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The relationship between geotechnical index properties and the pore-size distribution of compacted clayey silts

Abstract: In this study, the relationships between geotechnical index properties and the pore-size distribution of compacted natural silt and artificial soil mixtures, namely, silt with two different clays and three different clay percentages (10%, 20%, and 40%), were examined and compared. Atterberg's limit tests, standard compaction tests, mercury intrusion porosimetry, X-ray diffraction, scanning electron microscopy (SEM) analysis, and Brunauer-Emmett-Teller specific surface analysis were conducted. The results show that the liquid limit, the cumulative pore volume, and specific surface area of artificially mixed soils increase with an increase in the percentage of clay. The cumulative pore volume and specific surface area with geotechnical index properties were compared. High correlation coefficients were observed between the specific areas and both the liquid limit and the plasticity index, as well as between the cumulative pore volume and both the clay percentage and the *S* coefficient. In addition, qualitative SEM analyses were conducted.

Keywords: clay; mineralogy; pore-size distribution; scanning electron microscope; silt.

1 Introduction

Soils have pores, and therefore pore-size distribution is one of their most important properties. The pore-size distribution of a soil depends on the particle-size distribution of the soil, the shape of the particles, and the clay mineralogy. In particular, clay mineralogy is a factor that affects the geotechnical properties and pore-size distribution of soils. Clay is a very important material in geotechnical engineering. Geotechnical applications include road construction, dams, slurry walls, and waste landfills. Clay soils are fine-grained soils, but not all fine-grained soils are clays. Clay minerals are very active electrochemically and have a very large surface area [1, 2]. Because of the properties of these soils, conducting a pore-size distribution analysis will help in the interpretation of the geotechnical properties of soils. The pore volume between soil grains is affected by the introduction of small grains. In particular, studies on the micro-, meso-, and macropores found in soils are needed to improve our understanding of the physical properties of compacted soils. Sing et al. [3] classified the diameters of pores as follows: macropores, >0.05 μm (>50 nm); mesopores, >0.002 μm and <0.05 μm (2–50 nm); and micropores, <0.002 μm (<2 nm). Generally, the most common techniques for pore-size distribution analysis are mercury intrusion porosimetry (MIP) and gas adsorption isotherms [Brunauer-Emmett-Teller (BET) specific surface analysis]. In this study, the pore-size distributions of silt and artificially mixed soil samples were determined by MIP.

Studies of the effects of the mineralogy of clay on the microstructure of soils exist in the literature [4–11]. Mitchell [12] provided evidence that Atterberg's limits and grain-size distribution are indicators of the mineralogy of a soil. Many researchers have emphasized the importance of Atterberg's limits for the determination of many properties of fine-grained soils [12, 13]. Atterberg's limits affect grain-size distribution and mineral composition. Generally, an increase in the surface area shows increased liquid limits [12, 14]. In recent years, correlations between liquid limits and specific surface areas have been proven by Kuzukami et al. [15], Wetzel [16], and Dolinar et al. [17].

Dumbleton and West [18] investigated the relationship between plastic and liquid limits and the clay contents of natural and artificial mixtures of pure clay minerals with quartz sand-silt. They concluded that, with some types of soil, a correlation is found between the soil properties and the clay minerals present, but other factors are also involved. Grabowska-Olszewska [19] presented the relationship between the colloidal activity and the specific surface area of model soils of kaolinite and bentonite mixtures. The test results of soils show that, when the clay fraction increases, the total surface area also increases. Rahardjo et al. [20] studied the results of the index property and engineering property tests on residual soils from two major geological formations in Singapore.

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The results of those tests indicate that the variations in the index and engineering properties of the residual soils at different depths are largely influenced by the pore-size distributions, which vary in accordance with the degree of weathering.

Ouhadi and Yong [21] investigated the application of four current XRD methods for mineral quantification purposes, which were experimentally reviewed for clayey soils. In conclusion, they observed an agreement between the computed and the real quantity of each mineral present in a series of artificial samples by validating the XRD method. Dananaj et al. [22] evaluated the differences in microstructure formation and geotechnical properties between Ca-bentonite and Na-bentonite using XRD, chemical analysis, and scanning electron microscopy (SEM). They reported that the differences in bentonite quality and smectite quantity influence the permeability. Andrejkovičová et al. [23] investigated the chemical and mineralogical composition and geotechnical properties of Ca^2 bentonite samples from the Lieskovec deposit, Slovakia. They reported that smectite content in bentonite samples is the dominant variable affecting their geotechnical parameters. Dimitrova and Yanful [24] investigated the factors affecting the shear strength of mine tailings and clay mixtures with varying clay content levels and clay mineralogy. They found that adding clay to mine tailings generally caused a decrease in frictional strength, but the magnitude of this decrease was greater when the clay was bentonite and lower when it was kaolinite.

