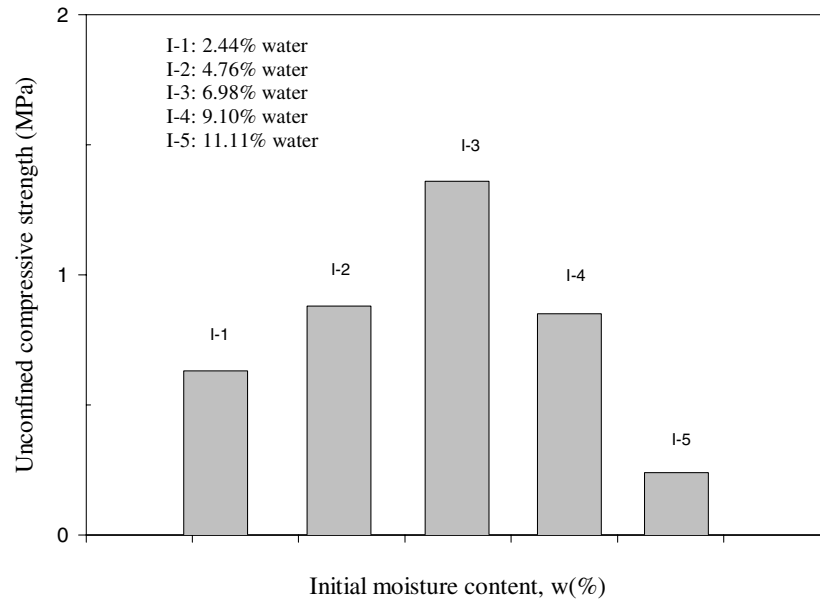


Figure 4-a. UCS Values of BCS Specimens with Different Initial Moisture Content

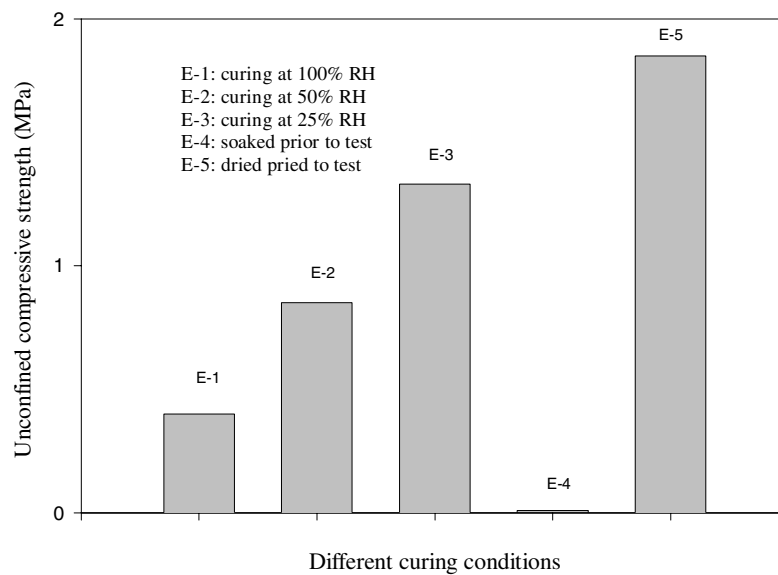


Figure 4-b. UCS Values of BCS Specimens with Different Cured Condition

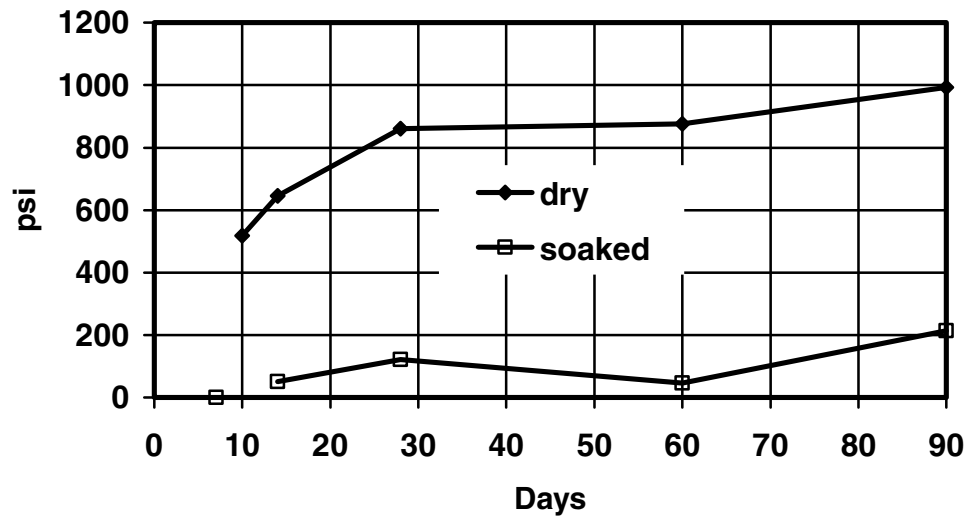


Figure 5. BCS - Unconfined Compression Test



