International Journal of GEOMATE, Sept., 2022, Vol.23, Issue 97, pp.90-96
ISSN: 2186-2982 (P), 2186-2990 (O), Japan, DOI: https://doi.org/10.21660/2022.97.3221
Geotechnique, Construction Materials and Environment

OEDOMETRIC BEHAVIOR OF SOIL-DIATOM MIXTURES BEFORE FRUSTULE CRACKING

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ABSTRACT: The aim of this research is the characterization of the effects of diatom content on the consolidation of soil-diatom mixtures before the breakage of frustule particles. Results of implemented laboratory tests that characterized the compressibility of soil mixtures made of fine-grained soil and *Cyclotella Distinguenda* diatoms were analyzed to define the physical properties of specimens including water content by mass, Casagrande test, specific gravity, and grain size distribution. Moreover, the compressibility was evaluated using a one-dimensional oedometer test. The compressibility index of the samples decreased as the diatom content increased. Additionally, the coefficient of consolidation increases at higher diatom contents, leading to a faster process of pore water dissipation. Regarding those two parameters, this behavior is associated with a higher diameter of diatom particles when compared to clay soils. This bigger diameter influences the frictional characteristic of the soil samples, giving them sand-like properties for low confinement pressures. Indeed, the compressibility index decreases from 0.27 with a diatom content of 0% to 0.08when the diatom content are 60%. The tests evaluated the properties of soil-diatom mixtures with uncracked frustules showing a different behavior when compared to oedometer reports of diatom-soil mixtures.

Keywords: Diatom mixtures, Compressibility index, Diatom shape, Consolidation, Diatom liquid limit.

1. INTRODUCTION

Geotechnical characteristics of soft soils like diatomaceous soils are important to be characterized. Then the performance of engineering constructions could be assessed. Structures placed over these deposits may have considerable settlements due to the low bearing capacity of diatomaceous soils [1]. These diatomaceous soils are natural lacustrine and marine deposits that can be found in areas with high concentrations of phytoplankton and volcanic activity that promotes silica production. Countries that satisfy these requirements are typically near the Pacific fire belt, such as Colombia, Chile, and Mexico among others [2].

Diatomaceous soil deposits are conformed by fossilized frustules and fine-grained material. The frustules are the external siliceous shell that protects the unicellular algae against predators [3]. Diatom frustules are sedimented once their organic matter has decomposed, remaining in the soil throughout geological ages [4]. These microfossils generally have symmetrical shapes with a similar size to silt soil particles. The shape of the particles contains voids that cause high Atterberg limits when trying to characterize the material [5]. The content of diatoms, loading history, and stress level, modify the mechanical response of soil-diatom mixtures, changing their compressibility, shear strength, void ratio, and water content [6].

Diatomaceous soil mixture deposits behave as plastic materials when diatom content is above 50% [2]. Diatomite soil mixtures behave differently than standard cohesive soils. This particular situation influences the mechanical response of the mixture base con the content of diatoms. As an example, the Mexico city lacustrine with high diatoms content behaved differently than other areas during the 1985 Michoacan Earthquake. Additionally, compressibility is affected by diatomaceous contents [4]. Even though, diatoms have a bigger particle size than fine-grained soils the compressibility index increases for high diatomaceous contentment tested using high stress [1-3]. Even though the recent studies of soil diatoms, there is little evidence of the effect of diatomaceous content in the compressibility index of the mixtures tested at low stresses.

Diatomaceous soil has counterintuitive properties. The mechanical properties do not change following the principles of standard soils. For instance, diatoms have high water carrying capacity due to the shape of the frustule [2]. When these soils are classified, their behavior could be related to fine-grained soil properties but their behavior is similar to non-plastic materials. Therefore, this research aims to explore the effect of diatom content on the compressibility index at low confinement stresses.

2. RESEARCH SIGNIFICANCE

Laboratory tests were conducted in mixtures made of fine-grained soil with a varying percentage of diatom *Cyclotella Distinguenda* to characterize their compressibility, geotechnical properties, and index properties. The compressibility results of samples using the oedometer test are discussed in this investigation when the diatom frustules have not reached the yielding stress. This research contributes to the understanding of soil-diatom mixtures. The purpose of the research is to explore the mechanical behavior of soil-diatom mixtures at low confinements.