This study investigated the relationship between geotechnical index properties and the pore-size distribution of compacted natural silt and artificially mixed soils. The relationships are shown using the results of Atterberg's limit tests, standard compaction tests, MIP, X-ray diffraction (XRD), and specific surface analysis (BET). In addition, SEM analysis was performed on all soil samples.

2 Materials and methods

2.1 Geotechnical index properties of silt and artificially mixed soils

The soil used was silt obtained from Adapazarı, Turkey. The soil was classified as a low-plasticity silt (ML). The geotechnical index properties of the silt used are given in Table 1. The particle-size distributions of both the soil and the artificially mixed soils were obtained by hydrometer analysis. The soil was classified as a low- to medium-plasticity silt. The liquid limit for the soil and artificially mixed soils was determined by means of the fall cone method. The physical properties of the silt and the two different clays are given in Table 1.

Artificially mixed soils with clay I and clay II were prepared in this study. Silt was the base material. The mixed soils were prepared using 10%, 20%, and 40% of the dry weight of clay I and clay II to silt. The geotechnical index properties of the artificially mixed soils used are given in Table 2. The liquid limit increased with an increase in the clay I or clay II content, and the plasticity index increased more than the increase in the percentage of clay II. The clay percentage of artificially mixed soils with clay I was more than that of artificially mixed soils with clay II.

2.2 Sample preparation

In this study, standard proctor tests were carried out on remolded samples of silt and artificial mixture soils. The optimum water content (OWC) and maximum dry density (γ_{dmax}) values of the silt and artificially mixed soil samples were calculated using standard compaction tests, and these values were used to analyze these samples. The standard proctor test was conducted according to ASTM D698 (2000) [25]. The effects of the clay content on the γ_{dmax} and OWC of the mixtures are shown in Table 3. Whereas the OWC increased with an increase in the clay I or clay II content, the γ_{dmax} decreased with an increase in the clay I or clay II content.

2.3 XRD analysis

The mineralogical analysis of soils is very important with regard to geotechnical properties. Soil mineralogy is generally used owing to its strong influence on soil behavior. Therefore, mineralogical analyses of the silt and artificially mixed soil samples were conducted using an X-ray diffractometer. The XRD patterns of silt samples showed the presence of illite, calcite, and quartz, as shown in

Table 1 Geotechnical index properties of silt and clays.

Mixtures	Liquid limit[a]	Plastic limit	Sand (%)	Silt (%)	Clay (%)	Specific gravity
Silt (S1)	32	–	15	79	6	2.66
Clay I	50	25	4	34	62	2.67
Clay II	103	38	13	46	41	2.66

[a] Fall cone method.

Table 2 Geotechnical index properties of artificially mixed soils.

Soil/ mixtures	Name of additional clay	Ratio of additional clay (%)	Liquid limit[a]	Ip	Sand (%)	Silt (%)	Clay (%)	Specific gravity
S2	Clay I	10	33	12	17	73	12	2.67
S3	Clay I	20	34	14	15	57	26	2.67
S4	Clay I	40	35	16	13	44	43	2.67
S5	Clay II	10	36	14	15	75	10	2.65
S6	Clay II	20	41	17	15	69	16	2.65
S7	Clay II	40	51	25	17	57	26	2.63

[a]Fall cone method. Ip, plasticity index.

Table 3 Physical properties of silt and artificially mixed soils.

Soil/mixtures	OWC	γ_{dmax} (kN/m^3)
S1	12.25	16.63
S2	13.00	16.50
S3	16.00	16.15
S4	21.00	16.05
S5	13.50	16.55
S6	18.50	16.45
S7	23.00	16.35

OWC, Optimum water content; γ_{dmax}, maximum dry density.

Figure 1. The XRD patterns of clays showed the presence of kaolinite, montmorillonite, anorthite, and quartz, as shown in Figure 1. In Figure 2, the XRD patterns of artificially mixed soil samples indicate the presence of kaolinite, illite, calcite, montmorillonite, and quartz.

2.4 MIP and BET analysis

Pore-size distribution is one of the most important and fundamental parameters influencing the geotechnical behavior of a soil. In this study, the pore-size distribution was determined using the MIP method. MIP has been commonly used to assess the pore-size distribution of bulk materials such as soils with pore structures. With the MIP method, the mercury pressure is increased in steps and the intruded volume of mercury is recorded for each increase in pressure. MIP was conducted on soil samples prepared with the standard proctor test.

