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Effects of additives on the microstructure of clay

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Some soils under construction cause numerous problems in terms of geotechnical engineering. Clay soils cause significant problems in the construction of roads, airports, pavements and highways. Some soils contain mixed additives, such as lime, cement, fly ash and bitumen. In this study, the microstructures of uncured and cured lime/cement-stabilised clay samples are investigated. Compacted soil samples were evaluated by mercury intrusion porosimetry, X-ray diffraction, scanning electron microscopy (SEM) analyses and specific surface area analysis. The results indicated that the addition of lime and cement was effective in the treatment of compaction properties. The pore sizes in the SEM images vary with an increase in the percentage of lime and cement and an increase in curing time.

Keywords: soil improvement; lime; cement; pore size distribution; microstructure

Introduction

Clay soils are often observed in geotechnical engineering practice. Generally, these soils have numerous problems because of their low strength, high compressibility and high volumetric changes. Clay soils need to be improved before they can be used in roads, airports, pavements and highways. Improved soil gradation, a reduction in the plasticity and swelling potential of soil and an increase in the strength and workability of soil generally improve the stabilisation of soil (Attom, Taqieddin, & Mubeideen, 2000; Sherwood, 1993). Stabilisation is applied via mechanical or chemical methods. In cases of insufficient mechanical stability, chemical stabilisation is used. To improve the strength, durability and compressive characteristics of soil, soil should be replaced or improved by stabilisation using additives. Soil stabilisation has been achieved by mixing additives, such as cement, lime, asphalt and fly ash, with soil. Soil stabilisation with additives comprises the oldest and most common method of soil improvement. Known applications date back as far back as ancient Greek, Egyptian and Roman times (Fang, 1990). Cement and lime stabilisations are highly prevalent throughout the world. Cementation and ion exchange reactions occur in stabilised soils. The chemical and mechanical properties of soils change during this chemical reaction. Previous studies have focused on the geotechnical behaviours of improved soils (Al-Rawas, Hago, & Al-Sarmi, 2005; Cuisinier, Auriol, Borgne, & Deneele, 2011; Davoudi & Kabir, 2011; Galvão, Brito, Elsharief, & Simões, 2004; Ganjidoust, Hassani, & Ashkiki, 2009; Ho & Chan, 2011; Jauberthie, Rendell, Rangeard, & Molez, 2010; Millogo, Morel, Traore, & Ouedraogo, 2012; Oyediran & Kalejaiye, 2011). Lime and cement are effective additives to mix with fine-grained soils. Cement or lime can change the properties of clays by cation exchange, flocculation and agglomeration, cementitious hydration and pozzolanic

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reactions (Prusinski & Bhattacharja, 1999). Cation exchange occurs within a short period of time, whereas pozzolanic reactions occur within a longer period of time. Cation exchange and pozzolanic reactions are observed in stabilised clays. Reactions in soil and lime may have reaction products similar to that of the reactions in soil and cement, but the difference between the reactions of soil–cement and of soil–lime is that considerably more time is required for reactions of soil–cement for the curing conditions. The soil–additive (lime or cement) reaction can create products such as hydrated calcium silicate (CS), hydrated calcium aluminates, hydrated calcium aluminate silicate (CAS) and calcium magnesium carbonate (CMC) (Bell, 1996; Brown, 1996; Chen, 1975; Raj, 1999; Saitoh, Suzuki, & Shirai, 1985). Several authors have investigated the relationship between the microstructures of improved soils and the geotechnical engineering behaviours of improved soils (Horpibulsak, Rachan, Chinkulkijiniwat, & Raksachon, 2010; Metelková, Boháč, Sedlářová, & Přikryl, 2011; Muhmed & Wanatowski, 2013; Onitsuka, Modmoltin, & Kouno, 2001; Russo, Vecchio, & Mascolo, 2007; Sante, Fratalocchi, Mazzieri, & Pasqualini, 2014).

Rajasekaran, Murali, and Srinivasaraghavan (1995) examined the influence of lime and sodium hydroxide on the microchanges in two marine clays by scanning electron microscopy (SEM). They observed that the addition of lime and sodium hydroxide creates an optimal pozzolanic reaction. They also investigated the results of X-ray diffraction (XRD) and reported the formation of cementitious compounds. Horpibulsak et al. (2010) examined the strength development in cement-stabilised silty clay with microstructural changes. They conducted SEM, mercury intrusion and thermal gravity analyses for qualitative and quantitative analyses of the microstructure of samples. They noted that the volume of large pores increases due to the presence of coarser particles in a short stabilisation period, whereas the volume of small pores decreases due to the solidification of the hydrated cement. Because the large pores are filled with cementitious products, the small pore volume increases with time and the total pore volume decreases with time.

A review of recent literature indicates that the majority of tests of soil microstructure and mineral composition are conducted to develop an improved understanding of the mechanical properties of soil (Cui & Tang, 2011; Dananaj, Frankovska, & Janotk, 2005; Low, Phoon, Tan, & Leroueil, 2008; Prashant & Penumadu, 2007; Robinson & Allam, 1998; Yamamuro &Wood, 2004). The lime and cement stabilisation of clay with different aspects was examined. In this study was investigated on the relationship between microstructures and the geotechnical engineering behaviors of improved soils. Few studies have evaluated the pore size distribution for improved soil. This study examines the changes in the pore size distribution for stabilised clay with cement and lime. Stabilised clay with cement and lime was compacted in a standard compacted test. Samples were cured in a desiccator. The pore size distribution of compacted uncured soil and different curing times for stabilised soils were investigated. The relationships are demonstrated using the results of a standard compaction test, XRD, mercury intrusion porosimetry (MIP), SEM analyses and specific surface area (SSA) analysis which were performed on all stabilised soil samples.

