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Abstract:	Contribution of fly ash (FA) as a complementary additive for dredged sediment (DS) stabilization was studied. The study is focused on definition of an efficient combination(s) to raise the DS properties. FA with lime and cement with both was used. Then, micro and macroscale investigations was performed. Test results demonstrate that FA additions promoted nucleation, formation of cementitious compounds and fabric modifications in sediment. DS stabilized using FA component show lower shrinkage and higher mechanical resistance than that stabilized using conventional binders. FA is found to be efficient in sediment-lime and sediment-cement mixtures since it accelerates cementation and strength gain.

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Highlights

- FA acts as filler when incorporated with sediment at early age
- FA promotes nucleation, cementitious compound growth resulting in porosity refinement
- FA reduces shrinkage and increases strength gain of mixtures, regardless curing time
- The effectiveness of FA addition is confirmed when used with sediment-lime and sediment-cement

Abstract

This study deals with the definition of an efficient combination of fly ash (FA) with lime or cement and with both, to improve the dredged sediment (DS) properties. At early age, filler and nucleation effects of FA lead to a refinement of the microstructure in addition to the macro porosity reduction induced by lime and cement. At long term, the microstructure becomes denser due to the pozzolanic property of FA. At macroscale, DS stabilized using FA show lower shrinkage and higher mechanical resistance than that stabilized without FA, with more pronounced effects when FA is mixed with cement.

Key words: dredged sediment, fly ash, soil stabilization, mechanical resistance, microstructure

1. Background

Selection of pavement materials is based on mechanical properties such as strength and stiffness. This procedure is adequate in case of raw and inert materials are used. In stabilized materials, physical changes and chemical reactions take place [1][2]. Therefore, it is important to identify modifications

which occur at different levels, in order to determine how the action mechanisms of different additives affect soil resistance.

Multiscale analysis leads to a comprehensive knowledge of improvement of stabilized soil using hydraulic binders. In such a way that microscale analysis provides evidences of pozzolanic reactions, cementing material formation and evolution of crystalline phases, and macroscale analysis explains enhances in mechanical properties resulted from previous interaction highlighted at microscale level [3][4].

At present time, Portland cement and lime are considered as the most convenient stabilizers for soil [2]. In soils stabilized using Portland cement, soil gradation changes since cement grains may fill a little portion of the soil voids [5] [6]. Hence, cement hydration is responsible for the significant strength gain over time. The mechanism of interaction between the different components can be explained as follows: Portland cement hydrates using available water in the soil and form a stone-like material [3][7].

Typical mineral components of Portland cement are calcium and silicon which are often in the form of oxides, such as CaO, SiO_2, Al_2O_3 and FeO_2. The main clinker constituents are tricalcium silicate (C_3S), dicalcium silicate (C_2S), tricalcium aluminate (C_3A), and calcium ferroaluminate (C_4AF). To control reaction rate, gypsum ($C\underline{S}H_2$) might be added to cement.

It is known that the hydration of clinker constituents produces cementitious compounds such as calcium silicate hydrates (C-S-H), calcium hydroxide (CH). In pre induction of hydration, cement reacts with the available water and the most active phase C_3S produces calcium ions and OH^-, SO^{2-}_4, K^+, Na^+. In dormant stage, C_3S hydration continues and C_2S begins to be hydrated. CH separates from hydrolysis of C_3S and C_2S and may precipitate into empty voids. Ettringite (Ett) also forms due to the reaction of gypsum with C_3S and C_4AF [8].

Over time (at early stage), C-S-H crystalline phases form an acicular morphology which branch, forming a honeycomb-shape structure. CH crystallizes in large crystals (~40μm), presenting hexagonal plate-

shape, depending on the produced lime amount in early stages and the available free space. Ettringite crystalizes in a needle-shape, with length up to 10μm and diameter of 0.25 μm, which does not branch. All aforementioned phases and processes have been observed in cement-based materials, included in soil-cement mixtures [3][7][9].

At microscale level, when cement is mixed with soil, the soil-cement mixture presents a significant gain of mechanical strength. Soil-cement strength results from cementation bonds and pore space reduction [10][11]. Accordingly, cementitious compounds fill up the pores and connect cement grains resulting in the increase in intra-aggregate pore volume [12].

When lime is used in soil stabilization, soil fabric changes since lime addition induces cation exchanges and generates flocculation/agglomeration mechanism, in short term. In other words, water dissolves some constituents of lime (CaO, $CaSO_4$, MgO and quartz) that react with soil and reduce double diffuse layer (DDL), resulting in flocculation (agglomeration). This process reduces soil plasticity and improves workability [13]. Over time, pozzolanic reactions take place at alkaline environment (pH=12), forming cementitious compounds i.e. C-S-H and C-A-S-H, that are responsible for increasing of long-term strength [3][10].

At microscale level, soil-lime strength gain is also explained based on the formation of cementitious compounds from pozzolanic reactions. However, the increase of soil resistance generated by lime addition is less significant than that observed in mixtures using cement; and the use of lime is preferably recommended for clayey soils, in order to improve soil fabric, plasticity and workability [1] [2][13]. It is also worth noting that the mechanical properties of lime-treated soils are affected by the curing temperature. By studying the stiffness evolution of a silt soil stabilized with quicklime cured at 30 °C, Silva et al. [14] observed two different stages on the stiffness evolution suggesting the existence of two different chemical phenomena involved. Evolution in the first stage seems to be mostly related to the formation of calcium aluminate hydrates (CAH). However, the evolution in the second stage can be more related to a structural rearrangement of CAH and the formation of calcium silicate hydrates

(CSH). These two distinct stages involved in the evolution of elastic modulus (E) with time suggest the existence of two apparent activation energies (one for each process).

To maximize the benefits of soil stabilization, a binary or ternary combination of hydraulic binder is proposed. Most of the time, lime and cement combination is recommended. Lime-cement stabilization combines workability enhancement from lime addition and resistance gain from cement addition. Both additives may be mixed without disturbing their own action mechanism. However, multicomponent mixed materials show differences in physical and chemical properties due to coexistence of cement hydration and mineral admixtures, changing hydration kinetics process and microstructure formation mechanisms [15]. According to [16] [17], the dosage of cement and the water content have a significant impact on unconfined compression strength (UCS) values. By testing soil-cement mixtures with dosage of cement between 10 and 13%, Ribeiro et al. [17] observed that UCS is always larger for highest dosage of cement independently from the water to cement ratio, whereas independently from the dosage of cement, there is a clear optimum water content providing the maximum UCS value. Finally, compressive strength develops faster with time when larger water and cement ratios are adopted. For treatment using lime, the effects of molding water content on UCS values seems to be insignificant independently from the dosage of lime [18].

For sustainable development purpose, the use of local soil, waste and industrial byproduct are encouraged to supply earthwork and earthen structure [6][19][20]. Two well-known industrial byproducts are often cited: bottom ash and fly ash. The benefits of use of bottom ash for road construction was been recently studied by different authors [21], [22], [23]. On the other hand, the use of fly ash still requires further investigation because different mechanisms of interaction need to be clarified.

Researches in concrete technology has demonstrated that fly ash (FA) improves mechanical properties of concrete, reduces the costs of production and is ecologically beneficial. FA addition may enhance durability of the matrix and reduces the loss of heat energy during cement hydration [15][24].

FA is a waste from coal-fired electrical power plants that is being studied as admixture in soil stabilization [6][12][20]. Commonly, FA is composed by calcium, silicon and aluminium. The main oxides are CaO, SiO_2 and Al_2O_3. Class C Fly ash contains about 20% of CaO which might induce cation exchanges, flocculation and pozzolanic reactions, resulting in strength gain. Aluminum rich composition of FA might promote a specific cementitious compounds formation, such as CAH and CASH [3][7][25].

In concrete, FA addition stimulates reaction rate of cement hydration, promotes nucleation and growth of cementitious compounds. However, FA may also retard the onset of acceleration (Stage III of hydration) because of (i) inhibition of CH precipitation due to the formation of water containing organic species and (b) slows formation of Ca rich surface layers on clinker phases, in the case of aluminum rich FA [26].

The effects of FA action on soils are then physical and chemical. At physical point of view, finer particles of FA would fill voids of soil particles. Chemically, pozzolanic products induced by FA presence would fill pores. Both effects reduce porosity so that microstructure becomes denser. As results, strength and stiffness are increased and compressibility is reduced [27].

Research findings suggest that FA disperses clusters of soil and strength development is controlled by FA hydration [20][28][29], leading to an analogy with the aforementioned processes in cement pastes. It is worth noting that water content of the fly ash stabilized soil mixture affects the strength [30]. The maximum strength reached in soil-fly ash mixtures generally occurs at moisture contents below optimum moisture content for density. For silt and clay soils the optimum moisture content for strength is generally four to eight percent below optimum for maximum density while for granular soils the optimum moisture content for maximum strength is generally one to three percent below optimum moisture for density. Therefore, it is crucial that moisture content be controlled during construction. Initial water content significantly affects the efficiency of soil stabilization.

In soil stabilization, FA addition alone is not sufficient to significantly increase strength to the design allowable levels, then a combination with other additives is required [29]. Particular attention must be paid when FA is added to soil-cement mixture. FA may compete with cement for the water available in the soil. This competition may be detrimental to cement hydration and it is more problematic in soil-cement because of the typical low water and cement ratio (w/c).

Interestingly, in binary combination involving lime and FA, stabilized soils present some benefits resulting in strength gain. Notwithstanding, Sivapullaiah and Jha [29] indicate that there may be particular FA contents to find greater resistance in the short and long term, respectively. Low contents of FA increase strength in 28 days, whereas high contents of FA increase strength in 4-7 days. Generally, FA in soil-lime changes the rate of strength gain and not the strength itself. Therefore, FA content up to 15% is recommended. The effects of initial water content on the compressibility, strength, microstructure, and composition of a lean clay soil stabilized by compound calcium-based stabilizer composed of cement, lime and fly ash was investigated by Yin et al. [31]. It was observed that as the initial water content increases in the range studied (from 11 to 19%), both the compaction energy and the maximum compaction force decrease linearly and there are less soil aggregates or agglomerations, and a smaller proportion of large pores in the compacted mixture structure. In addition, for specimens cured with or without external water supply and under different compaction degrees, the variation law of the unconfined compressive strength with initial water content is different and the highest strength value is obtained at various initial water contents. Finally, with the increase of initial water content, the percentage of the oxygen element tends to increase in the reaction products of the calcium-based stabilizer, whereas the primary mineral composition of the soil-stabilizer mixture did not change notably.

Regarding the microstructure organization, Furlan et al. [6] demonstrate that FA addition refines the pore structure prior the development of pozzolanic reaction. This previous study demonstrates that FA can be used as soil stabilization additive. However, some question related to the reactivity of FA combined with other chemical additive requires further investigation. This paper aims to investigate

the contribution of FA in soil stabilization using hydraulic binders, with the purpose of finding: (i) technically compatible material options, (ii) rational use of waste and byproducts and (iii) optimized mix dosage.