Figure 6-a. A BCS Specimen Tested on Dry



Figure 6-b. Two BCS Specimens after Soaked in Water

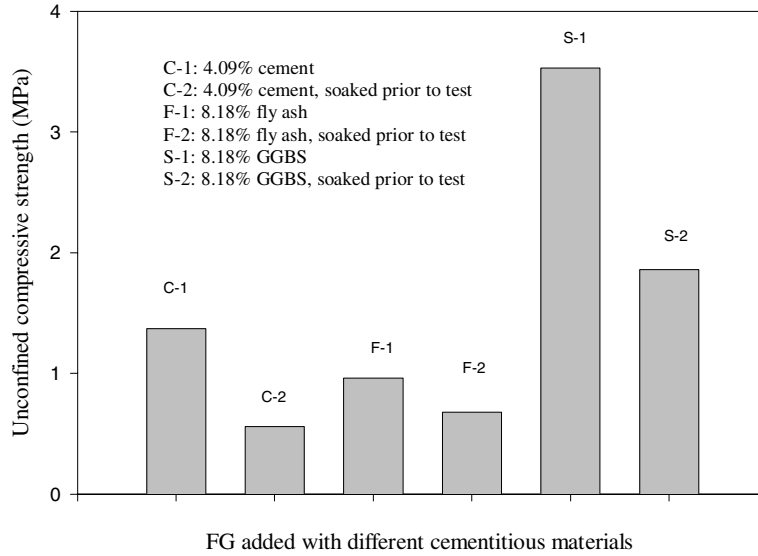


Figure 7-a. UCS Values of BCS Specimens Stabilized with Cementitious Materials

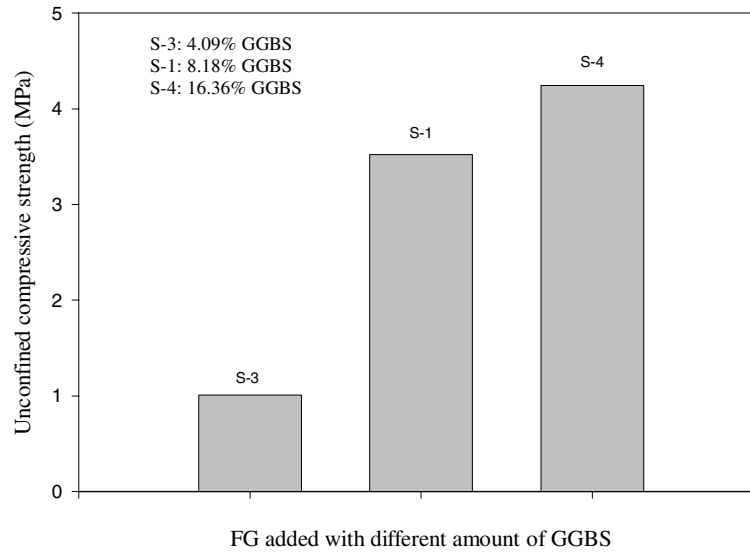


Figure 7-b. UCS Values of BCS Specimens Stabilized with GGBS