Materials and methods

Materials

The grain size distribution of the clay in this study is listed in Figure 1. The soil is a high plasticity clay (CH). The index properties of soil are listed in Table 1. The mineralogical composition of clay is listed in Figure 2. The stabiliser materials consisted of lime and cement that were obtained from commercial firms in Turkey. Industrial lime was used for the stabilisation. The cement was

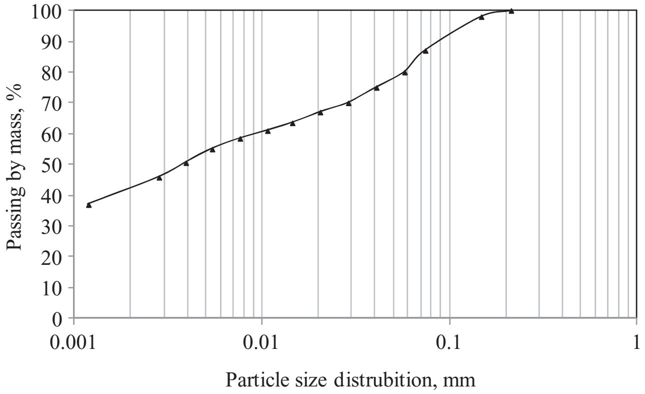

Figure 1. Grain size distribution of clay.

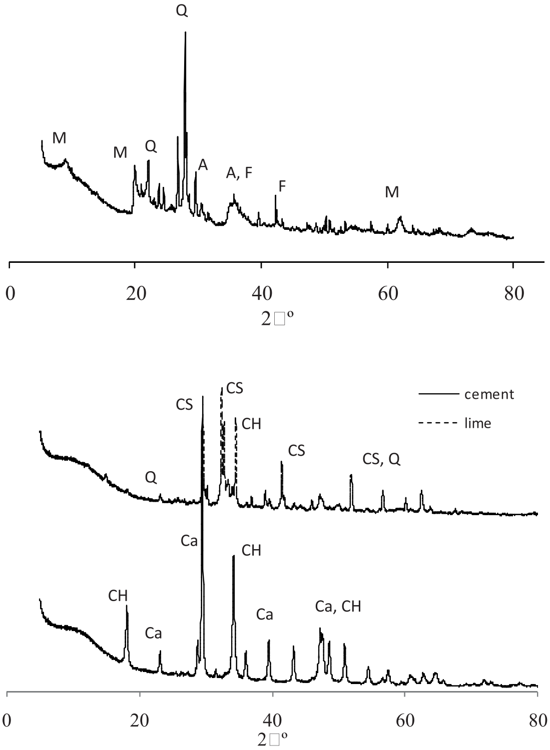

Figure 2. XRD patterns of clay, lime and cement (M: montmorillonite; A: anorthite; F: feldspar; Q: quartz; Ca: calsite; CH: calcium hydroxide; CS: calcium silicate).

Table 1. Geotechnical index properties of clay.

Mixtures	LL[a]	I_p	Gs
Clay	103	65	2.66

Note: LL: liquid limit; GS: specific gravity. [a]Fall cone method.

Table 2. Names of additional lime or cement and results of standard compaction.

Mixtures	Name of additional lime or cement	OWC	γ_{dmax} (kN/m^3)
C0L/C0C	0	27	14.30
C2L	2	28	13.86
C4L	4	30	13.23
C6L	6	32	12.85
C8L	8	35	12.05
C2C	2	25	13.70
C4C	4	23	13.80
C6C	6	21	13.98
C8C	8	18	14.38

Note: OWC: optimum water content. γ_{dmax}: maximum dry density.

classified as a CEMI. The mineralogical compositions of lime and cement are shown in Figure 2. The mineralogical composition of the lime included calcium carbonate, and the mineralogical composition of the cement included was aluminous.

Sample preparations

In all, 0%, 2%, 4%, 6% and 8% of the soil were replaced with lime and cement. The soil–lime and soil–cement mixtures are referred to by the following designations: C0L/C0C, C2L/C2C, C4L/C4C, C6L/C6C and C8L/C8C. For each mixture, at least three samples were tested to ensure the reproducibility of the data. The soil and the C0L/C0C, C2L/C2C, C4L/C4C, C6L/C6C and C8L/C8C mixture samples were prepared by standard compaction tests according to ASTM D698 (2000). The standard compaction tests were performed to obtain the optimum water content (OWC) and the maximum dry unit weight (γ_{dmax}) values of the soil and the C0L/C0C, C2L/C2C, C4L/C4C, C6L/C6C and C8L/C8C mixture samples. The descriptions of the soil–lime and soil–cement mixtures are listed in Table 2.

Standard compaction characteristics

OWC and γ_{dmax} values of the mixtures were determined by a standard compaction test according to ASTM D698 (2000). The OWC and γ_{dmax} values of the soil and the C0L/C0C, C2L/C2C, C4L/C4C, C6L/C6C and C8L/C8C mixture samples are listed in Table 2. The γ_{dmax} values of the samples substantially decreased with the percentage of lime, whereas the OWC increased with the percentage of lime. After cement was added on to clay, the γ_{dmax} values of the samples decreased. However, comparing the mixtures, the γ_{dmax} values of the samples substantially increased with the percentage of cement, whereas the OWC decreased with the percentage of cement. The samples were cured for 0, 14 and 28 days and subsequently analysed using XRD, MIP and SEM. In addition, SSA was determined on uncured soil sample.

X-Ray diffraction

Structural analyses of the clay and additive mixtures were carried out by XRD (PAN analytical) in the range 10–80° 2θ using a Cu Kα radiation source ($\lambda = 1.5405$ Å). The mineralogical composition of the soils is critical to its significant influence on soil behaviour. The XRD patterns of the clay revealed the presence of montmorillonite, anorthite and quartz (Figure 2). The XRD patterns of the lime and cement revealed the presence of calcite and silica (Figure 2).