2. Experimental program

Chemical stabilization of soils induces physicochemical changes of soils characteristics due to chemical interactions between additives and soil. These changes imply improvements in the mechanical behavior of the soil. Therefore, a multiscale analysis was selected in order to provide a holistic knowledge of the stabilization mechanisms.

If, on the one hand, microscopic analysis can show the new cementing products resulting from chemical reactions, on the other hand, it is worth quantifying their impacts on mechanical performance improvement of stabilized soils. Then, measurements of design and mechanical properties are necessary. Moreover, the combined use of these techniques allows to relate the changes in different scales, in order to highlight advantages and disadvantages of chemical stabilization.

Thus, the present experimental program aims to respond these questions, demonstrating the beneficial of using fly ash as a complementary additive in soil stabilization, indeed, when combined with conventional hydraulic binders.

In this study, dredged sediment from La Baule Le Pouliguen Harbor, France, and two cementitious additives were used. Fly ash was added to mixtures containing lime, cement and lime and cement. Only chemical stabilization was made (so without gradation correction) in order to find the best combination of additives to improve the characteristics of this dredged soil considered as waste, regarding economic and environmental aspects.

2.1. Materials

The geotechnical properties of the sediment were carried out in accordance with GTR guide [32] dedicated for embankment and pavement applications. Table 1 shows results of soil characterization based on results of maximum specific gravity, and organic material, pH and carbonates contents.

Table 1. Geotechnical properties of sediment

Parameters	Value
Sand fraction > 63µm (%)	23
Silt fraction - 2 to 63µm (%)	41
Clay fraction < 2µm (%)	36
Specific gravity (g/cm^3)	2,66
Plasticity limit (%)	36.06
Liquidity Limit (%)	54.54
Plasticity Index	18.48
Organic matter content (%)	10.97
Carbonates content (%)	22.21
pH	8.5

Proviacial® ST quicklime was added to the sediment soil. The quicklime was provided by LHOIST from Dugny-sur-Meuse, in Lorraine, France. This lime contains at least 90% of calcium oxide and at maximum 2% of magnesium oxide. The lime content added to soil was 2%.

Portland cement was a CEM II/B-LL 32,5R CE CP2 (French Standard) whose short-term resistance is 32.5 MPa. Clinker content is between 65 and 79%, being its chemical constituents: tricalcium silicate (66%), dicalcium silicate (10%) and tricalcium aluminate (7%). Limestone is the main natural component of this cement, presenting a total organic material less than 0.20% in mass. The cement content added to soil was 7% of the dry mass of the sediment.

Sodeline® Fly ash is manufactured and was provided by the central Emile Huchet in Saint Avold, France. The main constituents of this fly ash are silicon dioxide (47.36%), aluminum oxide (21.63%) and calcium oxide (8.52%). It is important to point out that sulfur content (4.02%) is higher than the conventional ones. The fly ash content added to soil was 9%.

The particle size distribution of sediment, hydraulic binders and fly ash are summarized in Figure 1. The particle size distributions help to understand the contribution of binders on the sediment particle arrangement. The particle size distributions were carried out with Malvern mastersizer.

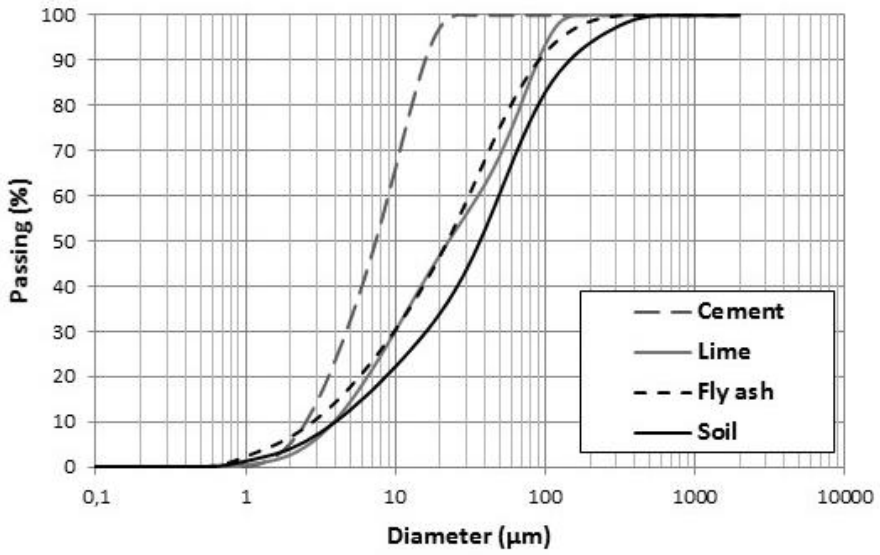

Figure 1: Grain size distribution of materials

Comparing the curves from Figure 1, it can be observed that the sediment is composed with coarsest material (D_{90} =150 µm), with particle diameters between 500 and 1 µm; and cement is the finest binder compared to lime and FA (D_{90} =15 µm), with particle diameters between 10 and 2 µm, that is also the most uniform distribution. Particle-size distributions of lime and FA are quite similar (D_{90} =90 µm), with particle diameters between 200 and 0.8 µm.

2.2. Procedures of specimen preparation

Raw sediments have been oven-dried at 50°C for 48h prior to the treatment process. The sediment mixtures considered in this study are respectively formulated with 2% lime (S2L), 7% cement (S7C) or 2% lime and 7% cement (S2L7C). The percentages considered here correspond to the dry mass of sediment. A binary or ternary binder mixture has also been proposed for the mixture including fly ash (FA). The rate of fly ash addition, which is equal to 9%, represents the sum of the standard binder rates used, i.e. 2% lime and 7% cement: S2L9FA, S7C9FA, S2L7C9FA.

Proctor tests were performed according to French standard (NF P94-093) to get the optimum parameters of the mixtures, that is optimum water content and maximum dry density. All mixtures were prepared based on several precedent dosages [33]. In addition to mixture design, shrinkage tests

have been carried out to measure the effect of the binder on the volumetric strain of the compacted mixtures. Mixtures were compacted at their optimum characteristics in an oedometric cell of 101 mm in diameter and 7mm thick.

Specimens for UCS test were prepared according to Proctor optimum parameters. Mixing procedure considered the addition of extra amount of water due to the binder presence, in order to preserve the optimum Proctor parameters. Thus, water content was increased in 1% for each percentage of added lime, 0.4% for each percentage of added cement, and 0.1% for each percentage of added fly ash.

Before compaction, the mixtures using lime underwent a previous period of 2 hour in a closed recipient for the former reactions of lime and to avoid carbonation reactions. Cylindrical specimens (76mmx38mm) were statically compacted. After compaction, specimens were packed in plastic film, to prevent the loss of moisture during curing. Curing was made in a room at controlled temperature (20±1°C) in different times, namely: 7, 28 and 90 days.

An extra specimen of each mixture was produced for the microstructure investigation. Cubic samples ($10mm^3$) were sampled and freeze-dried from a compacted specimen at 7 and 28 days. To stop the hydration and chemical reactions of the binders, cubic samples had the water removed by sublimation.

2.3. Methods

Unconfined compression strength (UCS) tests were performed according to French standard (NF P94-420) under a constant strain rate of 1mm/min. After test, water content (w) was measured by oven drying method at 105°C. In total, 3 specimens were tested and all results represent the average value of UCS and w.

The volumetric deformation induced by shrinkage was followed until full stabilization within 9 days for all mixture. Volumetric deformation of all samples was monitored by means of a digital caliper.

Microscale analysis was based on X-ray diffraction (XRD) analyses, Scanning Electron Microscopy (SEM) observations and Mercury intrusion porosimetry (MIP) measurements.

XRD analyses were used not only to identify the crystallized component resulting from the hydration of cement and pozzolanic reactions but also to characterize sediment matrix. The XRD analyses were performed on powder using a Brucker diffractometer with a detector over the range 5°80°2θ using Cu-Kα_1 radiation (1.55Å). Diffractometer operated with input voltage of 30 kV and current of 10mA. Crystallography Open Database (COD) was used to identify constituents and new crystalline phases in the patterns.

SEM images were used to investigate the mineral phases resulting from the interaction between the sediment and the binder components. SEM observations were carried out using a FEI Inspect F-50 SEM instrument coupled to an energy dispersive X-ray analyzer (EDX). To improve the image quality, samples are coated with gold.

The impact of the mineral compound development on the sediment structure resulting from hydration mechanism of the binder mixture was followed by the pore distribution. The pore size distributions were plotted from MIP data obtained with Micromeritics Autopore IV.

3. Results

3.1. Microstructure investigation

3.1.1. Microstructure investigation by XRD

The main strength gain in chemical stabilized soil is mostly attributed to cementitious compounds formed from cement hydration and/or pozzolanic reactions. Figure 2 shows XRD patterns of all mixtures at 7 and 28 days. These patterns compare mixtures with original soil, in order to identify new crystalline phases addressed to chemical reactions. Different curing times are considered in attempt to evidence the formation and growth of cementitious compounds. New peaks in patterns confirmed not only the formation of cementitious compounds but also indicated the presence of non-hydrated materials.

In Figure 2, it is observed that untreated sediment presents quartz (Q), feldspar (F), calcium carbonate (C), Illite (I) and kaolinite (K) in its composition. Samples at 7 days (Figure 2a) showed the presence of

gypsum (S2L9FA), C_2S (S2L7C and S2L7C9FA) and anhydrite (S7C). It may indicate (a) insufficient water to hydrate the binders, (b) delay in cement hydration due to FA addition, (c) restricted water availability of anhydrous grains surrounded by hydration products due to the formation of strong bounds between soil aggregates which can modify the porous structure and thus can slow down or hinders the permeation and/or diffusion process of water, (d) a combination of previous assumptions.

A remarkable peak of calcium carbonate ($CaCO_3$) was particularly observed in S2L7C9FA at 7 days. The occurrence of $CaCO_3$ was associated to the presence of limestone in the cement (CEM II) and carbonates in the untreated sediment. The non-observation of such $CaCO_3$ peak in S7C or S2L7C could be explained by the heterogeneous distribution of binders in the sediment, the sediment itself and the low percentage of cement used. Since the specimens have been kept packed during curing, carbonation reactions were disregarded.

Regarding hydration products, new peaks pointed out CH in S7C9FA, C-S-H in S2L and S2L7C, and ettringite in S7C and S7C9FA. The mixture with S2L9FA presented CASH peak. Even though some crystalized new phases from cement hydration have been identified, they did not occur in an evident way and this might be a consequence of the low water/cement ratio (w/c).