3. LITERATURE REVIEW

Test procedures and equations were based on ASTM specifications to compute the physical and index properties of samples. The specific gravity by water pycnometer, the water content, and the liquid limit were computed according to ASTM D854-02 (Method A) [7], ASTM D2216-19 (Method A) [8], and ASTM D4318-00 (Method A) [9], respectively. The oedometer test procedure was applied according to ASTM D2435-03 [10], and the calculations were based on the standard method "Taylor square root of time". This method estimates the time corresponding to the 90% primary compression point to calculate the coefficient of consolidation (Cv). [11]. The oedometer test was used to compute the compressibility parameters (i.e., Cc and Cv). Time (t) - deformation (s) curves were obtained during the test for each step of loading according to the diatom content (DC) on the mixture. Changes in the void ratio (Δe1), and the final void ratio (e1), were calculated using the initial dimensions of each sample. The data was used to compute the consolidation curve (σ' vs e) for each mixture. Finally, the compression index (Cc) and swell index (Cs) were obtained graphically by analyzing the consolidation curves.

The graphical method "square root of time" was implemented to define the time corresponding to a 90% primary consolidation (t_{90}),. After analyzing the t_{90} for each load increment on the corresponding diatom content, Cv was calculated with equation (1) [11]. Eq. 1 requires a time factor (Tv) that considers the flow velocity in the z-direction (v_z) for a one-dimensional consolidation test. This factor, presented by Shukla et al [15], assumes a constant initial excess pore water pressure throughout the depth of small samples. Tv value of 0.848 was selected for the 90% average degree of primary consolidation [11]. In Eq. 1, Hdr is the average longest drainage path during consolidation, and $\boldsymbol{t_{90}}$ is the time corresponding to the 90% primary consolidation.

$$Cv = \frac{Tv * (Hdr)^2}{t_{90}} \qquad (1)$$

4. MATERIALS

Cyclotella Distinguenda diatoms (D) and commercial fine-grained soil (S) were used to create the different soil mixtures. This kind of diatom is disc-shaped with central symmetry. The valve of the diatom frustule presents a circular center tangentially undulate with an external stria contour. These striae are conformed by porosities with an alveolar distribution pattern in the diatom contour. Furthermore, the smooth central area conforms between 30% to 50% of the valve face [12]. The SEM image of Fig. 1 shows the shape and initial condition of the tested diatom.

The Cyclotella Distinguenda has a circular center and an external striae ring. The central area presents undulations that range from slight to distinct. This taxon is commonly found in lakes and rivers, predominantly alkaline waters

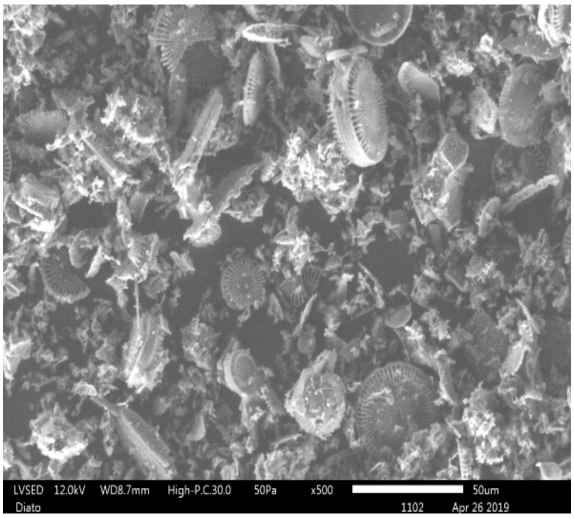

Fig. 1 Scanning electron microscopy (SEM) image of *Cyclotella Distinguenda* tested diatoms

Table 1 Soil-diatom mixtures

Mixture	Fine-grained Soil Content (%)	Diatomite Content (%)
1	100	0
2	80	20
3	60	40
4	40	60
5	0	100

Table 2 Specific gravity, median particle size, and Plastic Limit of soil-diatom mixtures

Mixture	Gs	(μm)	Plastic Limit (%)
1	2,65	1,2	23
2	2,58	-	32
3	2,51	-	Not Achieved
4	2,44	-	Not Achieved
5	2,32	8,5	Not Achieved

Table 3 Liquid limit, swell index, and compression index of soil-diatom mixtures

Mixture	Liquid Limit (%)	Cs	Cc
1	37	0,007	0,28
2	39	0,011	0,20
3	46	0,026	0,11
4	75	0,026	0,08
5	-	-	-

Physical properties for 5 different soil-diatom mixtures are summarized in Tables 1, 2, and 3. The specimens were prepared with diatom contents of 0%, 20%, 40%, and 60%. Median particle size (D_{50}) is reported in Table 2 just for pure materials, represented as 0% (S) and 100% (D). The liquid limit (W_L) of 100% of DC could not be measured due to the limitations present in ASTM tests.