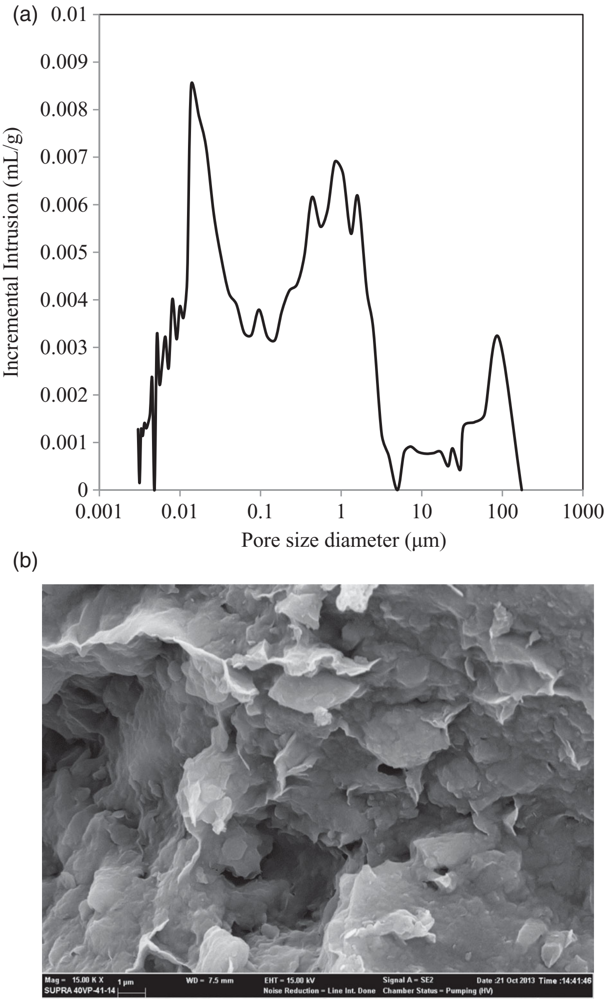

Figure 3. (a) Pore size distribution graph of clay from the MIP. (b) SEM images of clay samples for × 15,000 magnification.

Mercury intrusion porosimetry

The pore size distributions for the clay and additive mixtures were also measured by MIP. Mercury porosimetry was performed from 0.34 to 413 MPa, and the measured internal porosities were in the range of 3 nm and 900 μm. In geotechnical engineering, pore size distribution can significantly influence the geotechnical behaviour of a soil. The pore size distributions for the clay from the MIP tests are displayed in Figure 3(a).

Scanning electron microscopy

The microstructures of the clay and the additive mixtures were analysed using SEM. The Eksta High Tension voltage and Working Distance were 15 kV and 12 mm, respectively. The microstructural changes in the soils due to the addition of different materials serve a significant role in the geotechnical properties of soils. In this study, × 15,000 magnification was selected for the SEM images. The SEM images of clay are presented in Figure 3(b).

Specific surface area

The SSA of the clay and additive mixtures was evaluated by the Brunauer–Emmett–Teller model and the surface area measuring range of the device ranged from 0.005 to 5000 m^2/g. The SSA is affected by the physical and chemical properties of soils. The SSA of clay was 36.91 m^2/g.

Results and discussion

The size of the grains in the soil and the water in the pores can affect the geotechnical properties of soils. In this study XRD, MIP, SEM and SSA were conducted to examine the microstructural properties of the samples after compaction.

X-Ray diffraction

XRD was conducted to analyse all soil samples after curing periods of 0, 14 and 28 days. The microstructures of the soils were examined to assess short- and long-term reactions. The XRD results for the uncured C2L, C4L, C6L and C8L samples are shown in Figure 4(a). With an increase in the percentage of lime, the XRD patterns of the samples revealed the presence of CAS, CS and CMC within a short period of time. The XRD results for the uncured C2C, C4C, C6C and C8C samples are shown in Figure 5(a). With an increase in the percentage of cement, the XRD patterns of the samples also revealed the presence of CAS, CS and CMC within a short period of time. The XRD results for the C2L, C4L, C6L and C8L samples after curing for 14 days are shown in Figure 4(b). The peak intensity of CS increased with an increase in the percentage of lime. The XRD results for the C2C, C4C, C6C and C8C samples after curing for 14 days are shown in Figure 5(b). The peak intensity of CS and CAS also increased with an increase in the percentage of cement during this time period.

The XRD results for the C2L, C4L, C6L and C8L samples after a 28-day curing period are shown in Figure 4(c). The peak intensity of CS increased with an increase in the percentage of lime over a long period of time. This state is observed in C6L and C8L because of the increased lime percentage. The XRD results for the C2C, C4C, C6C and C8C samples after a 28-day curing period are shown in Figure 5(c). The peak intensity of CS, CAS and CMC increased with an increase in the percentage of cement over an extended period of time. This state is observed in C6C and C8C because of the increased percentage of cement. The XRD patterns for the lime- and cement-stabilised samples revealed additional peaks due to the cementitious reactions. With the addition of 6% and 8% of lime and cement, respectively, this condition was observed after a long curing period. The extended curing period is attributed to the cementitious reactions (Bell, 1996; Brown, 1996; Raj, 1999).

Mercury intrusion porosimetry

MIP was conducted for all soil samples after curing periods of 0, 14 and 28 days. The pore sizes of the soils were examined for short- and long-term reactions. The pore size distributions for the uncured C2L, C4L, C6L and C8L samples are shown in Figure 6(a). The lime-stabilised samples exhibited a gradual increase in the pore sizes in the range of 1–180 µm and in the range of 0.1–0.003 µm; no significant change was observed in the range of 0.1–1 µm. The pore size distributions for the uncured C2C, C4C, C6C and C8C samples are listed in Figure 7(a). The cement-stabilised samples exhibited a gradual increase in the pore sizes in all ranges; however, this increase was more pronounced in the range of 0.1–10 µm. According to the results obtained for the 0-day MIP curing, with increased additives in the mixtures, the pore size distribution increased due to flocculation and cation exchange. The pore size distributions for the C2L, C4L,