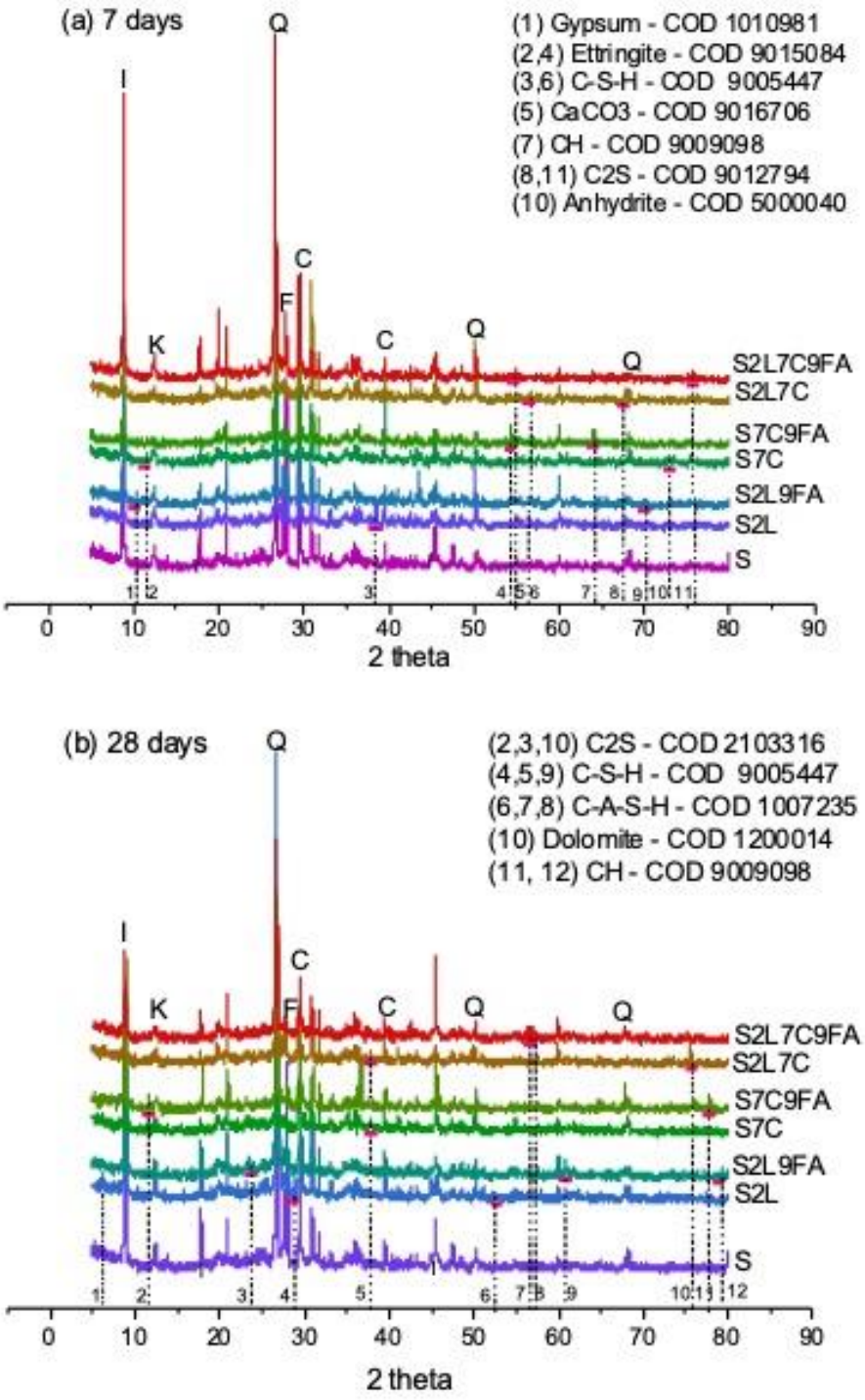

Figure 2: XRD results of mixtures at 7 days (a) and 28 days (b)

Figure 2(b) shows the pattern of mixtures at 28 days. Belite is still identified in S7C9FA and S2L7C. Other new peaks refer to C-S-H in S2L, S7C, S2L9FA and S2L7C, CH in S2L9FA and S7C9FA, and CASH in S2L and S2L7C9FA. The presence of non-hydrated material suggests hydration delay or water

insufficiency. This might be related to (i) lime addition, because the change of texture (agglomeration) would interfere with the amount of available water for hydration, since water may be trapped in the soil cluster; and/or (ii) FA addition, because water demand for FA is generally important due to its appreciable porosity, it might create a competition for available water. Besides, it is important to remember that cementitious compounds in early ages are poorly crystalized and they are not well detected by diffractometer.

Regarding cementitious compounds, one may observe that all treated samples had produced them at 28 days, unlike what happened at 7 days. Curing time was preponderant to development of cementitious compounds, since time allows binders to hydrate according to their own reaction rates (pozzolanic reactions) and allows also the transport of water necessary to the reactions of anhydrous grains surrounded by hydration products. This implies that it would be recommended at least 28 days to verify strength gains, at macroscale.

Hydrated silicates exhibit different morphologies and it might be more problematic for chemically stabilized soil due to the several factors, such as the type of additive, the low additive contents, the low water content, the complexity of hydration mechanisms and cementitious material formation of multicomponent mixed materials [15][25].

In other words, since C-S-H are amorphous and/or poorly crystallized, it may be advisable to be careful when reading XRD patterns because of the disturbing factors (producing a heterogeneous sample) and to combine other microstructural data sources to help to support the results analyses.

3.1.2. Microstructure observation by Scanning Electron Microscopy (SEM)

SEM images are often used to provide evidences of cementitious compounds development and mixture changes. Firstly, Figure 3 attempts to demonstrate visually constituents and compounds, which were previously identified in the mixtures using XRD tests (Figures 2 and 3). SEM Images are presented in appropriate magnificence to highlight details of the materials. As it can be seen in figure

3, almost all compounds identified with the XRD patterns are observed and confirmed, such as gypsum, $CaCO_3$ (found in sediment and in mixtures with cement), hydrated silicates and FA as well.

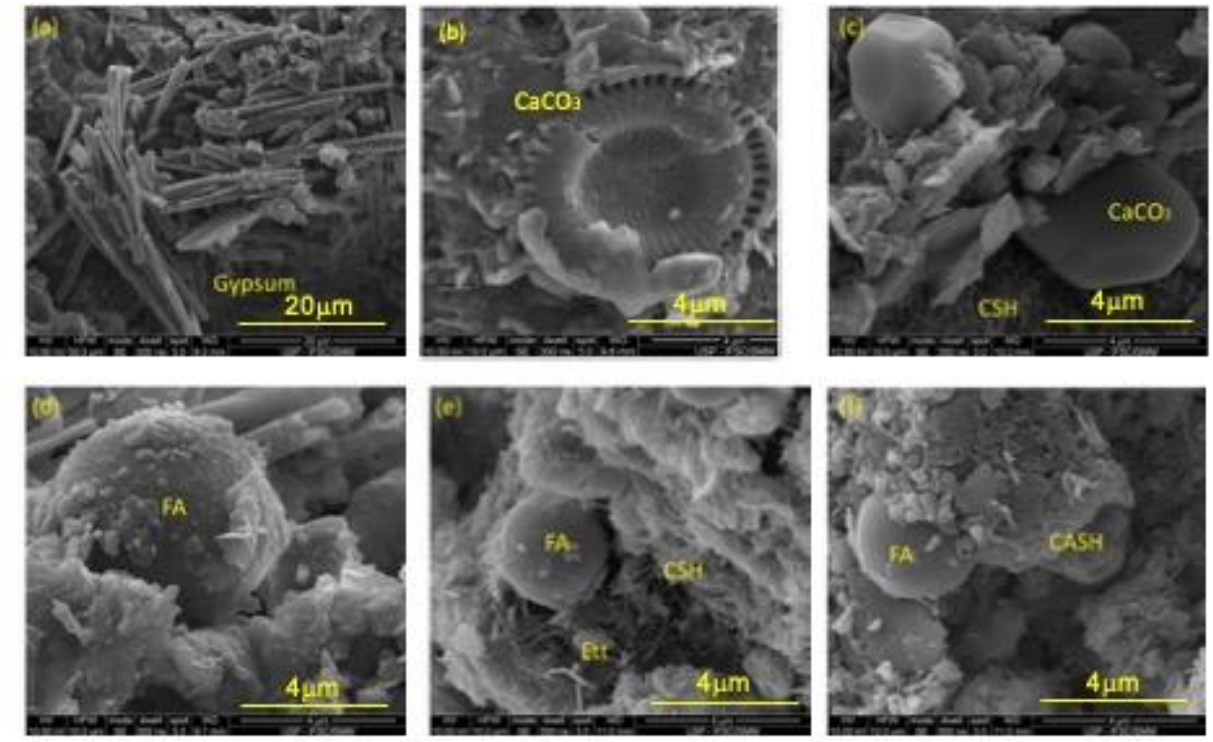

Figure 3: Identification of constituents and cementitious compounds of mixtures from (a)S2L9FA-7d, (b) S7C, (c)S2L7C9FA – 7d, (d) S7L9FA – 7d, (e)S2L7C9FA-7d,(f) S2L9FA-28d

Secondly, Figures 4 shows SEM images obtained from sediment mixtures without and with FA (4a and 4b), in order to observe structural changes and cementitious compound presence from 7 to 28 days. Figure 4(a) shows images from mixtures using conventional binders. Mixture with lime (S2L) at 7 days exhibits a flaked-like structure, as a result of cation exchange and flocculation mechanism. At 28 days, a block-like structure may be observed, indicating pozzolanic reactions, as demonstrated by identification of needle-shape C-S-H.

Regarding mixture with cement (S7C), block-like structure may be observed already at 7 days. Besides it might be identified the formation needle-shape C-S-H. Shrinkage fissure (due to cement hydration) are observed at 28 days.

The mixture with binary binder, lime and cement (S2L7C), exhibited a combination of the aforementioned observations related to structure, i.e. flaked and block-like. Notwithstanding, in this mixture, C-S-H and ettringite are identified and as well as some shrinkage fissures at 7 and 28 days.