In Figures 3–5, the pore-size distributions of silt and artificially mixed soil samples from the MIP tests are displayed. The changes in the cumulative and incremental pore volumes vs. the pore-size diameter are presented in these figures. In Figures 4 and 5, the pore-size distributions of artificially mixed soil samples are compared. In both figures, a very gradual increase is shown in the slope of the curve up to the 1-µm pore diameter. The curve then steepens, and there is a rapid increase in the slope between 1 and 0.01 µm. Subsequently, the curve starts flattening out from 0.01 to 0.001 µm. In artificially mixed soil samples, the cumulative pore volumes obtained from Figure 4 were 0.0782, 0.0957, and 0.1477 mL/g for samples of 10% clay I additive mixture, 20% clay I additive mixture, and 40% clay I additive mixture, respectively. In the other artificially mixed soil samples, the cumulative volumes obtained from Figure 5 were 0.0654, 0.0745, and 0.1399 mL/g for samples of 10% clay II additive mixture, 20% clay II additive mixture, and 40% clay II additive mixture, respectively. Increased clay I additive in the samples showed an increase in the cumulative pore volume. The cumulative volume increased according to the increasing clay percentage, but the increase in the clay I additive mixture was higher than that in the clay II additive mixture owing to the greater percentage of clay.

The specific surface area represents the affected physical and chemical properties of fine-grained soils. The specific surface area is predominantly affected by the grain-size distribution and by the types and amounts of different clay minerals. In this study, the specific surface area was determined using the BET method. The adsorption of nitrogen and its application to the isotherms of the BET equation are a recognized method for determining the surface area of soil particles. The BET method was used to determine the specific surface area of the silt and artificially mixed soil samples. In Figure 6, the isotherm for artificially mixed soil samples is provided. The BET isotherms measured in artificially mixed soil and silt samples are plotted in this figure. The specific surface areas obtained were 12.31, 12.69, 12.20, and 13.81 m^2/g for the silt samples, 10% clay I additive mixture, 20% clay I additive mixture, and 40% clay I additive mixture, respectively. In the other artificially mixed soil samples, the specific surface areas obtained were 12.85, 17.07, and 21.23 m^2/g for samples of 10% clay II additive mixture, 20% clay II additive mixture, and 40% clay II additive mixture, respectively. The specific

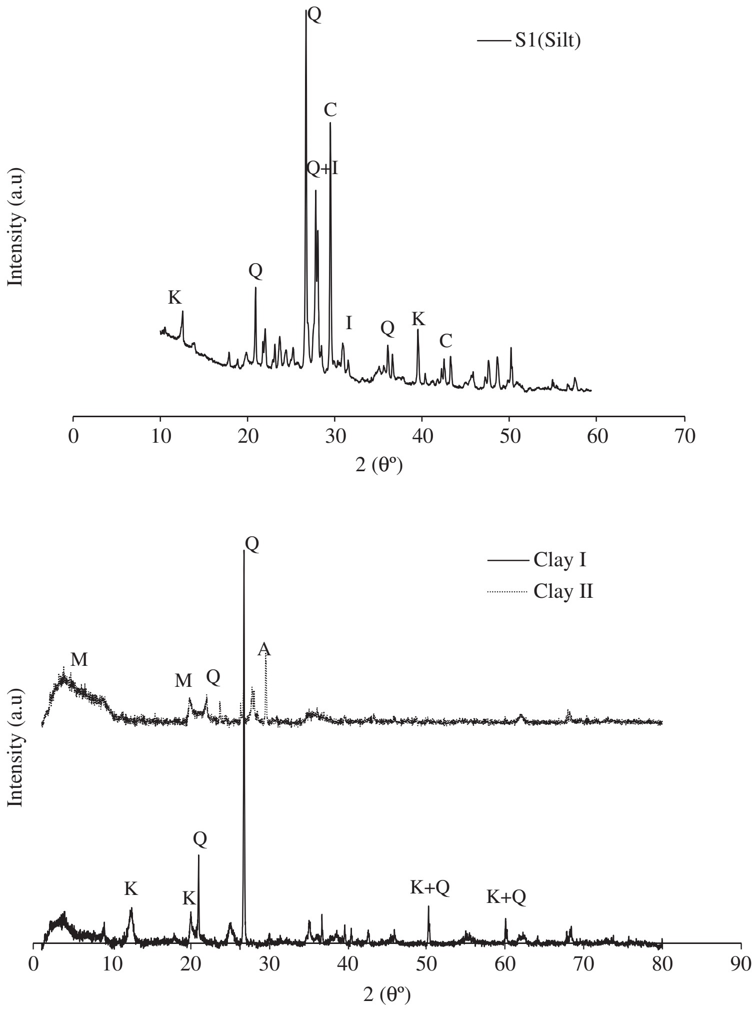

Figure 1 X-ray diffraction patterns of silt and clays. I, Illite; Q, quartz; C, calcite; K, kaolinite; Q, quartz; M, montmorillonite; A, anorthite.

surface area increased by increasing the clay percentage, but an increase in the clay II additive mixture was higher than that in the clay I additive mixture owing to the clay mineralogy including montmorillonite. The changes in the pore volumes vs. the pore-size diameters are given in Figure 7. It was observed from the curves that the soils contain meso- and macropores.