The plastic limit test according to the ASTM D4318-00 specification was applied to each specimen. Values for samples 2, 3, and 4 are not reported because thixotropic properties compromised the results. For that reason, the liquid limits (W_L) shown in Table 3 are used as indicators of the water carrying capacity.

Particle size distribution for S and D was constructed using the hydrometer test according to ASTM D422-63 **[13]**. Fig. 2 shows the particle size distribution curve for S and D. Analyzing the D_{50}, W_P, and W_L of S, the particle size of the specimen can be considered as a passing material for sieve N° 200. Therefore, according to ASTM D2487-17, this soil can be classified as low plasticity clay (CL) **[14]**.

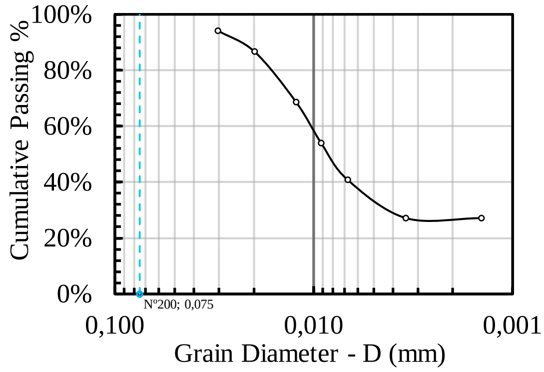

Fig. 2 Fine-grained soil particle size distribution

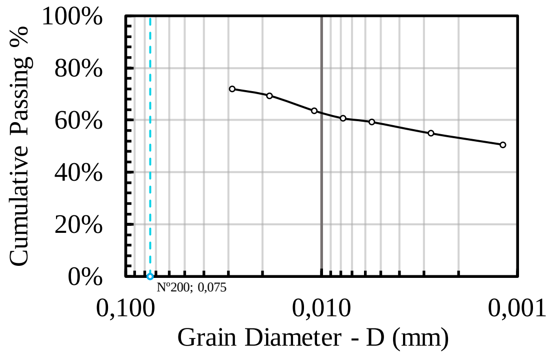

Fig. 3 Diatom particle size distribution

Fig. 3 shows the particle size distribution curve for the *Cyclotella Distinguenda* diatom (D). The D_{50} and W_L were used to characterize the diatoms using W_P = 0. Consequently, the three S-D mixtures were classified as sandy silt (ML).

5. PROCEDURE

The laboratory tests implemented in this research were focused on physical and mechanical characterization. Physical tests included water content by mass, Casagrande test (W_L test), plastic limit test, specific gravity by water pycnometer, and grain size distribution by hydrometer test. The mechanical test to evaluate compressibility was a one-dimensional oedometer test. Procedures for these tests were based on ASTM specifications.

The characterization of fine-grained soil and diatom specimens with physical tests is summarized in Tables 1, 2, and 3, Fig. 2, and Fig. 3. Four remolded samples were used for the one-dimensional oedometer test following diatom contents of 0%, 20%, 40%, and 60% by total dry mass. These samples were prepared with a water content of 1.5 times their Liquid Limit. This W_L was used to erase the geological pressure history of the samples **[15]**. In addition, the samples were preconsolidated to 0.45 kPa to stand the weight of the mechanism.

The consolidation process was developed in a conventional oedometer under saturated conditions. Samples were prepared with two porous stone discs at the top and bottom of the consolidation mold. The load increment ratio used for loading and unloading cycles presented the following stress steps: 4, 8, 15.5, 31, and 62 kPa. These stresses were selected because they are sufficiently lower compared to the yielding stress of diatom particles **[6]**. Loading and unloading cycles were completed in approximately 2 days. The end of primary consolidation was monitored with the Taylor Method – square root of time, defining enough values during tests to compute the t_{90}. As a result, the tested samples were 63.4 mm in diameter, 13.53 mm as a constant initial height, and a varying final height for each sample.