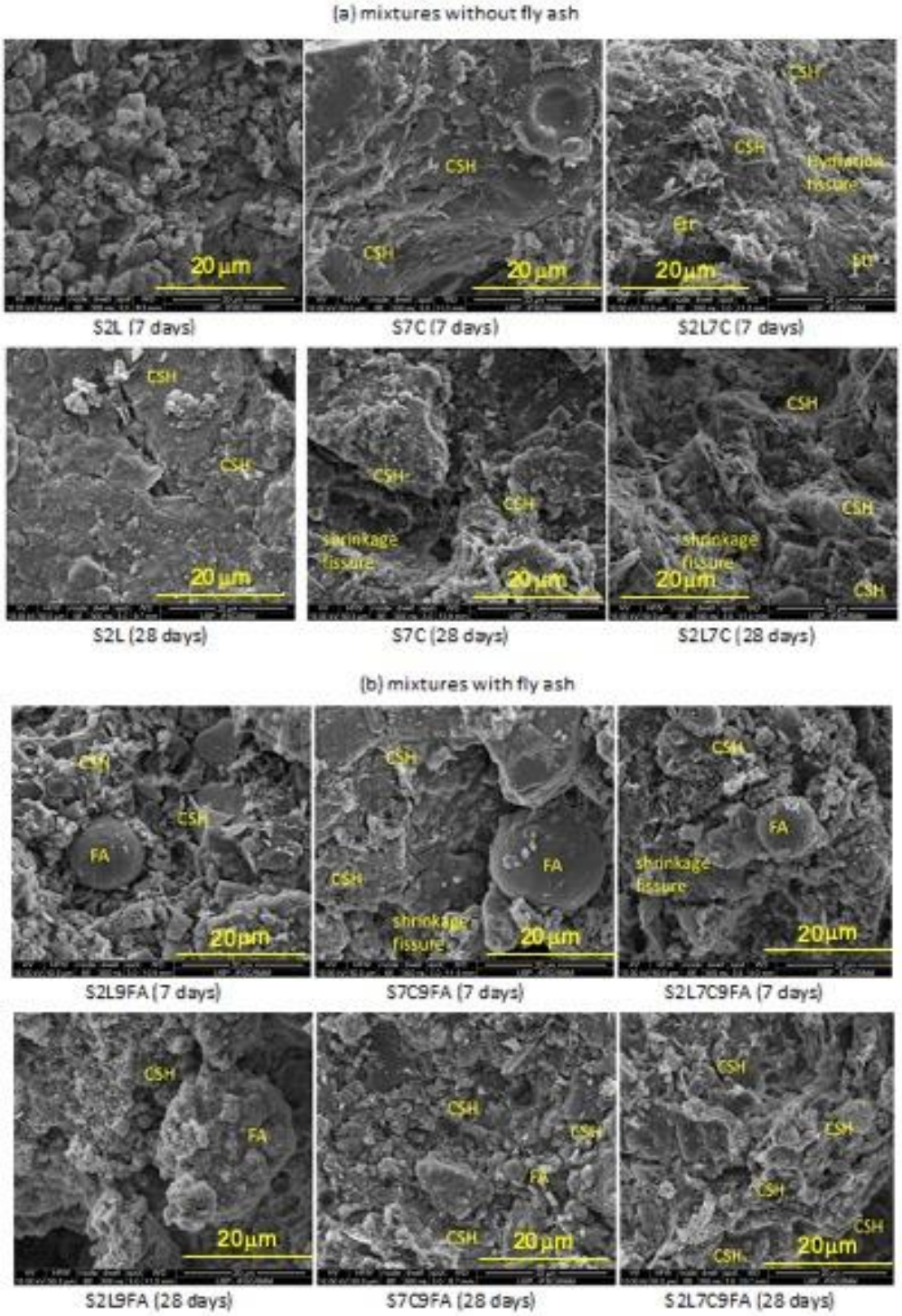

Figure 4: SEM images from mixtures without FA (a) and with FA (b)

The observation of cementation products and the resulting block-like structure lead to infer some increase in strength at macroscale, for instance. Again, that is in accordance with the assumption that cementation enhances the mixture cohesion and controls strength gain of mixtures using cement [12].

Figure 4(b) presents images of binary and ternary mixtures using FA, in attempt to observe its effect when combined with conventional binders. Generally, mixtures using FA revealed significant occurrence of pozzolanic and hydration products. These phenomena are more effective surrounding FA spheres. This finding is consistent to nucleation process and reaction stimulation reported in technical literature [3][12][25].

Concerning visual aspect, at 7 days, it can be observed that mixture with lime and FA shows a flaked-like structure. At 28 days, mixture revealed a blocky structure. The change in structure is remarkable and is attributed to the development of cementation between the soil clusters and also covering FA, as seen in S2L9FA mixture at 28 days.

For mixture with cement and FA (S7C9FA), blocky structure is observed at 7 and 28 days. C-S-H gel covers the surface of soil and FA and shrinkage fissures were identified. For the mixture with lime, cement and FA (S2L9FA), once again the flocculated structure was observed (at 7 days) as well as its evolution to a blockier structure (at 28 days).

Cementitious compounds and hydration fissures seem to occur more frequently in mixtures using FA for both curing time. The increase of CaO content due to addition of FA as long as the nucleation process would promote formation of cementitious materials [3][4][26].

3.1.3 MIP results

The XRD patterns (Figure 2) showed the occurrence of cementation from new crystalline phases in the mixtures such as hydrated silicates. The SEM images (Figures 4) confirmed the presence of the cementitious compound and qualified the changes of the structure caused by the additives. MIP tests were carried out, in attempt to clarify the mechanism of interaction between FA and conventional binder which govern the alteration of sediment fabric.

Figure 5 presents cumulative intrusion versus pore diameter curves. In general, additives reduced the pore diameter of original soil. In mixtures S2L and S2L9FA, for large pores (from 10 to 0.1 µm), it is observed that there are reductions of diameter of pore. Whereas, for small pores (from 0.1 to 0.001 µm) there is an increase of cumulative intrusion, i.e. an increase of percentage of small pores. Curves are approximately parallels, but it can be seen that FA tends to change further large pores.

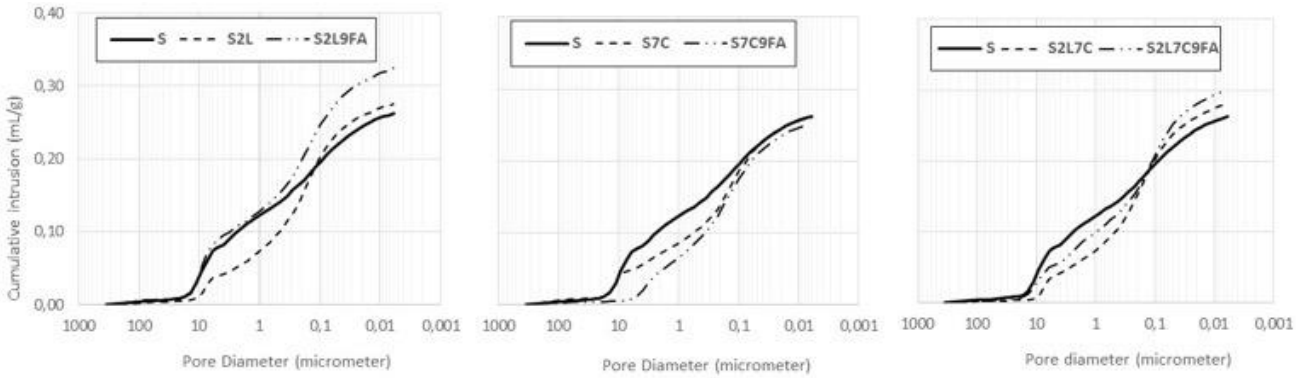

Figure 5: Pore distributions of all mixtures (7 days)

Mixtures S7C and S7C9FA presented significant changes mostly in large pores. This finding suggests that changes in the pore distribution depend on the type and amount of binder added, and particularly the gradation of binders.

Pore distributions revealed the soil fabric changes resulting from addition of binders. For instance, at 7 days, it is expected that:

(i) Lime generates a formation of aggregates/flocculates with finer pore width resulted from the exothermic reaction which take place during lime hydration [10][34].

(ii) Cement hydration produces cementitious compounds that fill large pores and also causes shrinkage reducing original soil pores diameter. No significant evolution of small pores domain is observed for both treatments using lime and cement. This is due to the fact that small pores are mainly associated with intra-aggregate porosity which is less affected by the treatment.

(iii) both lime and cement combine these two mentioned effects, and

(iv) in addition to the role of filler played by FA, it also promotes nucleation and formation of cementitious compounds around FA, increasing the percentage of small pores of the original soil [12].

Since total pore volume (i.e. the highest cumulative intrusion value) is an indicator of material densification, one could assume that, at macroscale, the resistance of mixtures using FA would be higher than that of mixtures without FA. Nevertheless, accordingly [28], cementation controls predominantly the resistance development of soil-cement mixtures.

3.2. Macroscale analysis

Macroscale analysis was done in two steps. The first consisted of immediate property assessment of the mixtures based on Proctor, shrinkage, and UCS test results. The latter was based on UCS results throughout curing time. First and second steps focused on distinguishing the filler effect from curing effect, respectively, in an attempt to remark the most adequate stabilization process, in terms of additive content and strength gain.

3.2.1. Design parameters and immediate strength

Binder addition and content may change soil gradation. Since additives are finer than the soil (Figure 1), they shall fill partially empty voids of soil and increase the initial strength values, due to the filler effect. Table 2 present design parameters and immediate strength. Comparing to untreated sediment, it is observed that binder addition promoted (a) the reduction of maximum dry densities (ρd_{max}) and the increase of optimum water content (w_{opt}), (b) the reduction of the volumetric variation (dV) and (c) the increase of immediate strength (UCS_{0d}). Nevertheless, the intensity of changes depends on the type and the content of binder.

Table 2. Design parameters and immediate strength

			Proctor Parameters		Shrinkage Parameters			
Type of mixture	Legend	% filler	w_{opt} (%)	ρd_{max} (g/cm^3)	Time (days)	dv (%)	dv/T (%/hours)	UCS_{0d} (MPa)
Soil	S	0,0	27.8	1.48	3,0	27.0	0.44	0.28

Soil+ 2% lime	S2L	2.0	32.2	1.38	2.6	20.5	0.50	0.21
Soil+ 2% lime + 9% fly ash	S2L9FA	11.0	32.0	1.37	2.8	17.0	0.39	0.30
Soil+ 7% cement	S7C	7.0	28.1	1.46	2.4	19.0	0.32	0.31
Soil+ 7% cement + 9% FA	S7C9FA	16.0	30.3	1.41	3.0	15.5	0.33	0.37
Soil+ 2% lime+ 7% cement	S2L7C	9.0	30.7	1.43	2.2	18.0	0.25	0.37
Soil+ 2% lime+ 7% cement+9% FA	S2L7C9FA	18.0	31.6	1.37	2.0	12.5	0.17	0.49

To illustrate filler effect, Figure 6 shows correlations between Proctor and shrinkage parameters, and immediate UCS values in function of additive content. Figure 6(a) shows that the reduction of ρd_{max} is a result of the increase of binder content, but this behavior depends on the type of binder considered. It is observed that the evolution of the ρd_{max} follow a linear regression law with high degree of accuracy (R^2). R^2 values for mixtures containing lime (L) and cement (C) are 0.81 and 0.88, respectively. FA addition reduced ρd_{max} in mixtures with cement. In mixtures with lime this behavior was not observed.