2.5 SEM analysis

The microstructure of the soils was observed using a versatile, analytical, and ultrahigh-resolution field-emission SEM. A scanning electron microscope is a scientific instrument that utilizes a beam of electrons to image a specimen. SEM provides a high level of magnification. It magnifies bulk or powder specimens up to 100,000 times and enables the evaluation of the differences in the surface by means of imaging surface structures. The changes in the microstructural development of soils due to the addition of different materials play a significant role in the geotechnical properties of soils. In particular, these parameters could lead to a better understanding of the engineering properties of compacted soils. In this study, the magnification of the SEM images chosen was ×5000. SEM images of the silt samples are presented in Figure 8, and SEM images of the artificially mixed soil samples are presented in Figures 9 and 10. Figure 8 shows a dispersed structure for compacted silt samples. Figures 9A and 10A show that the samples present a flocculated structure. Figures 9B and 10B show that the samples present a more flocculated structure. Figures 9C and 10C show that the samples present an aggregate structure. Briefly, it was seen that, as the clay content increased, the soil structure became more like a flocculated structure.

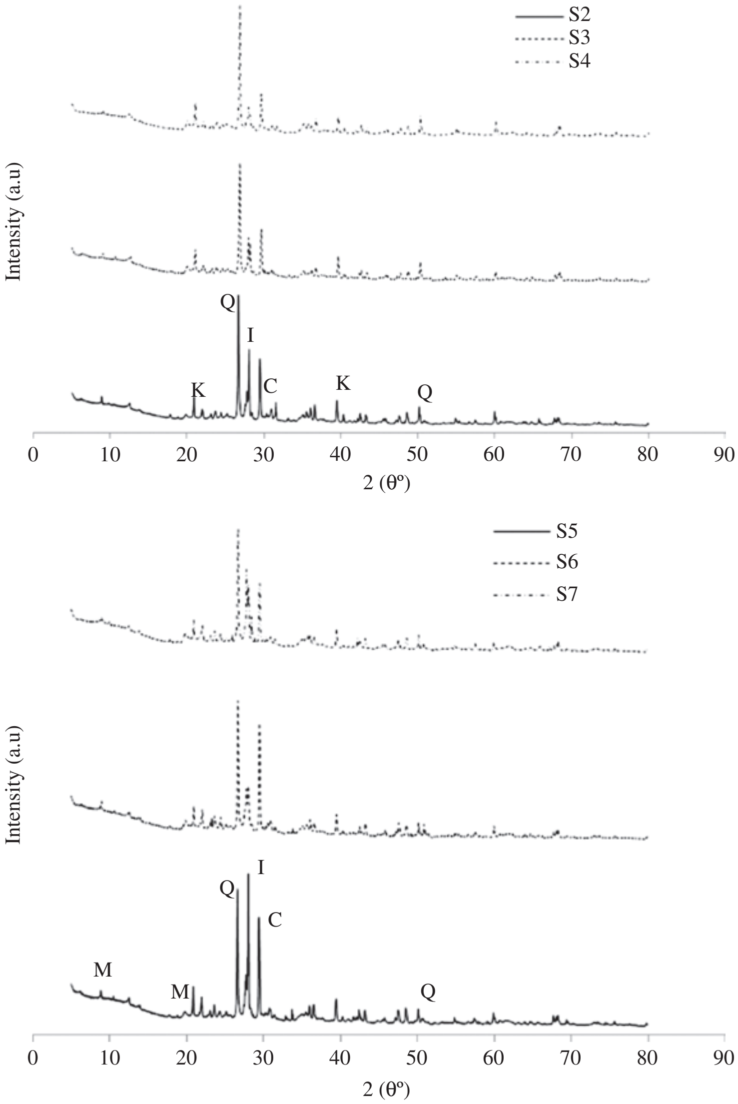

Figure 2 X-ray diffraction patterns of artifically mixed soil samples. K, Kaolinite; I, illite; Q, quartz; C, calcite; I, illite; Q, quartz; C, calcite; M, montmorillonite.