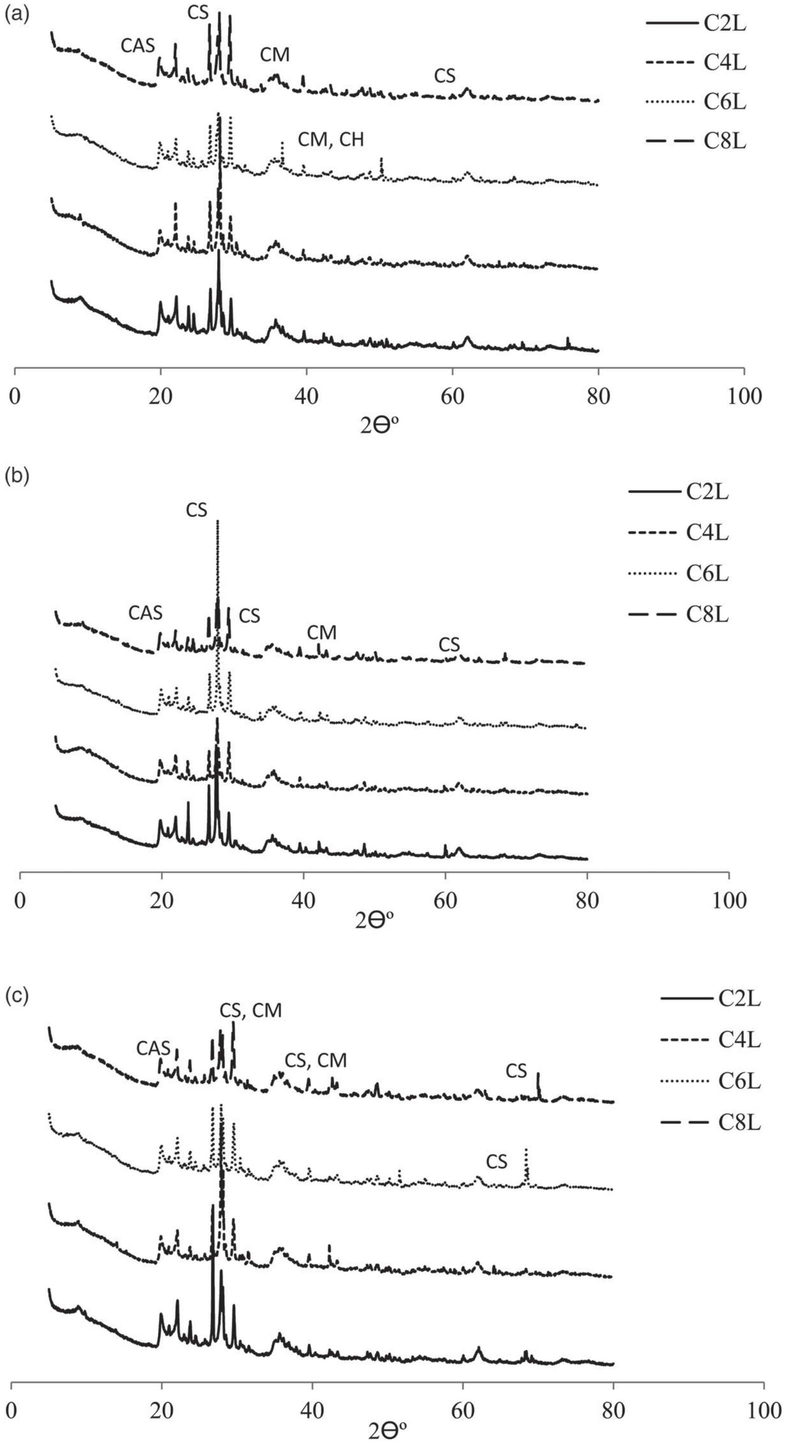

Figure 4. (a) XRD patterns of C2L, C4L, CL6 and C8L (0 day curing). (b) XRD patterns of C2L, C4L, CL6 and C8L (14-day curing). (c) XRD patterns of C2L, C4L, CL6 and C8L (28-day curing).