Linear law demonstrates that cement addition reduces maximum dry density less than lime addition. Both curves converge to the ρd_{max} value of the SLCFA mixture, which was the minimum one.

Volumetric deformation is often consequence of shrinkage which occurs in cemented materials due to the water consumption for clinker component, generating fissures. Shrinkage test reports the variation of volume (dV) that a soil-cement may present and gives an idea about material durability in dosage phase. Figure 6(b) shows a strong correlation between dV and additive content (R^2=0.93), indicating that dV decreases with increasing additive content. In other words, partial filling of the voids would inhibit shrinkage.

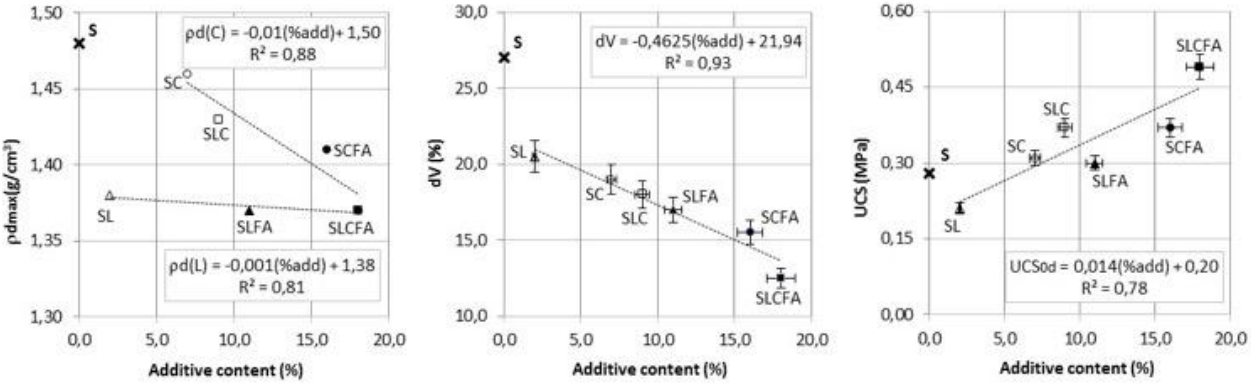

Figure 6: Immediate Properties and characteristics of mixtures

It would be reasonable to indicate the addition of inactive fillers in order to reduce shrinkage in soil-cement type mixtures. Figure 6(b) led to infer that FA plays this role in this phase, because its addition in the mixtures further reduced dV. In mixtures without FA, the reduction of dV was approximately equals to 30% in relation to untreated soil. When FA was added, this reduction was about 40% for SL and SC mixtures, and 55% for SLC mixture.

Regarding the filler effect on immediate strength, Figure 6(c) shows a good correlation (R^2=0.79) between additive content and UCS of the mixtures, where the higher the additive content the greater the UCS_{0d}. It is important to point out that immediate UCS_{0d} of SL mixture was lower than the untreated sediment. This behavior is attributed to flocculation reactions of lime that increase of voids in the mixture, decreasing ρd_{max} and increasing water content (as seen in Table 2).

It is worth emphasizing that the combination of additives changes gradation of untreated sediment. As additives are finer than the sediment soil, they may fill empty voids, increasing the sediment strength. As a rule, the initial strength values gradually improve as binder percentages increase.

In spite of the good correlation between immediate UCS and additive content, the strength gain may be also related to the type of additive, or combination between them, since the greatest immediate UCS were observed in mixtures SLC, SCFA and SLCFA. The common point of these mixtures is the presence of cement associated to other(s) additive(s). It seems to be a contribution of additive gradations to the particle size arrangement of mixtures, resulting in the increase of their immediate UCS. Immediate UCS is a reference point that might lead to dosage optimization attempts. Accordingly, from this point on, it shall be observed the strength gain over time due to chemical reactions.

3.2.2. UCS and water content results over time

In order to highlight the effects of additives on the mechanical resistance of mixtures, Figure 7 presents the kinetics of the UCS over time. Regarding compressive strength values (UCS), one may observe that additives increased initial property of untreated sediment (0,27 MPa), even though some mixtures

required more time to present significant strength gains, such as mixture S2L that presented reduction of UCS value at 7 days. All other additives combination increased UCS values and these increases are more expressive when FA is added.

From 0 to 7 days, the strength gain of S2L and S7C was about 0.05 MPa that represents respectively 20% and 16% of their initial UCS values (0.21 and 0.31 MPa). For conventional binders, the mixture with lime and cement (S2L7C) had the higher strength gain (~0.15 MPa), that is 40% of its initial strength (0.37 MPa).

For mixtures containing FA, strength gains were in average of 0.15 MPa. It is worth emphasizing the strength gain of the mixture S2L9FA that was about 70%. In contrast, the strength gain at 7 days of the mixture S2L7C9FA was the lowest one (0.10MPa) that represents about 20% of initial UCS value.

At 28 days, the UCS value of S2L was 0.31 MPa, S7C was 0.40 MPa, and S2L7C was 0.87 MPa. From 7 to 28 days, the gain of UCS remains about 0.05 MPa for S2L and S7C. Interestingly, FA addiction doubled the strength gains in these mixtures. The faster strength gain was also observed by Kang et al. [27].

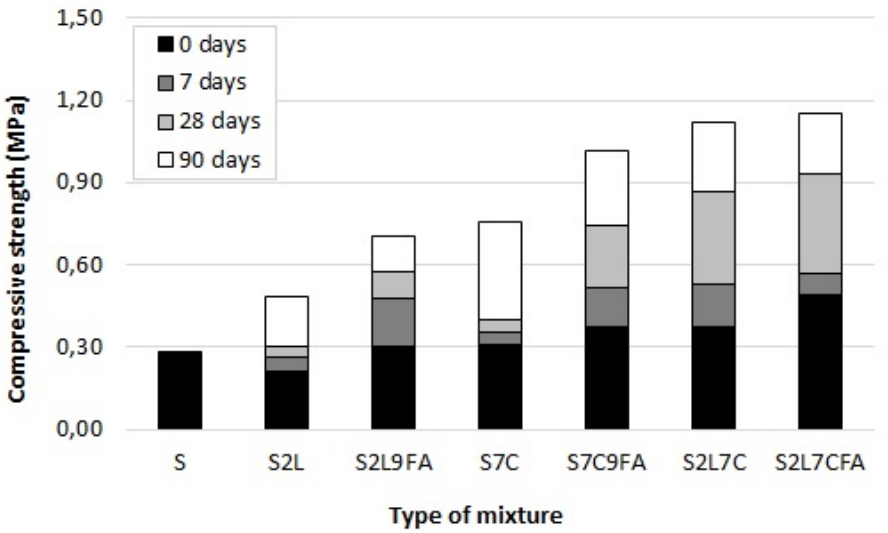

Figure 7: UCS over time

For the mixture S2L7C9, the strength gain was 0.35 MPa. In this case, FA addition did significantly not increase strength gain of SLC mixture. In general, mixtures with FA presented the higher UCS values.

In order to observe the tendency of strength evolution, Figure 7 also presents UCS at 90 days. As it can be noticed, UCS values continued increasing, with emphasis on the highest strength gains of mixture S7C and S2L. Another point to remark is that mixtures using FA showed slower strength gains compared to mixtures with conventional binders, unlike what was observed for other curing time (7 and 28 days), suggesting a deceleration of these gains at 90 days.

UCS Kinetics confirmed the additive mechanism-based assumptions. Regarding mixture with lime, cation exchanges and flocculation did not improve soil resistance. Over time, pozzolanic reactions were not effective to increase significantly the strength even in long term (from 28 days). In the mixture with cement, the occurrence of cementitious compounds increased strength at early age (7 days). Interestingly, it continued to increase up 90 days but at lower intensities, in contrast to expectations of fast reacting cements. This delay is probably associated to the salt presence and organic matter in the marine sediment that alters the kinetics of hydration cement, as mentioned by Horpibulsuk et al. [35].

On the other hand, FA addition has led to the higher strength gains in all tested mixtures. This observation is consistent with the hypotheses of nucleation and growth of cementitious materials promoted by FA [15][26][27], which here were observed and discussed based on the results of microscale test (Figures 2 to 4).

Assuming that the gain of resistance over time results from the cementing products of the additive components [28], it is also necessary to understand how the hydric state of the mixtures evolves. Figure 8 presents the evolution of water content over time. The plotted curves indicate that water content reduces over time and the intervals of variation are significant over long periods, as can be seen for all mixtures using cement.

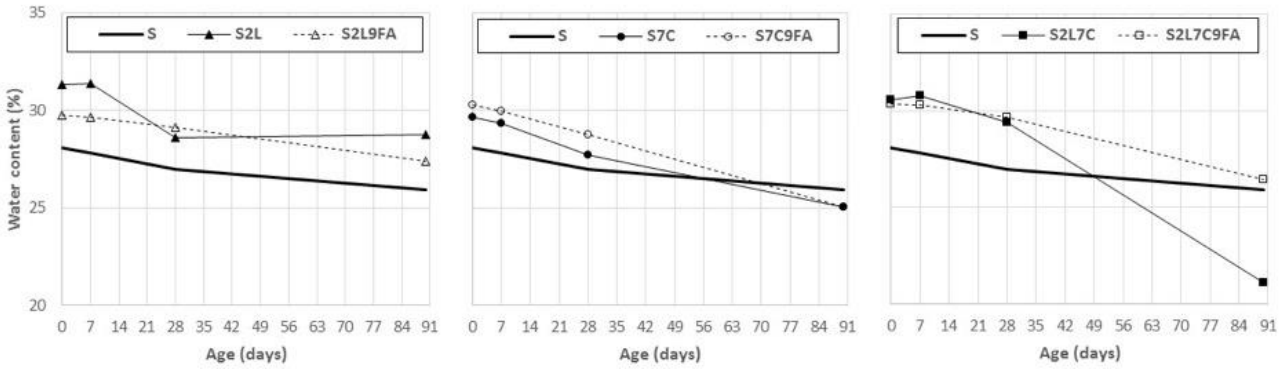

Figure 8: Variation of water content over time

In early age (7 days), water contents change slightly, maintaining practically the initial values, i.e. close to the w_{opt} (Table 4). However, for intermediary age (28 days), these changes evidence the reduction of water content, the average reduction was 5%. For longer period (90 days), the average reduction of water content is 12%, except for SLC, which was 31%.

FA addition also alters hydric conditions of stabilized mixtures over time but at lower intensity. As a rule, one may suppose that the greater the decrease in moisture content the greater the gain in resistance. This hypothesis is true since water is consumed in formation and/or crystallization of cementing products, becoming the structure denser and more resistant than the original soil.