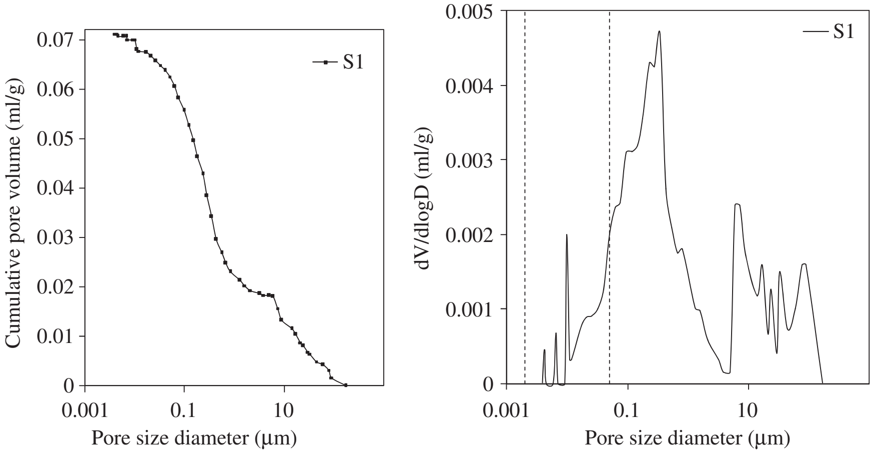

Figure 3 Pore-size distribution of silt measured by MIP.

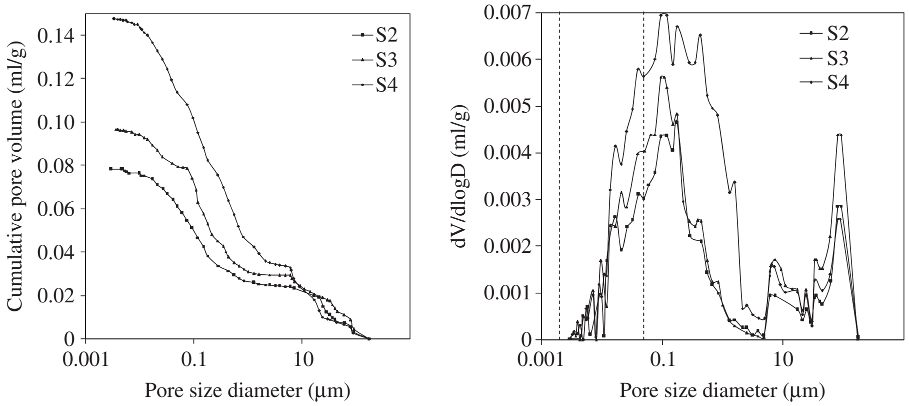

Figure 4 Pore-size distribution of artifically mixed soil samples measured by MIP.

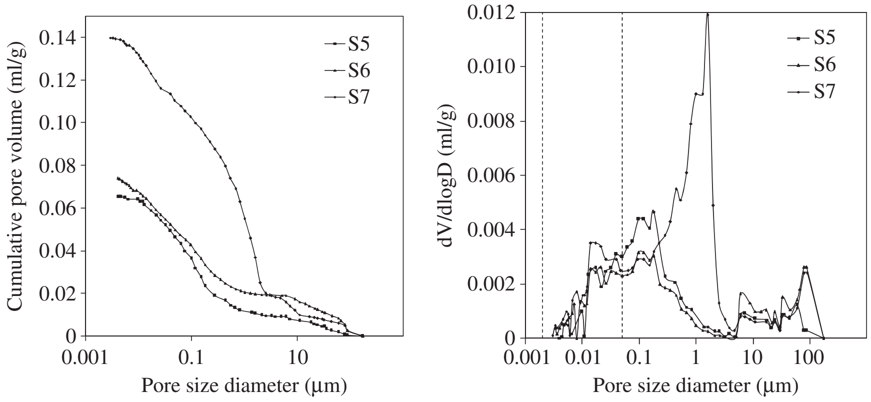

Figure 5 Pore-size distribution of artifically mixed soil samples measured by MIP.

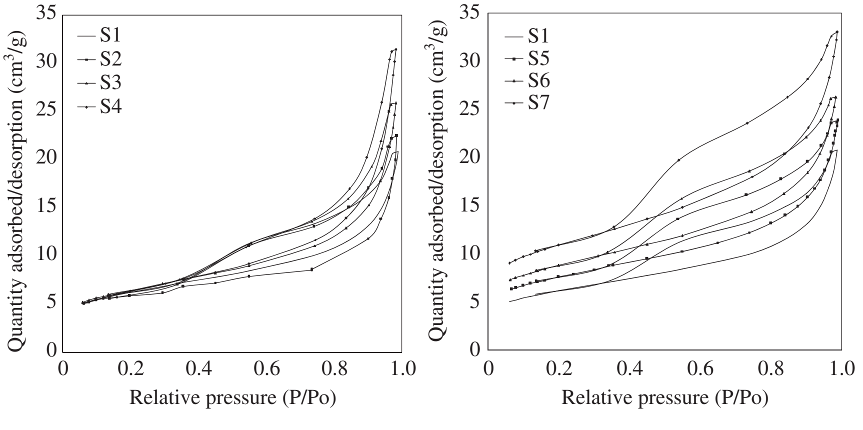

Figure 6 BET isotherm of artifically mixed soil and silt samples.