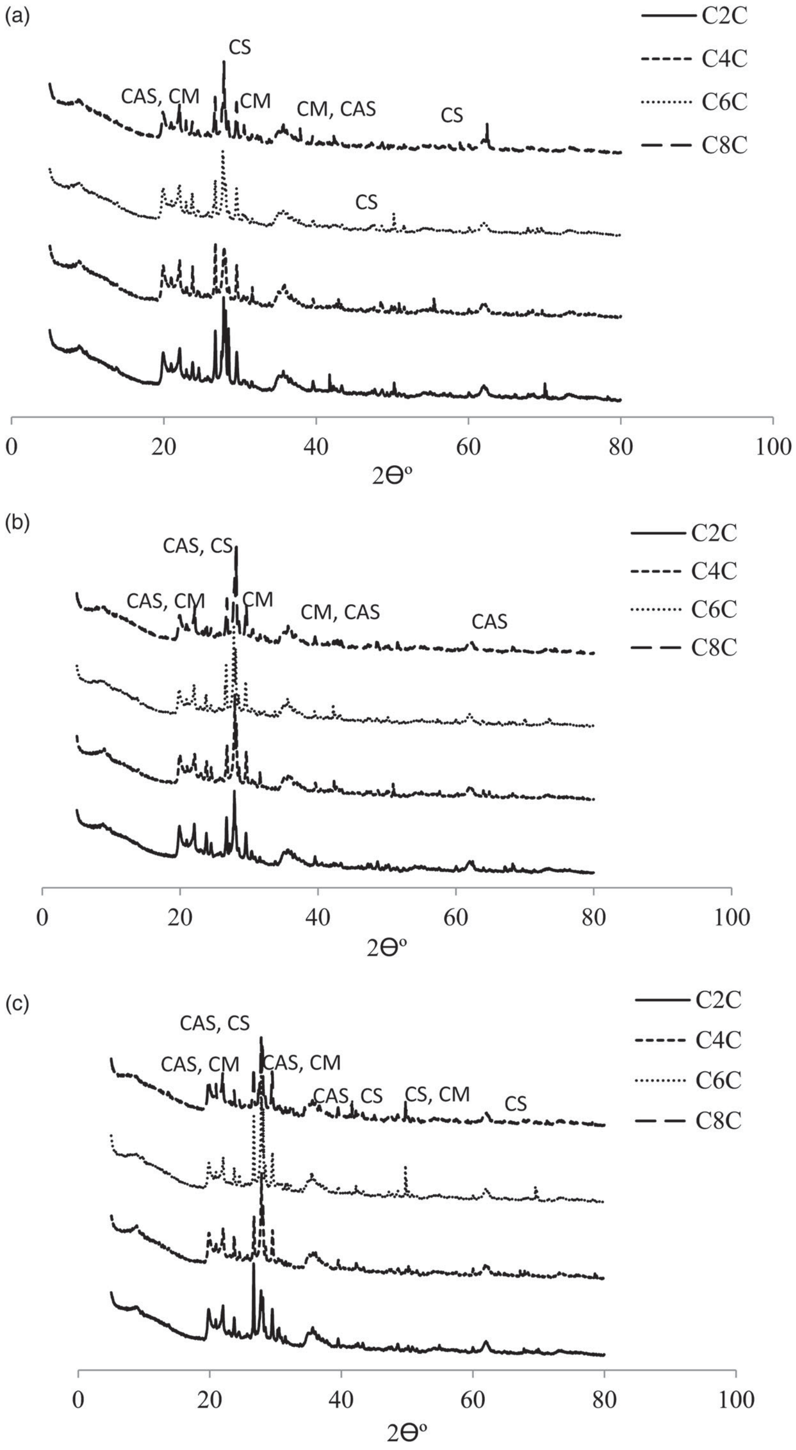

Figure 5. (a) XRD patterns of C2C, C4C, C6C and C8C (0 day curing). (b) XRD patterns of C2C, C4C, C6C and C8C (14-day curing). (c) XRD patterns of C2C, C4C, C6C and C8C (28-day curing).

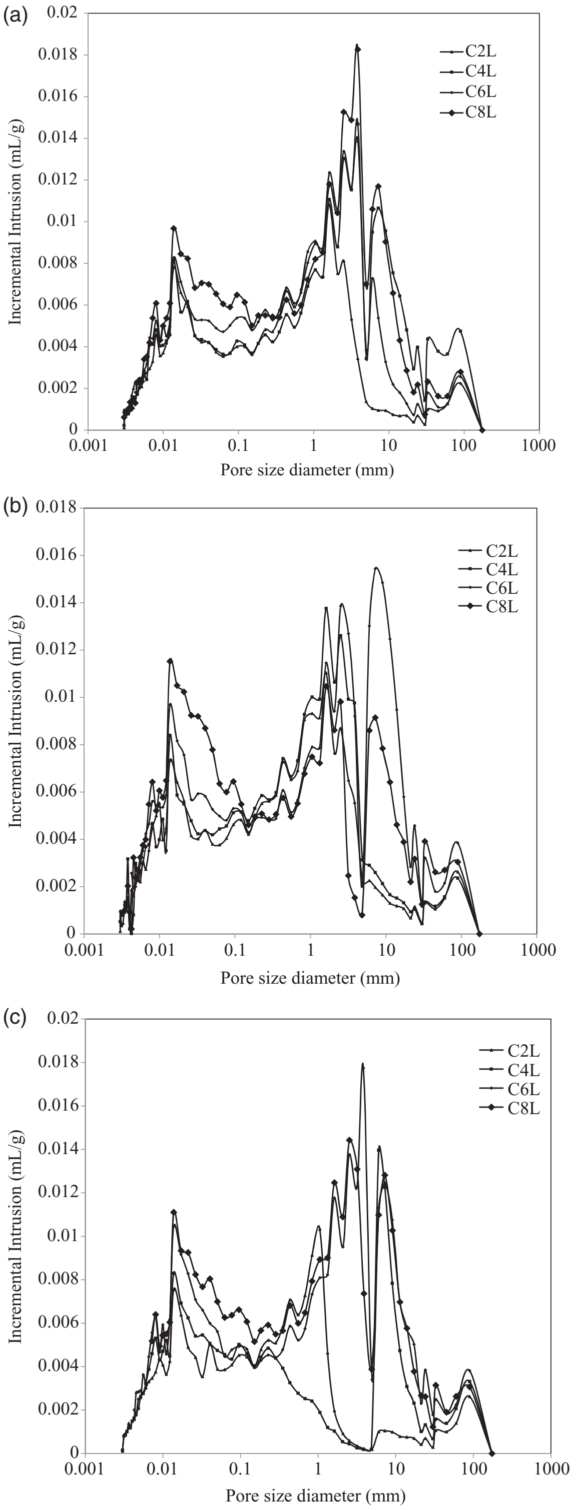

Figure 6. (a) C2L, C4L, CL6 and C8L (0 day curing). (b) C2L, C4L, CL6 and C8L (14-day curing). (c) C2L, C4L, CL6 and C8L (28-day curing).