6. RESULTS AND DISCUSSIONS

Fig. 4 shows the liquid limit of the mixtures for different DC. The curve shows an increment in W_L as the diatom content increases. Consequently, the water storage capacity of the sample rises due to the intraparticle porosities within the frustules. This property explains the high W_L and low dry density on samples with higher diatom content **[16]**.

Despite the W_L increases at high DC, the W_P could not be determined with the ASTM D4118-00 specifications. Regarding the mentioned ASTM, its application evidenced the thixotropic properties of the remolded samples. This behavior justifies the indeterminacy of W_P, leading to presume that the mixture could behave as non-plastic **[16]**.

The oedometer test results were used to plot the samples' variation of height in time, distinguishing load increments and DCs. These results were analyzed to compute the consolidation curves presented in Fig. 5. In addition, the geologic pressure history of the specimens was erased to obtain the virgin compression curve for the samples with lower vertical stress steps **[15]**.

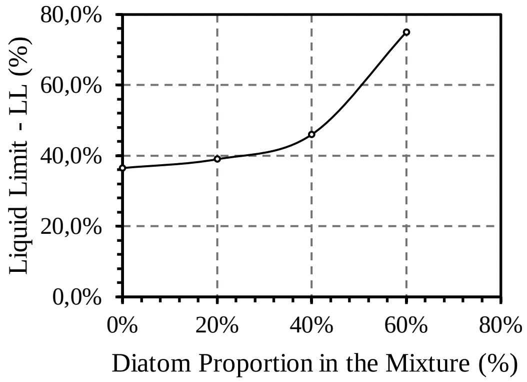

Fig. 4: Liquid limits of the tested samples

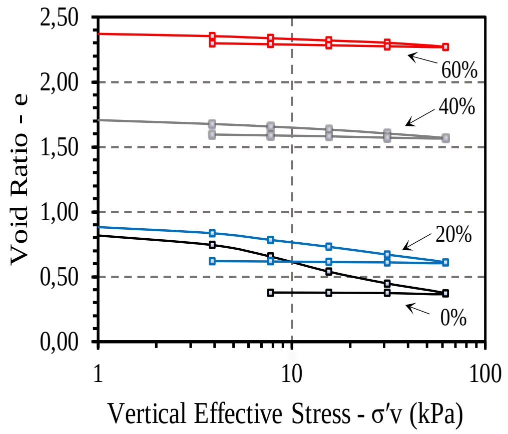

Fig. 5: Consolidation curves of tested samples

Fig. 5 shows an increment of e_o at high DC due to the intraparticle porosity of frustules. The following values of e_o were registered for 0%, 20%, 40% and 60% DC: 0.82, 0.90, 1.71 and 2.37 respectively. Moreover, the distance between the curves of loading and unloading cycles shows a reduction of compressibility at higher DC. Oedometer results of researchers such as Caicedo et. al **[4]** and Sonyok & Bandini **[6]**, used pre-consolidation stresses between 30 and 60 kPa, and a maximum stress step between 800 and 1000 kPa. Those testing values resulted in higher compressibility at higher diatom contents. The mentioned test reports differ from the values of this paper. Therefore, the reduction of compressibility at higher DC is reduced due to uncracked frustules.

Materials with 65% of diatom microfossils presented high frustule crushing at large stresses (up to 1600 kPa) **[6]**. Besides, the breakage of microfossils at higher stress levels depends on the frustules' composition, geometry, size, and orientation **[6]**. The applied short-term one-dimensional oedometer tests reached a maximum stress step of 62 kPa. For that reason, the tested specimens showed an initial gradual compression that did not generate a disturbance of the soil microstructure by cracking. However, the data presented some volumetric deformations due to particle reorientation within the void space **[6]**.

From Fig. 5 the Cc and Cs parameters were registered in Fig. 6 and 7 in terms of Cc – diatom content (%) and Cs – diatom content (%) respectively. Fig. 6 shows the compression index for the different diatom contents on samples. The following values of Cc were registered for 0%, 20%, 40% and 60% DC: 0.28, 0.20, 0.11, and 0.08 respectively. Those values show a reduction of Cc at higher DC due to uncracked frustules as formerly explained. The non-disturbance of the soil microstructure meant lower compressibility.