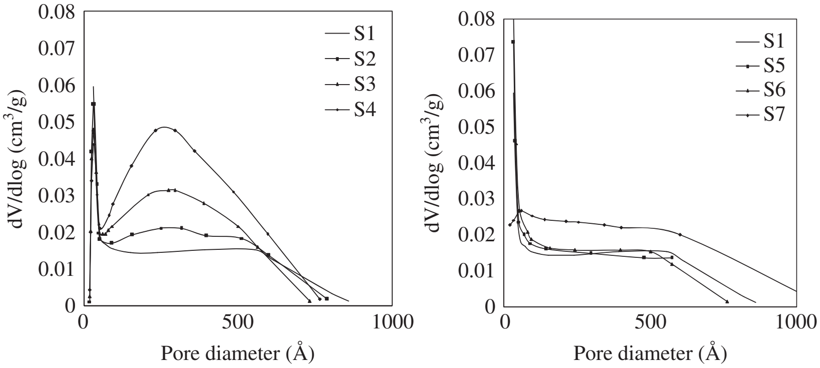

Figure 7 Pore-diameter distribution of artifically mixed soil and silt samples.

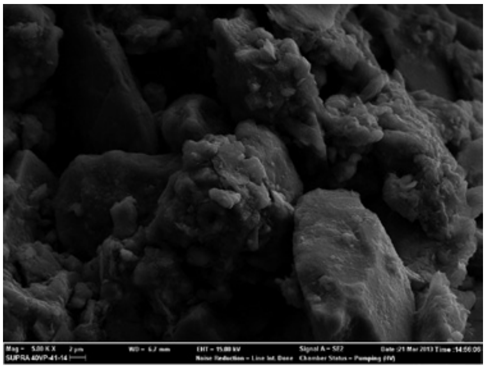

Figure 8 SEM images of silt samples (magnification ×5000).

3 Comparative analysis and geotechnical index properties of soils

3.1 Between liquid limit, plasticity index, and BET analysis

In this study, we compared the geotechnical index properties and the results of the BET analysis (Figure 11). Between a specific region involving the liquid limit and the plasticity index, there was a high correlation coefficient, whereas

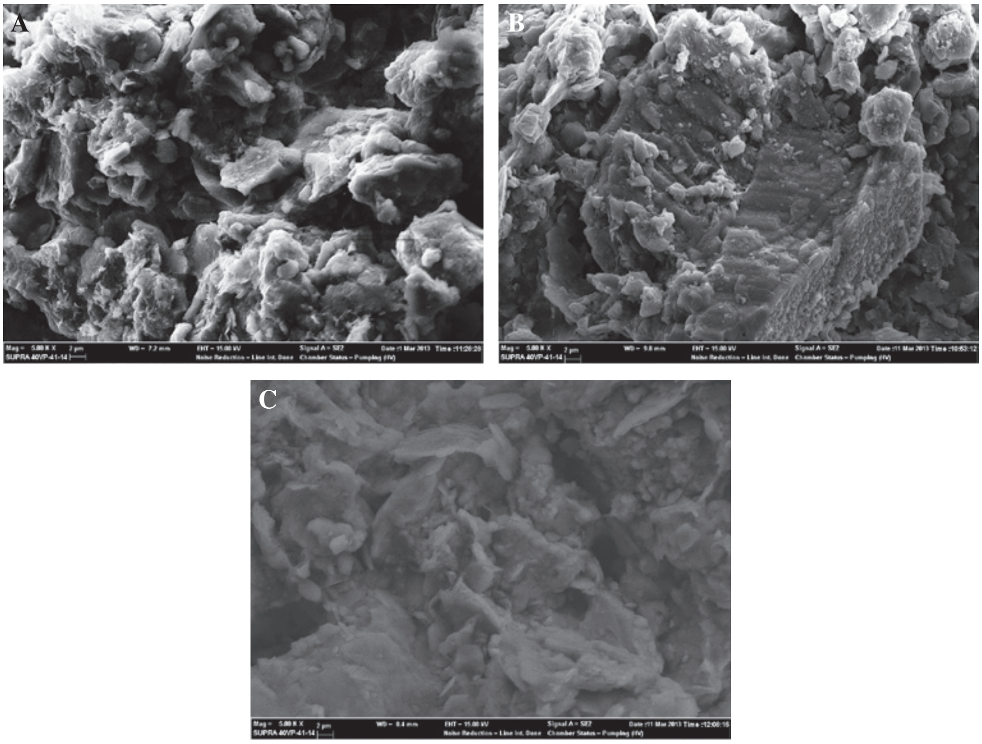

Figure 9 SEM images of artifical mixed soil samples (magnification ×5000): (A) 10% clay I, (B) 20% clay I, and (C) 40% clay I.