4. Discussion

The decision for a given product takes into account technical, economic and environmental parameters. This study has shown that combining FA with stabilized soil with lime and cement (or both) is promising because of the FA action mechanisms promoted an acceleration of strength gain regardless of the curing time. Besides, mixtures using FA have always shown higher resistance, either through physical interactions or chemical reactions. These findings support technically the choice for combining FA with chemical soil stabilization using conventional binders.

For instance, UCS value at 90 days of mixture using cement (S7C) is compatible with that of the mixture using cement and FA (S7C9FA) at 28 days, or with mixture using lime and FA (S2L9FA) at 90 days. These mechanical compatibilities indicate alternative material options that might save time and economy,

since it is possible to select mixtures that present greater resistance ahead of time, use cheaper additives or smaller quantities of conventional binders.

These benefits have a positive impact on environmental indicators because they promote the rational use of waste and byproducts (marine sediment and fly ash). In this sense, it is worth mentioning that the mixture with lime, cement and FA (S2L7C9FA) presented resistance compatible with the mixture without FA (SLC), leading to question the advantages of this mixture (S2L7C9FA) as well as to think alternatively about the possibility of reducing the cement content in order to optimize the mixture design in terms of economic and environmental.

5. Conclusions

The effects of FA addition on microstructural and resistance characteristics of mixtures stabilized with conventional binders were investigated. From this study the conclusions are made focusing the interrelations between microstructural and macrostructural analyses.

Regarding microstructural analyses, it was identified new crystalline phases in DRX patterns. Cementitious materials, such as ettringite, portlandite and hydrated silicates, are produced over time by cement hydration and pozzolanic reactions. The occurrence of these products depends on the curing time due to particular binder reaction rate. These findings are supported by SEM images, that confirmed the presence of hydrated silicates in different morphologies (gel, acicular C-S-H for instance), mainly in mixtures using FA. The SEM images also show physical modification of the soil fabric over time, which was also confirmed by analysis of pore distributions. The results of the pore distribution measurements show a structural densification of the sediment matrix at early age, demonstrating the contribution of finer gradations of the additives on the sediment pore rearrangement, i.e. filler effect.

Macroscale analyses led also to observe a prevalent filler effect on gain of immediate resistance and reduction of volumetric variation by shrinkage (dV). These physical responses are especially remarkable in mixtures using FA. These findings led to recommend more studies in dosage parameters

and properties in order to identify the best combination of binder blend for chemical stabilization purposes and to optimize the mixture design with several binders, i.e. the choice and the content of binder(s). UCS values increase and water contents decrease over curing time, with emphasis on the significant contribution of FA to the better mechanical performance. Based on microstructural analysis, it can be stated that available water was consumed for cementing and pozzolanic reactions over time.

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Parameters	Value
Sand fraction > 63µm (%)	23
Silt fraction - 2 to 63µm(%)	41
Clay fraction < 2µm (%)	36
Specific gravity (g/cm^3)	2.66
Plasticity limit (%)	36.06
Liquidity Limit (%)	54.54
Plasticity Index	18.48
Organic matter content (%)	10.97
Carbonates content (%)	22.21
pH	8.5

Table 1: Geotechnical properties of sediment

			Proctor Parameters		Shrinkage Parameters			
Type of mixture	Legend	% filler	w_{opt} (%)	ρd_{max} (g/cm^3)	Time (days)	dv (%)	dv/T (%/hours)	UCS_{0d} (MPa)
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Soil+ 2% lime	S2L	2.0	32.2	1.38	2.6	20.5	0.50	0.21
Soil+ 2% lime + 9% fly ash	S2L9FA	11.0	32.0	1.37	2.8	17.0	0.39	0.30
Soil+ 7% cement	S7C	7.0	28.1	1.46	2.4	19.0	0.32	0.31
Soil+ 7% cement + 9% FA	S7C9FA	16.0	30.3	1.41	3.0	15.5	0.33	0.37
Soil+ 2% lime+ 7% cement	S2L7C	9.0	30.7	1.43	2.2	18.0	0.25	0.37
Soil+ 2% lime+ 7% cement+9% FA	S2L7C9FA	18.0	31.6	1.37	2.0	12.5	0.17	0.49

Table 2: Design parameters and immediate strength

Figure 1

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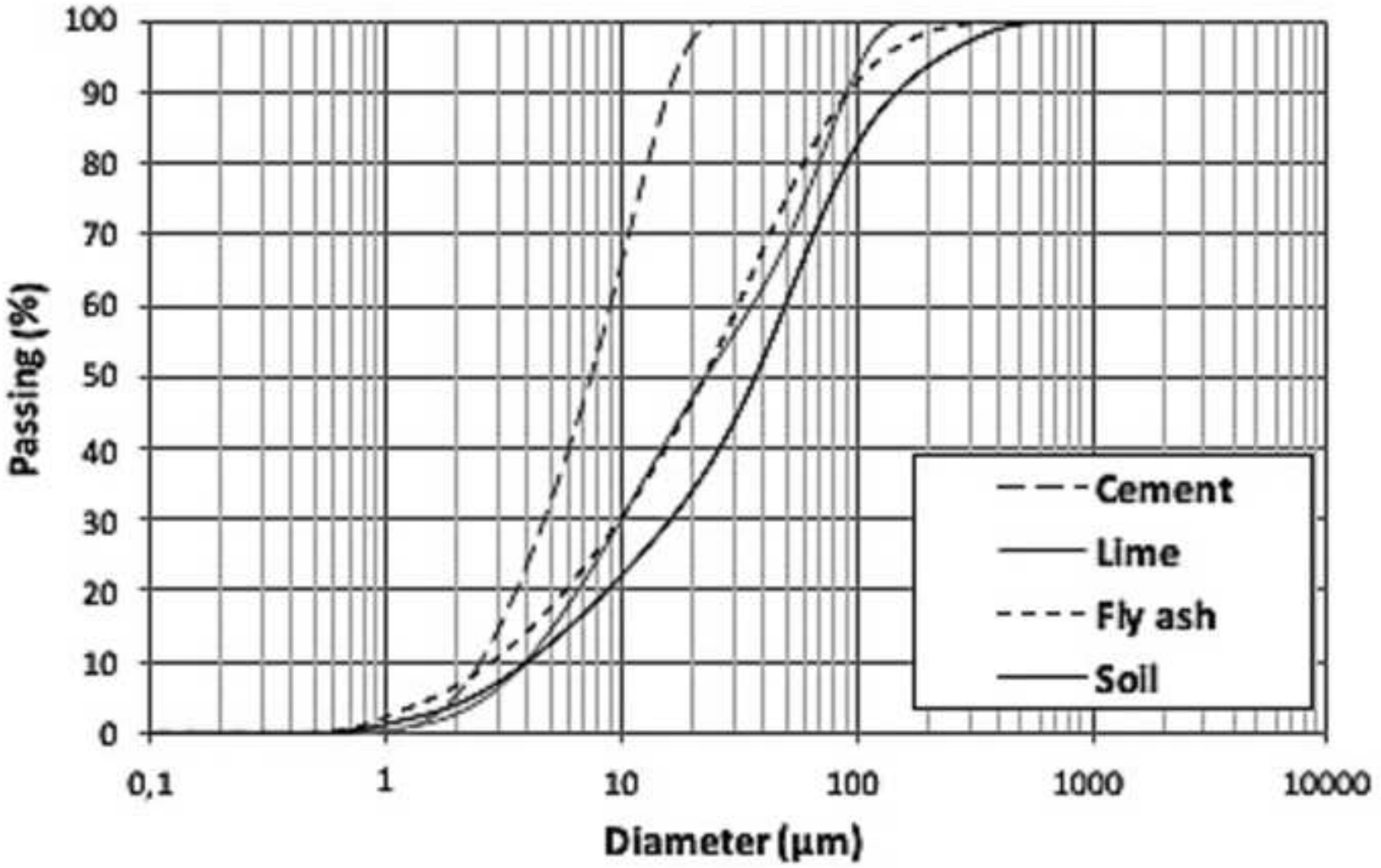

Figure 2

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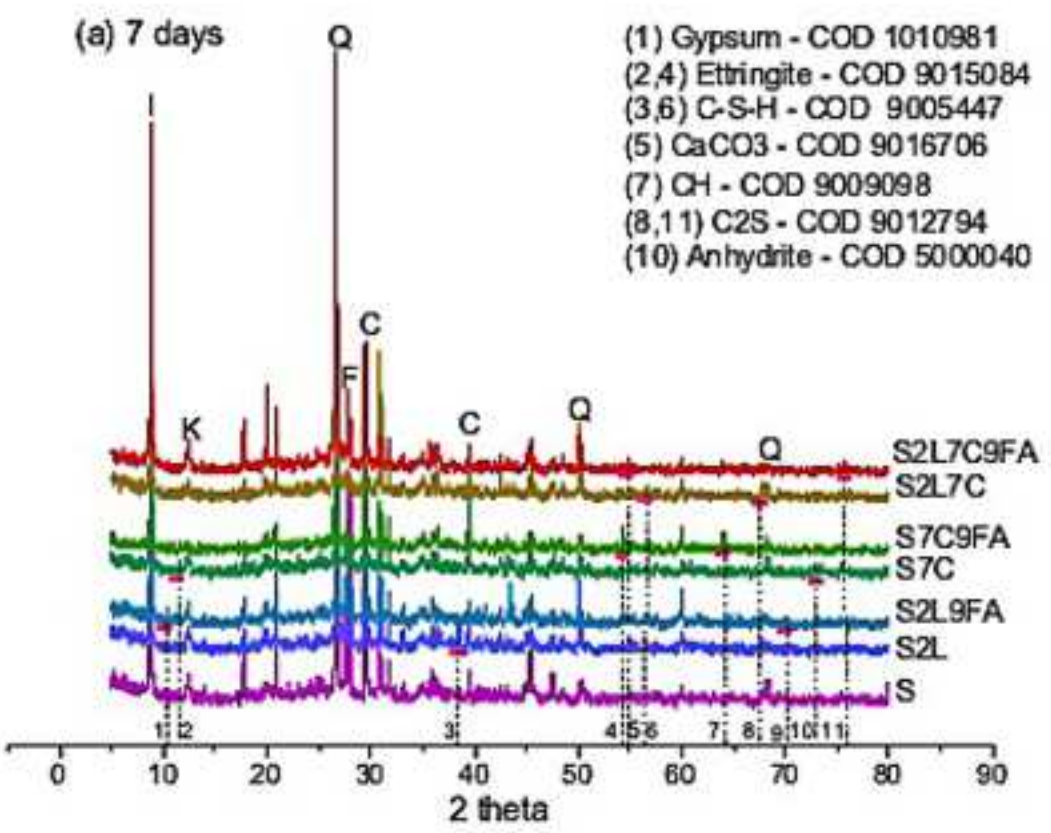