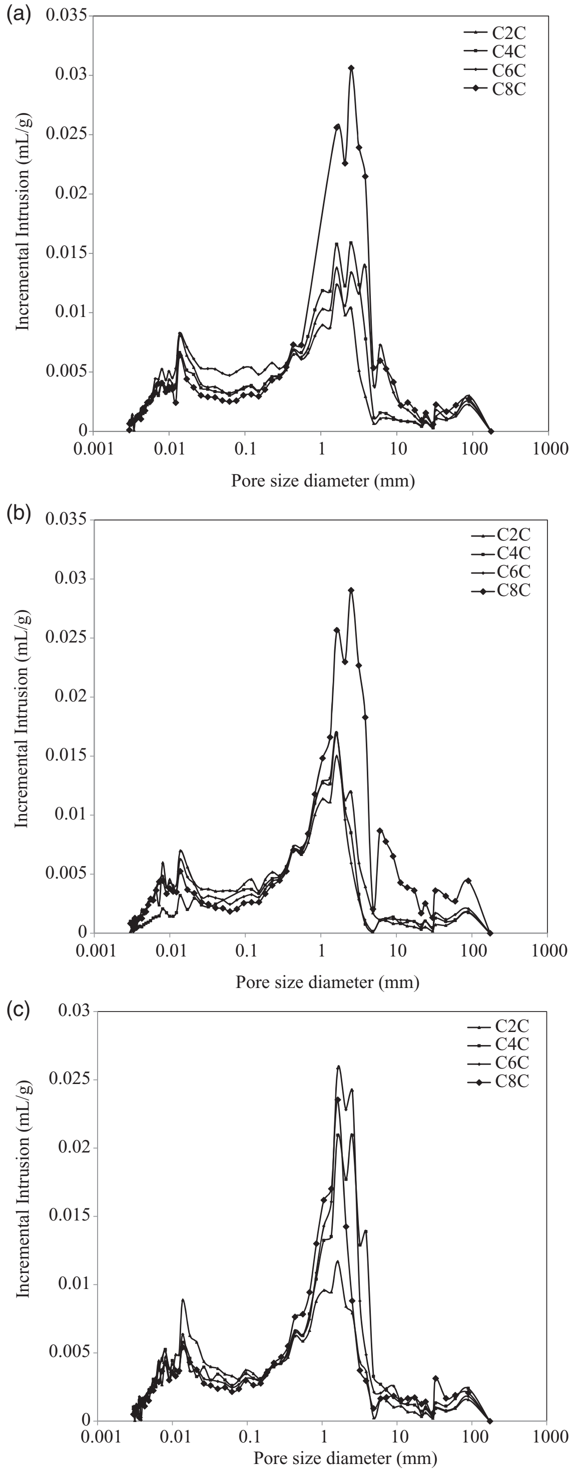

Figure 7. (a) C2C, C4C, C6C and C8C (0 day curing). (b) C2C, C4C, C6C and C8C (14-day curing). (c) C2C, C4C, C6C and C8C (28-day curing).

C6L and C8L samples after a 14-day curing period are shown in Figure 6(b). The lime-stabilised samples exhibited a gradual reduction in the pore sizes in the range of 1–180 µm and in the range of 0.1–1 µm and a gradual increase in the pore sizes in the range of 0.1–0.003 µm. The decreased pore size distribution is more active in the range of 0.1–180 µm due to the occurrence of the pozzolanic reaction with increased curing time. The pore size distributions for the C2C, C4C, C6C and C8C samples after a 14-day curing period are shown in Figure 7(b). The cement-stabilised samples exhibited a gradual increase in pore size in the range of 1–180 µm and a gradual decrease in pore size in the range of 1–0.003 µm for this curing period. This decreased pore size distribution was more active in the range of 1–0.003 µm due to the occurrence of pozzolanic reactions with an increased curing time.

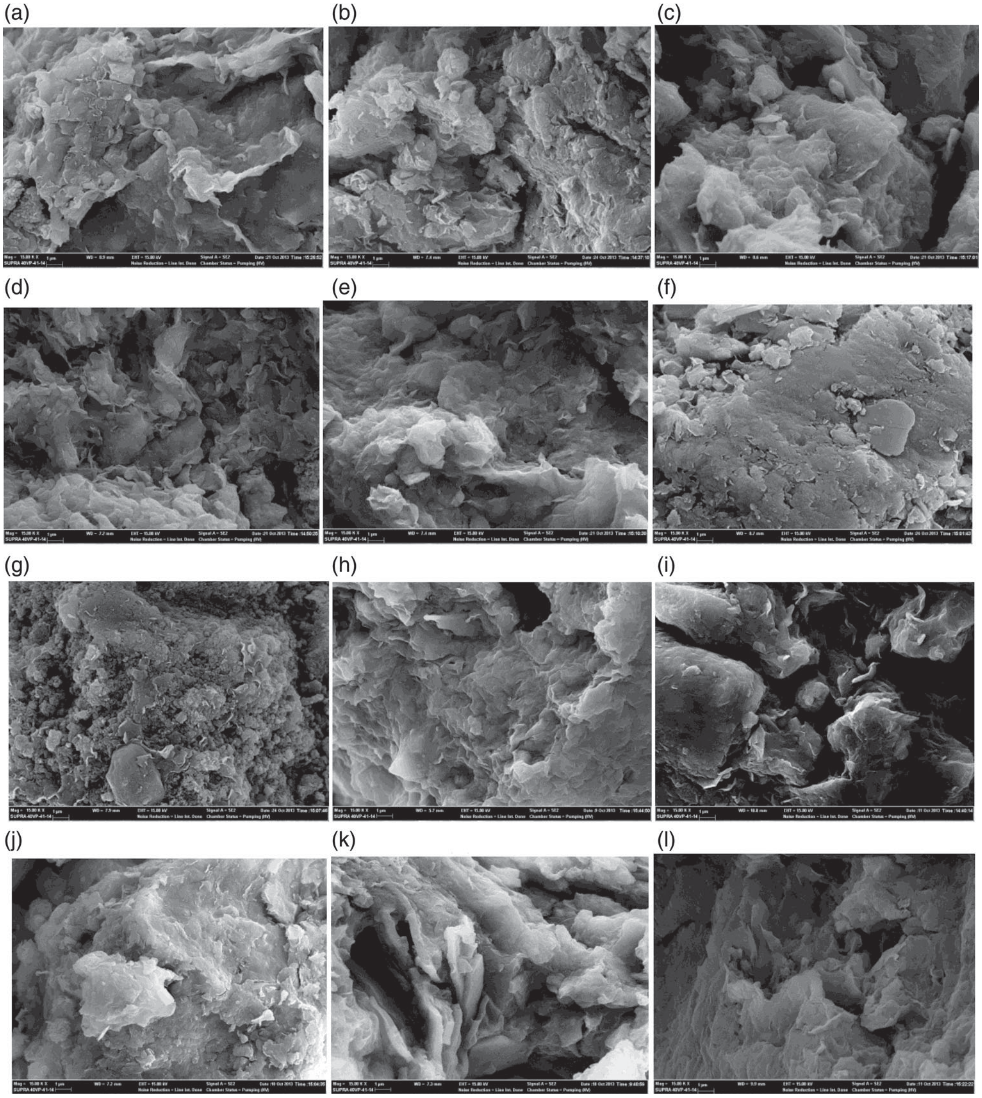

Figure 8. SEM images of samples at × 15.000 magnification (a) C2L (b) C4L (c) C6L (d) C8L (0 day curing) (e) C2L (f) C4L (g) C6L (h) C8L (14-day curing) (ı) C2L (i) C4L (j) C6L (k) C8L (28-day curing).