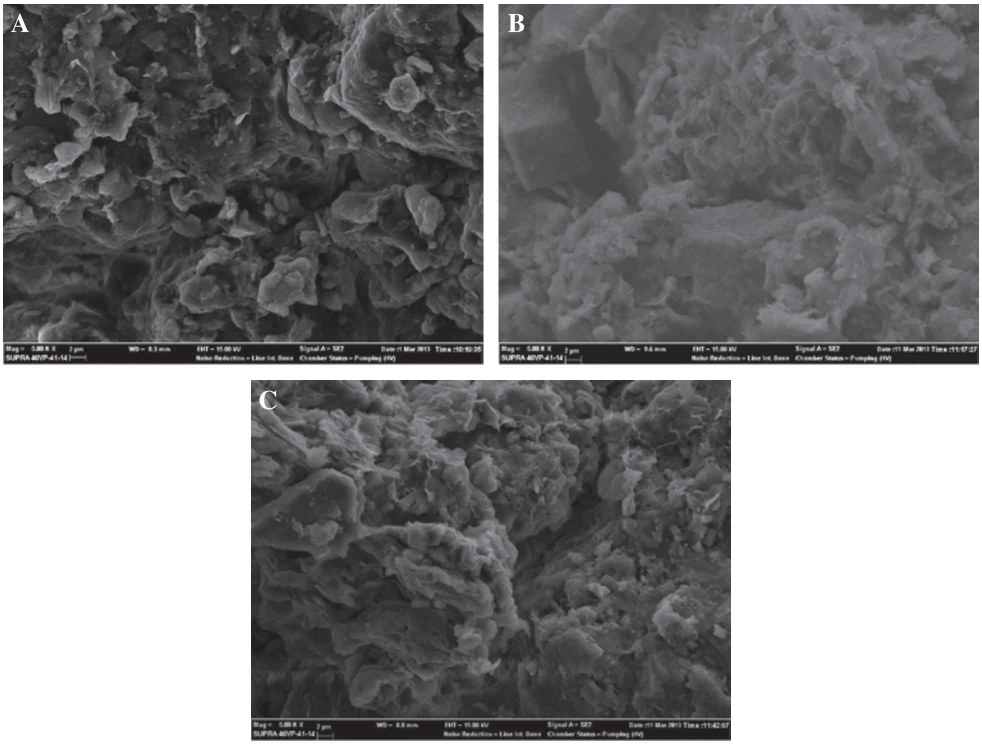

Figure 10 SEM images of artifical mixed soil samples (magnification ×5000): (A) 10% clay II, (B) 20% clay II and (C) 40% clay II.

the correlation coefficient between specific areas and the plastic limit was lower than in other regions.

3.2 Between the grain-size distribution and MIP analysis

The mechanical, physical, and physicochemical properties of soils affect the grain-size distribution. In this study, we compared the grain-size distribution and the results of an MIP analysis (Figure 12). Cumulative pore volumes with clay and silt had a high correlation coefficient, but the correlation coefficients of cumulative pore volumes involving sand were lower. A combination of all materials was thought to be better. To this end, in this study, all percentages were defined with the *S* coefficient. The *S* coefficient was computed as follows: [(clay percentage/fine-grain percentage)×sand percentage]. Cumulative pore volumes using the *S* coefficient had a high correlation coefficient.

4 Conclusions

The importance of the physicochemical characteristics of fine-grained soils combined with other geotechnical index properties was the focus of this research. In this study, we used two different types of clay and silt soil. Index tests (particle-size distribution, Atterberg's limits, and specific gravity), standard compaction tests, MIP, XRD, BET analyses, and SEM analyses were carried out to evaluate the geotechnical index properties and pore-size distribution of clay-silt mixtures with varying mineralogies. In conclusion, the relationship between the geotechnical index properties and the pore-size distribution for compacted natural silt and artificially mixed soil samples was investigated. The following conclusions were drawn based on the test results and on the discussion presented in this study:

- The liquid limit of artificially mixed soils increased with an increase in the percentage of clay. In particular, the liquid limits of clay II additive mixtures were higher than those of the clay I additive mixtures. The XRD patterns of clay II showed the presence of kaolinite, montmorillonite, anorthite, and quartz. Because of the presence of montmorillonite, the increase in the liquid limit in clay II and clay II additive mixtures was higher than in the others. Montmorillonite played a significant role in the resulting behavior.
- According to the pore-size distribution analysis, the cumulative pore volume increased with an increase