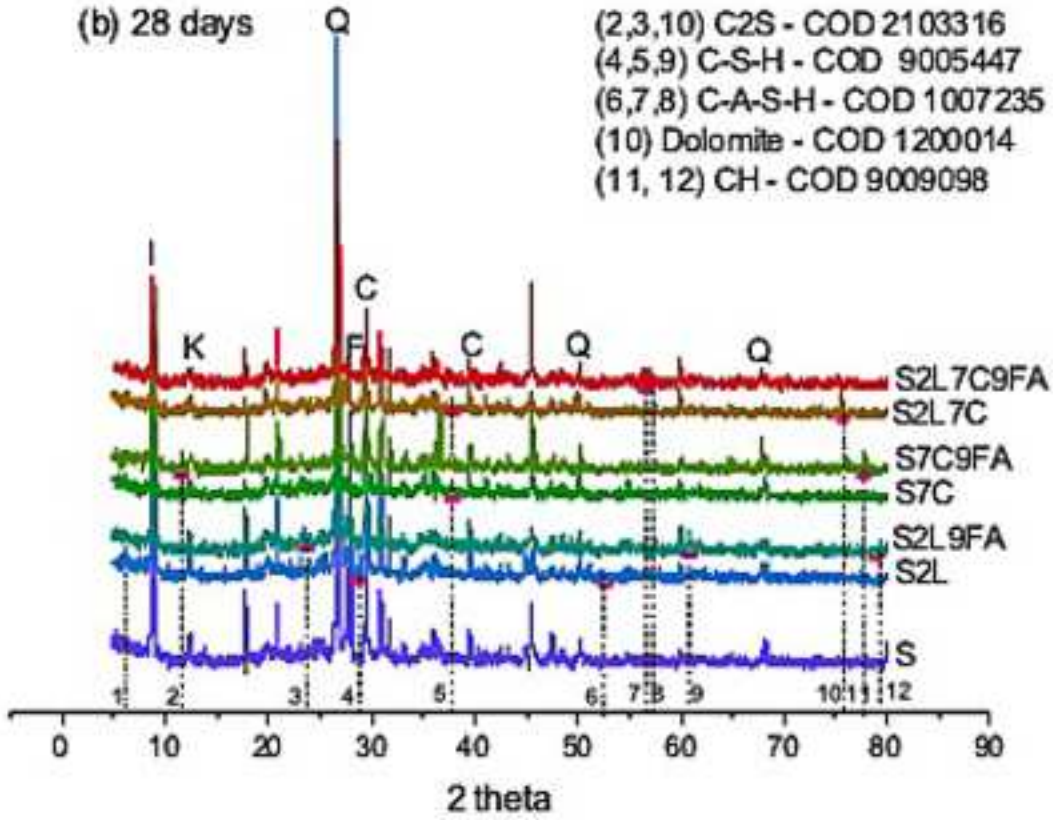

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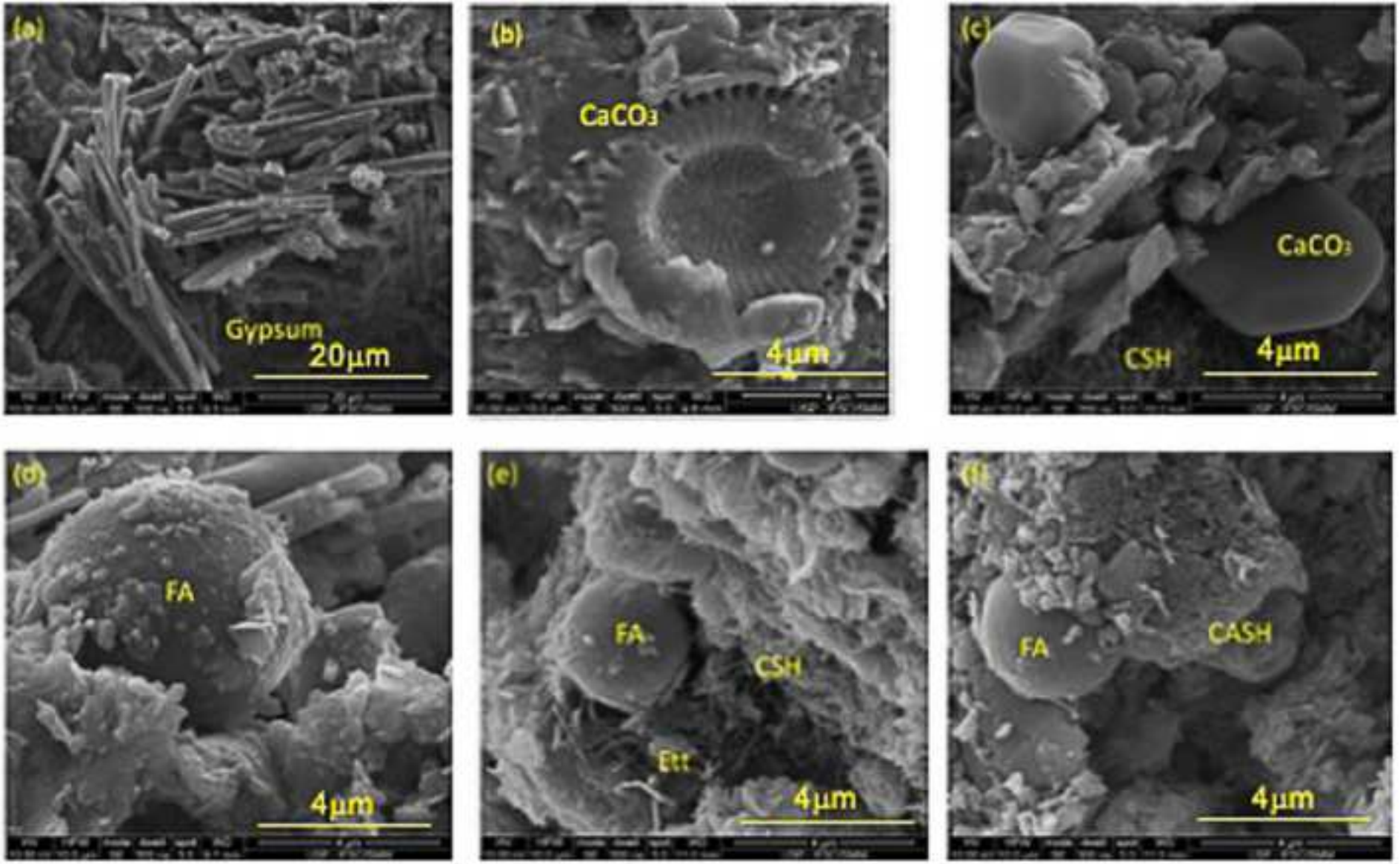

Figure 4

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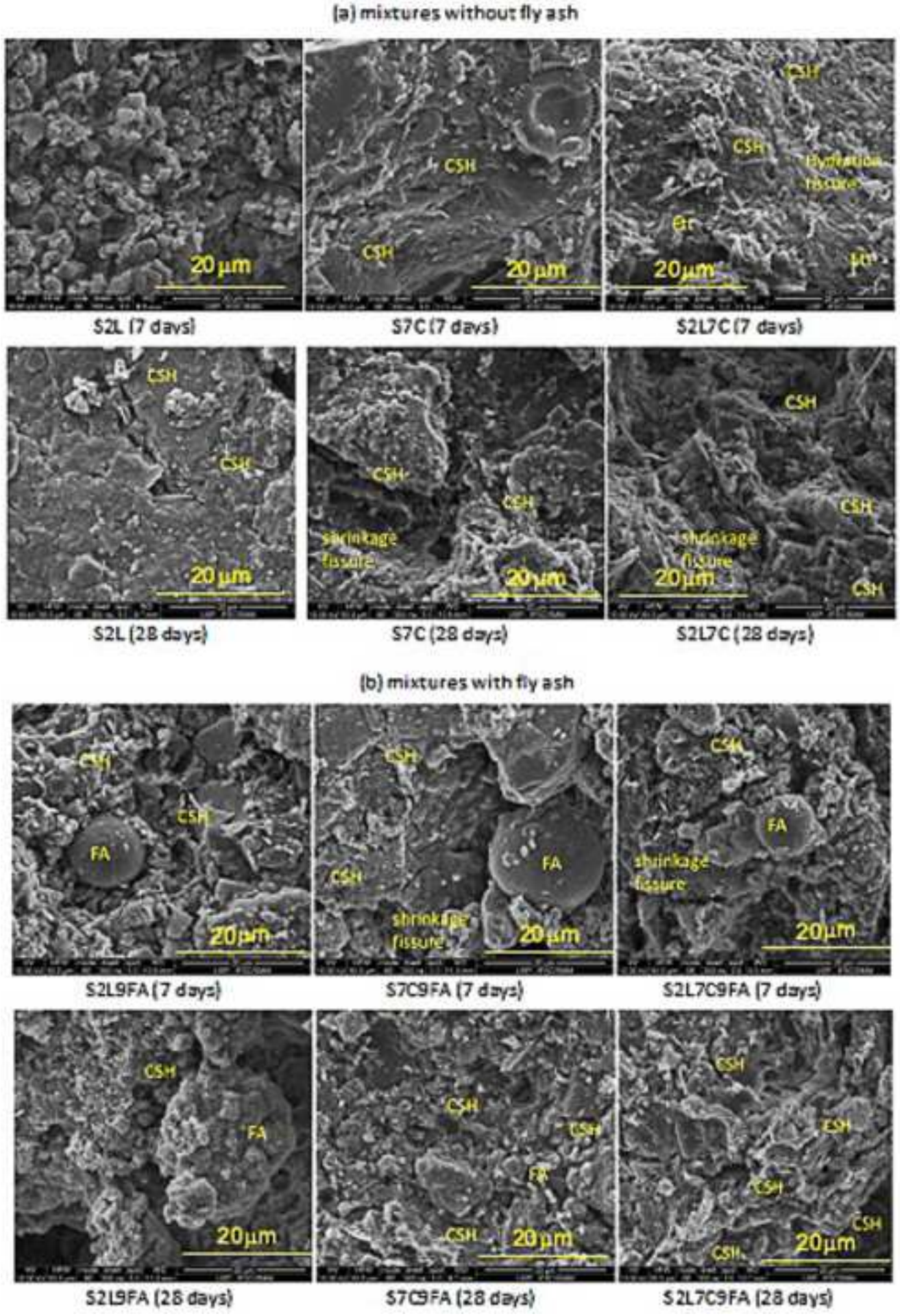