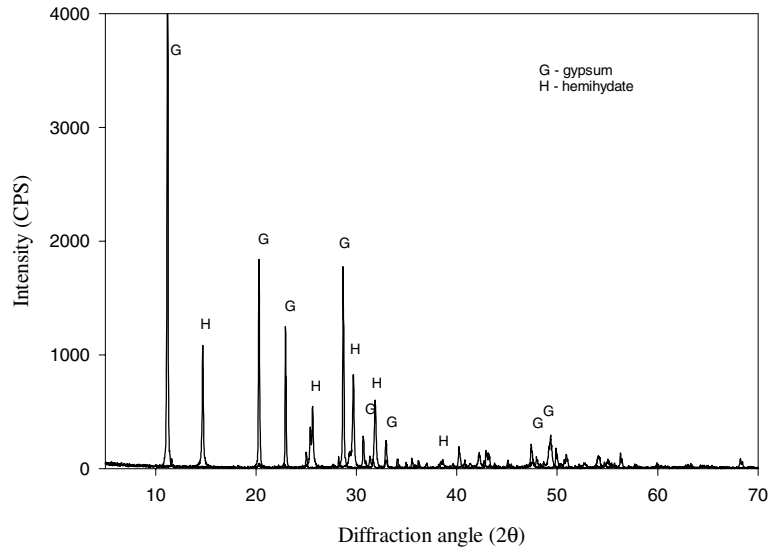


Figure 8-a. XRD Pattern of Pure BCS Material

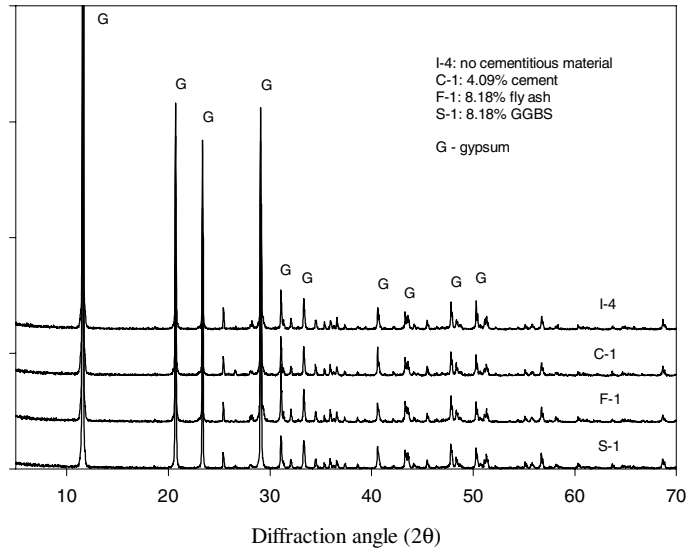


Figure 8-b. XRD Patterns of BCS Specimens with or without Cementitious Materials

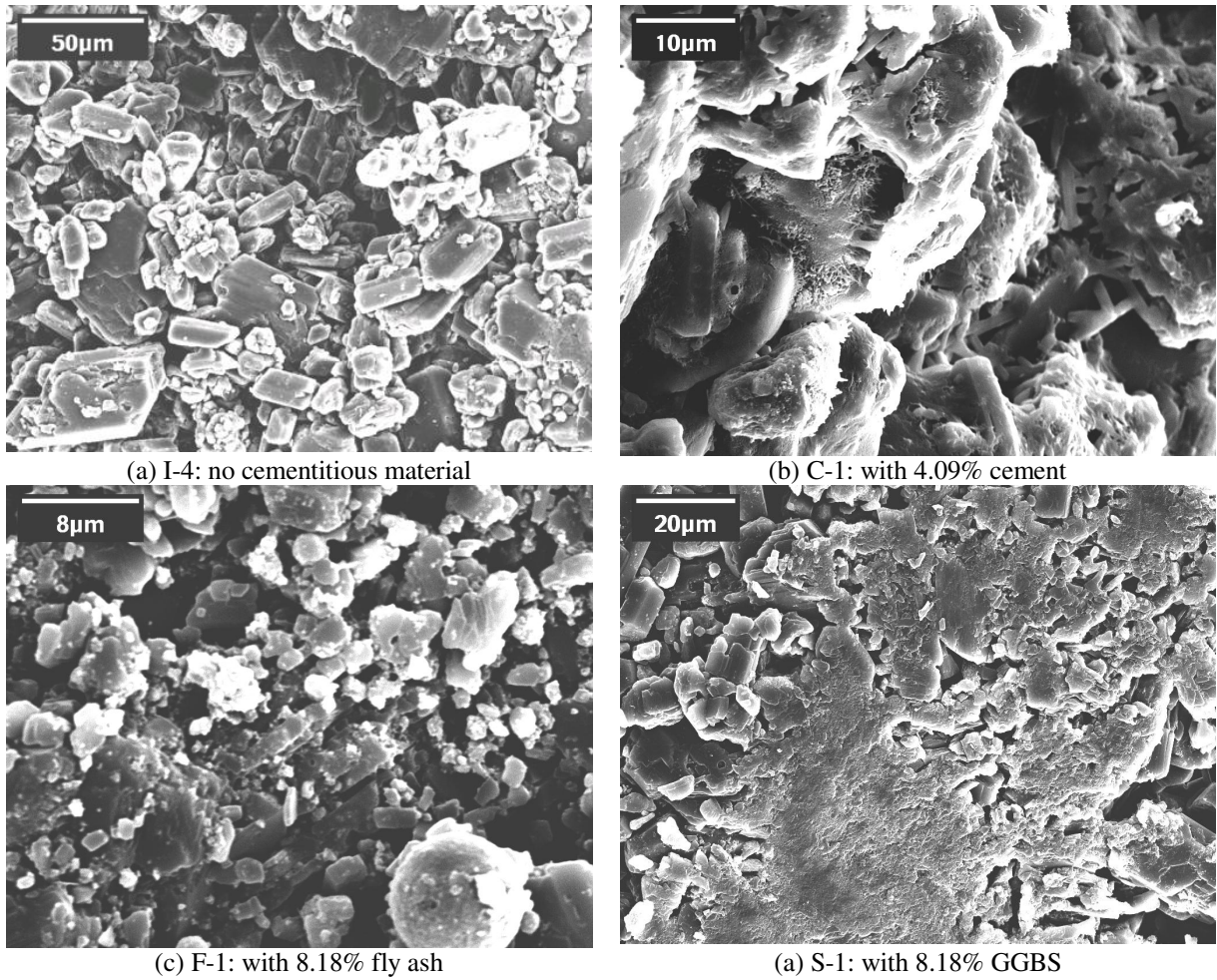


Figure 9. SEM Micrograph of BCS Specimens with or without Cementitious Materials