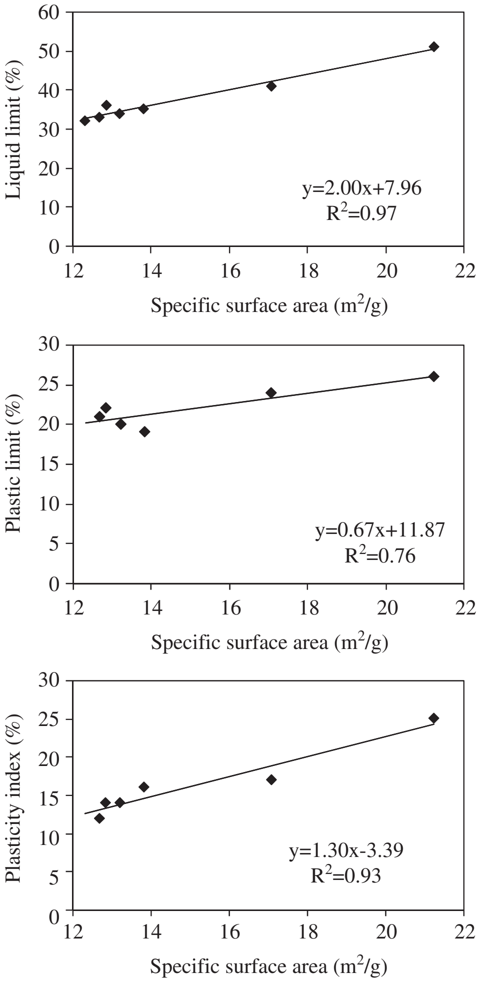

Figure 11 Relations between BET, surface area, liquid limit, plastic limit, and plasticity index.

in the percentage of clay. That is, the porosity of the soil samples increased as a result of the clay. The reason for this is that the layer of adsorbed water around the clay grains became thicker owing to the increasing liquid limit, and then the sample was dried for the test, so it had more spaces. The increase in the cumulative pore volume of the clay I additive mixture samples was higher than in the clay II additive mixtures owing to the clay percentage.

- The specific surface area of artificially mixed soil samples increased with increasing clay percentage. In particular, the increase in the adsorptive capacity was higher in the clay II additive mixture samples than in the clay I additive mixtures owing to the presence of montmorillonite.
- Based on the SEM images, it was observed that an increase in clay content resulted in a porous structure.

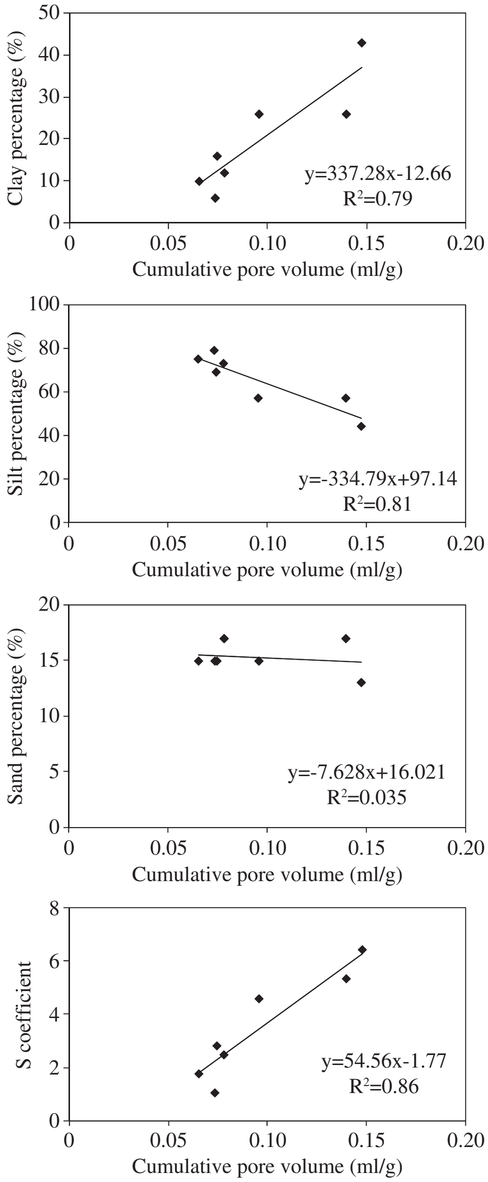

Figure 12 Relations between cumulative pore volume and clay, silt, and sand percentage and *S* coefficient.

Briefly, when the clay content was increased, the soil structure had a more flocculated structure. However, it was very difficult to identify pores using SEM. The pore-size distribution could be determined quantitatively only through MIP. SEM and MIP showed that the cumulative pore volumes increased with increasing clay percentages, indicating that the soil structure was better flocculated.

- The specific surface area and cumulative pore volumes with geotechnical index properties were compared. High correlation coefficients were observed between the specific surface areas and both the liquid limit and the plasticity index, as well as between the cumulative pore volume and both the clay percentage and the *S* coefficient. More tests need to be conducted using different clay percentages and mineralogy to establish more precise and generalized correlations between cumulative pore volumes and the *S* coefficient of soils.

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