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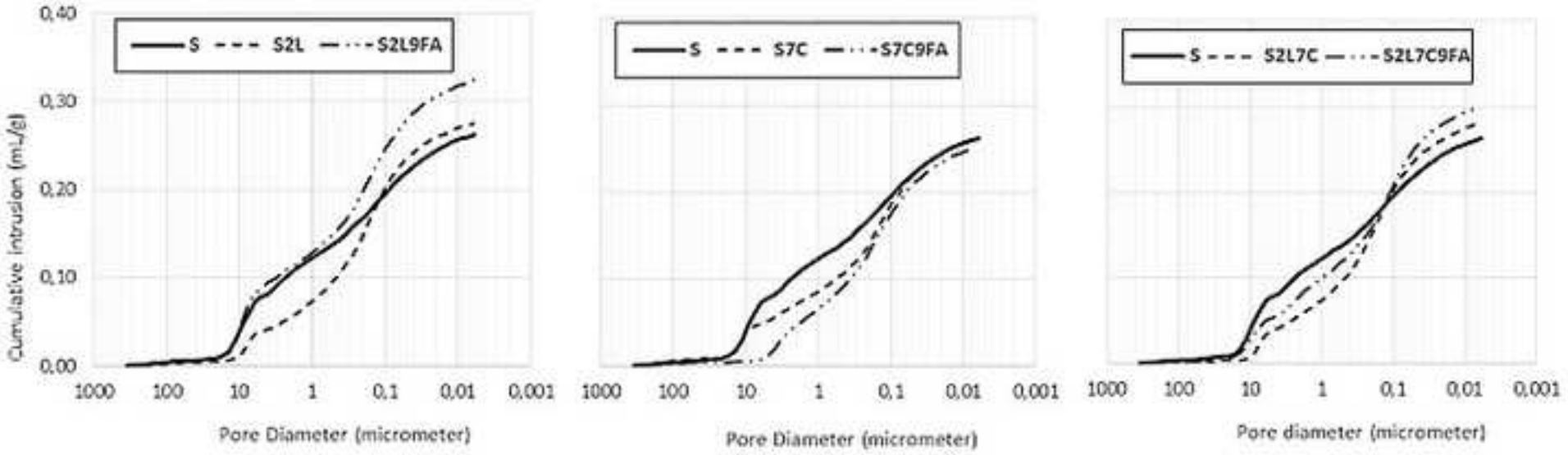

Figure 6

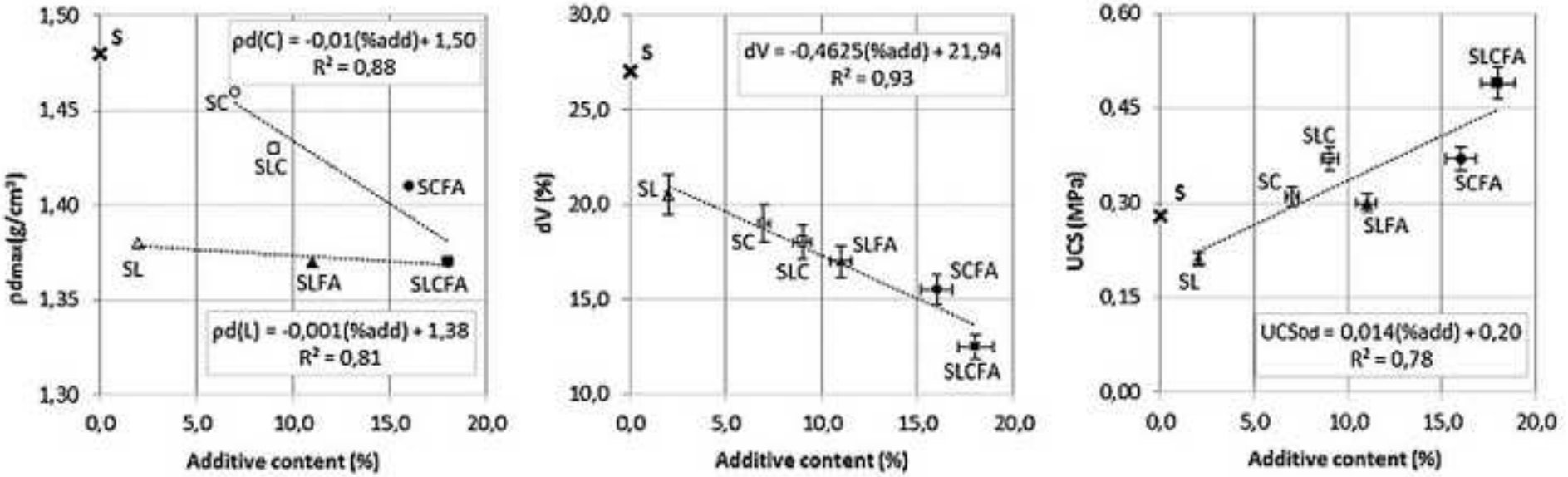

Figure 7

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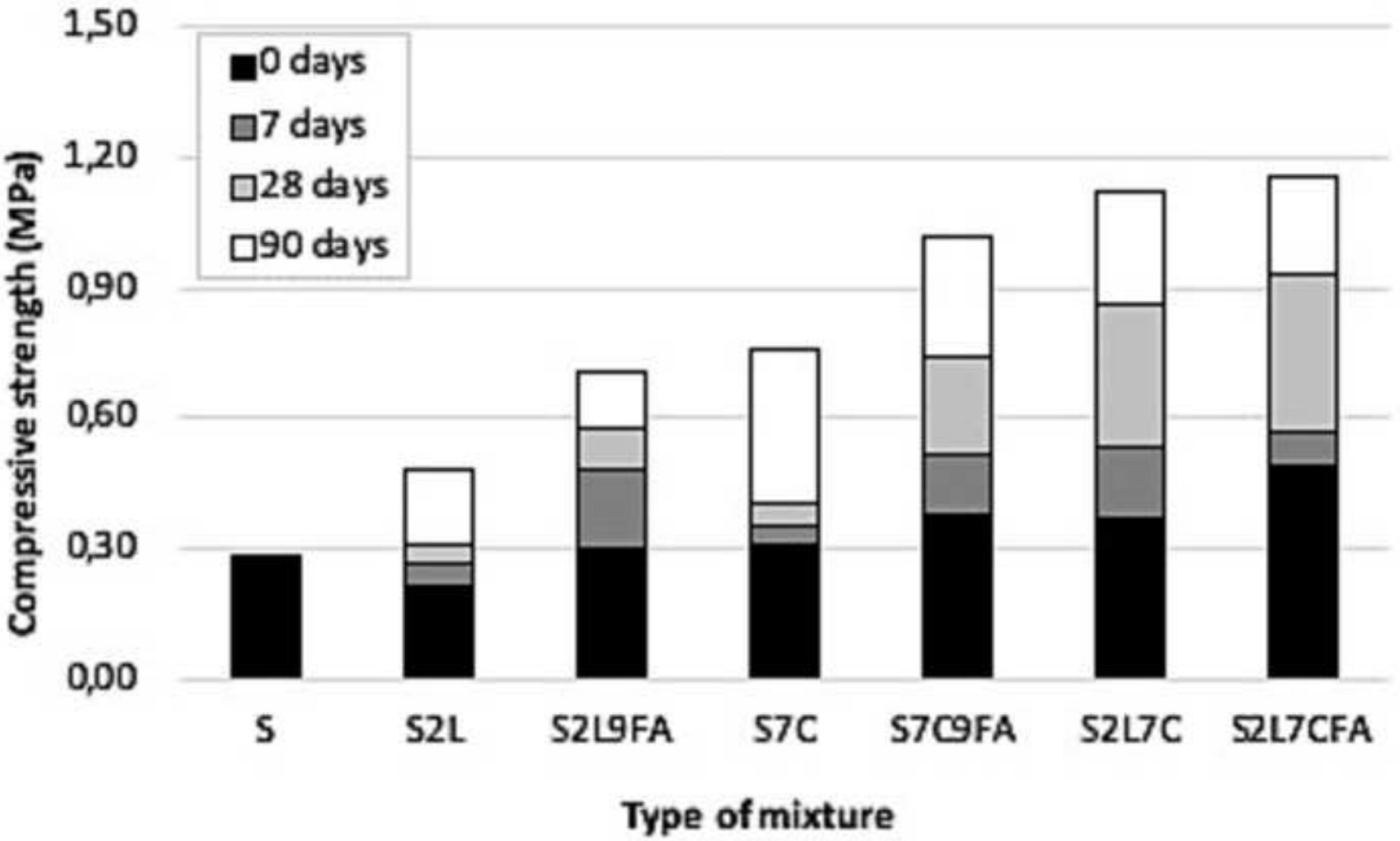
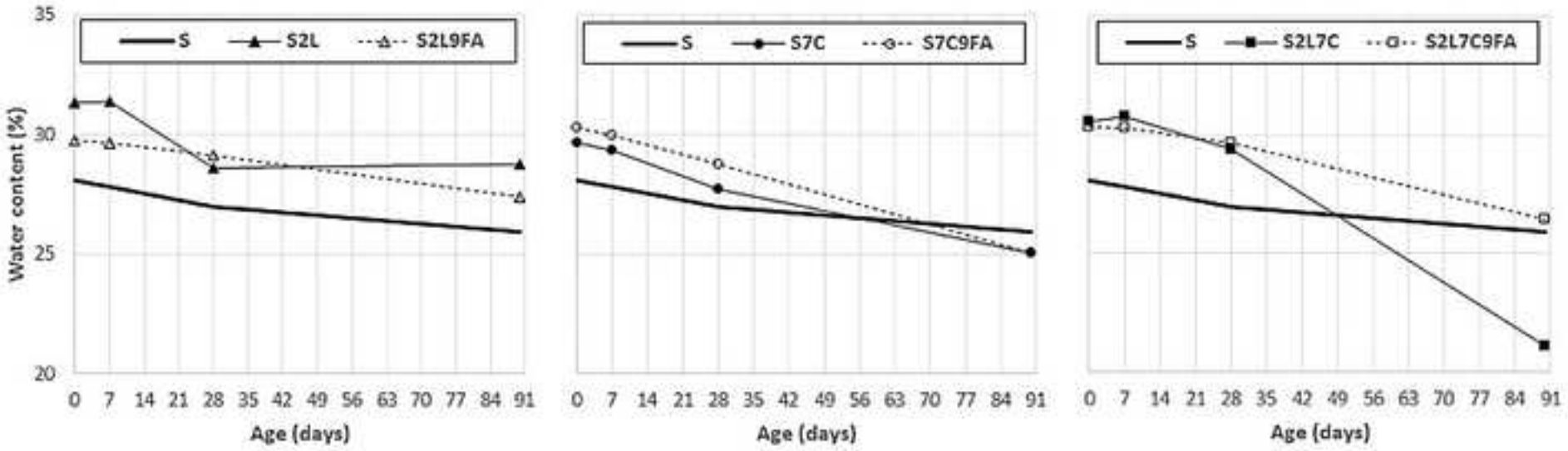


Figure 8

Click here to access/download;Figure;Fig-8.JPG



Effect of FA addition on microstructural and resistance characteristics of a stabilized soil using lime and cement

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Graphic Abstract