The pore size distributions for the C2L, C4L, C6L and C8L samples after a 28-day curing period are shown in Figure 6(c). The lime-stabilised samples exhibited a gradual reduction in the pore sizes in the range of 0.1–180 µm and a gradual increase in the pore sizes in the range of 0.1–0.003 µm. This decreased pore size distribution was more active in the range of 0.1–180 µm due to the occurrence of pozzolanic reactions with an increased curing time. The pore size distributions for the C2C, C4C, C6C and C8C samples after a 28-day curing period are shown in Figure 7(c). The cement-stabilised samples exhibited a gradual decrease in pore size for all size ranges. This decreased pore size distribution was due to pozzolanic reactions that occurred with an increased curing time. The effect from the addition of lime and cement was observed after an extensive period of curing. Generally, the reaction of lime and cement with

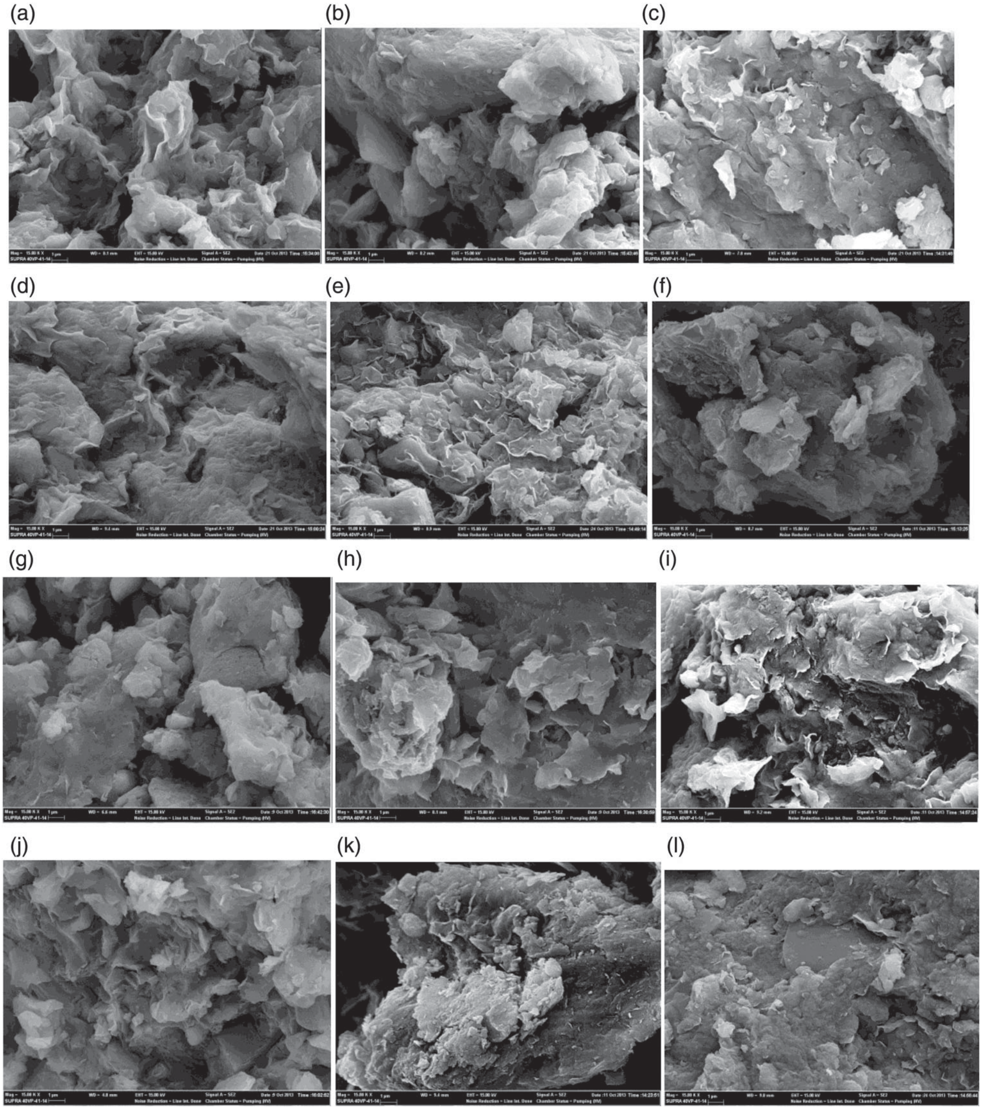

Figure 9. SEM images of samples at × 15.000 magnification (a) C2C (b) C4C (c) C6C (d) C8C (0 day curing) (e) C2C (f) C4C (g) C6C (h) C8C (14-day curing) (ı) C2C (i) C4C (j) C6C (k) C8C (28-day curing).

clay led to the formation of aggregates, and this state led to an increase in porosity among the soil mixtures in uncured samples. Similar pore size distributions were observed by Tran, Cui, Tang, Audiguier, and Cojean (2014) and Rajasekaran and NarasimhaRao (1997). A decreased pore size distribution occurred with increased curing days. Further, greater additions of lime and cement increased the pozzolanic reactions (Al-Mukhtar, Lasledj, & Alcover, 2010).

Scanning electron microscopy

SEM was conducted for all soil samples after curing periods of 0, 14 and 28 days. The microstructures of the soils were examined to assess short- and long-term reactions. The SEM images of the uncured C2L, C4L, C6L and C8L samples are shown in Figure 8(a)–(d). The microstructures of the clay sample contain fewer flaky layers with an increase in the lime percentage due to flocculation. The SEM images of the uncured and cured C2C, C4C, C6C and C8C samples are displayed in Figure 9(a) and 9(d). The C2C (Figure 9(a)) sample is similar to that of untreated clay (Figure 3(b)) due to an inadequate percentage of cement. Above this percentage, an increased percentage of cement was observed with a reduction in flaky layers because the percentage of cement is sufficiently high to be active. This is consistent with the findings of Horpibulsak et al. (2010). The SEM images of the C2L, C4L, C6L and C8L samples after a 14-day curing period are shown in Figure 8(e)–(h). An increase in the percentage of lime and curing time yielded a more flocculated structure. The SEM images after a 14-day curing period of the C2C, C4C, C6C and C8C samples are shown in Figure 9(e)–(h). An increase in the percentage of cement and curing time yielded a more aggregated structure.

The SEM images of the C2L, C4L, C6L and C8L samples after a 28-day curing period are shown in Figure 8(ı)–(k). The increase in the percentage of lime and the curing time yielded a more pronounced flocculation structure. In addition, the large pores between the pellets decreased in accordance with the results of MIP. The SEM images of the C2C, C4C, C6C and C8C samples after a 28-day curing period are shown in Figure 9(ı)–(k). The increase in the percentage of cement and curing time caused the pores between the pellets to decrease, in accordance with the MIP results, due to flocculation. In addition, white lumps were observed after 14 and 28 days of curing. This finding is also consistent with those of Kassim (2009) and Muhmed & Wanatowski (2013). The SEM images indicated that an increase in the percentages of lime and cement changed the microstructure of the additive soils. The reactions involving the two additives are similar. The difference between the reactions of additives is that more time is required for the soil–cement reactions.

Table 3. Specific surface area (g/cm^3) of mixtures.

Mixtures name	Specific surface area (g/cm^3)
C0L/C0C	36.91
C2L	30.30
C4L	29.30
C6L	21.19
C8L	19.12
C2C	24.58
C4C	17.70
C6C	14.66
C8C	15.11

Specific surface area

The SSA was calculated for all uncured soil samples. The SSAs of the uncured C2L/C2C, C4L/C4C, C6L/C6C and C8L/C8C samples are listed in Table 3. The SSA exhibits an important influence on the behaviours of many fine-grained soils. The SSA of mixtures decreased with an increase in the lime and cement percentage. Equivalent percentages of lime and cement were compared, and the SSAs of the cement mixtures were smaller than those of the lime mixtures.

Conclusions

In this study examining clay, standard compaction tests and XRD, MIP, SEM and SSA analyses were performed to evaluate the geotechnical index properties and the pore size distribution of soil. The following conclusions can be drawn from this study:

- The γ_{dmax} values of the samples substantially decreased with the percentage of lime, whereas the OWC increased with the percentage of lime. The γ_{dmax} values of the samples substantially increased with the percentage of cement, whereas the OWC decreased with the percentage of cement.
- XRD was conducted to analyse all soil samples after curing periods of 0, 14 and 28 days. The microstructures of the soils were examined to assess the short- and long-term reactions. With increased percentages of lime and cement, the XRD patterns of the samples revealed the presence of CAS, CS and CMC within a short period of time. The peak intensity of CS increased with an increase in the percentage of lime, and the peak intensity of CS and CAS increased with an increase in the percentage of cement during this time period. The peak intensity of CS increased with an increase in the percentage of lime, and the peak intensity of CS, CAS and CMC increased with an increase in the percentage of cement over an extended period of time. This state is observed in C6L/C6C and C8L/C8C based on the increase in the lime or cement percentage. The XRD pattern for the lime- and cement-stabilised samples revealed additional peaks of formation due to the cementitious reactions. With the addition of 6% and 8% of lime and cement, respectively, this condition was observed after a long curing period. The extended period of curing is attributed to the cementitious reactions. The XRD results confirm those obtained in previous studies.
- MIP was conducted for all soil samples after curing periods of 0, 14 and 28 days. The pore sizes of the soils were examined for short- and long-term reactions. According to the results obtained at 0 days of MIP curing, the pore size distributions of the mixtures were increased due to flocculation and cation exchange. Generally, the reaction of lime and cement with clay led to the formation of aggregates, and this state led to an increase in porosity among the soil mixtures in the uncured samples. Similar pore size distributions were observed in previous studies. With increased curing days, the pore size distribution decreased due to pozzolanic reactions. According to the results obtained for 28 days of MIP curing, the pore size distributions of the mixtures decreased due to the pozzolanic reactions with an increased number of curing days.
- SEM was conducted for all soil samples after curing periods of 0, 14 and 28 days. The microstructures of the soils were examined to assess the short- and long-term reactions. The SEM images of the uncured C2L, C4L/C4C, C6L/C6C and C8L/C8C samples showed that the microstructures of the clay sample contained fewer flaky layers with an increase in the lime or cement percentage due to flocculation. The SEM images of the C2L/C2C, C4L/C4C, C6L/C6C and C8L/C8C samples after a 14-day curing period showed that an increase in the percentage of cement and curing time yielded a more aggregated structure.

The SEM images of the C2L/C2C, C4L/C4C, C6L/C6C and C8L/C8C samples after a 28-day curing period showed that an increase in the percentage of lime/cement and curing time yielded a more pronounced flocculation structure. In addition, the large pores between the pellets decreased in accordance with the MIP results due to flocculation. The SEM images indicated that an increase in the percentages of lime and cement changed the microstructure of the additive soils. The reactions formed by the two additives are similar to each other. The difference between the reactions of additives is that more time is required for the soil–cement reactions.

- The SSA was calculated for all of the uncured soil samples. The SSA of the mixtures was decreased by increasing the lime and cement percentages. At equivalent percentages of lime and cement, the SSA of the cement mixtures was smaller than the SSA of the lime mixtures.

Disclosure statement

No potential conflict of interest was reported by the authors.

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