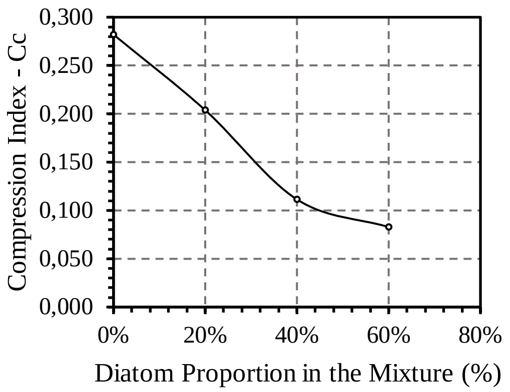

Fig. 6: Compression index of the tested samples

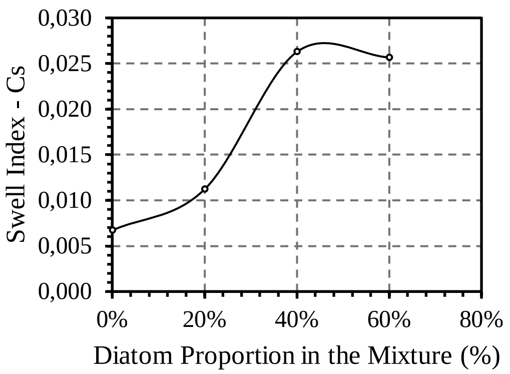

Fig. 7: Swell index of tested samples

The result presented in Fig. 6 suggests that the effect of diatom is similar to sand particles. The compressibility index reduces from 0.27 to 0.06. The frustules show a stiffer response for low confinement. The compressibility of the mixture increase when the yield stress of the frustules is reached. Many authors have recognized that the yield stress of the particles is over 1 MPa [14].

Fig. 7 shows the swell index with the following values for 0%, 20%, 40% and 60% DC: 0.007, 0.011, 0.026, and 0.026 respectively. The increment of Cs at higher DC justifies the high strength against compression due to the uncracked frustules. Also, the compressive strength of frustules presents elastic deformations under high compressive stresses, until they cracked. Hence, the increase of swell index is justified by the uncracked frustules and the recovering of elastic compression in some diatomite particles during the unloading cycle [6].

Fig. 8 shows the relationship between the liquid limit and the compression index. The effects of higher DC are seen in increments of W_L and decrements of C_c, as shown in Fig. 4 and 6 respectively. As a result, Cc decreases at higher W_L in the tested S-D mixtures. These results are based on the uncracked state of the diatom frustules. The increase of diatom content induces a higher compressive strength, despite the increase of void space. Otherwise, if the stress surpasses the elastic compression of the frustules, the behavior of the mixture could tend to an increment of Cc at higher W_L, due to the release of trapped water after diatom crushing **[4]**.

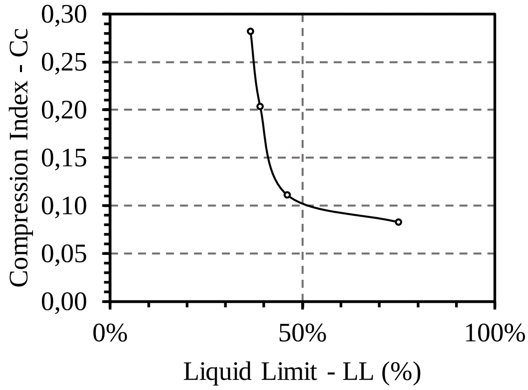

Fig 8: Relationship between Cc and W_L

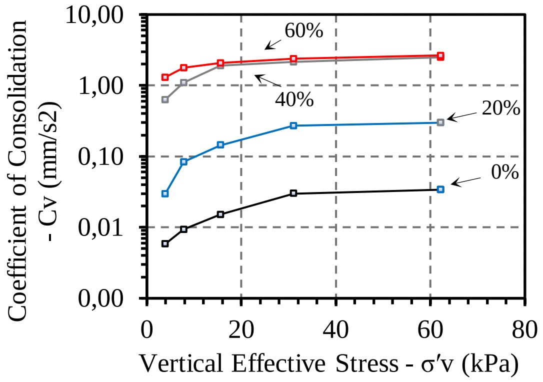

Fig 9: Coefficient of consolidation of tested samples

The results of Fig.8 show a different behavior from a similar study conducted by [16]. For low stresses, the frustules reduce de compressibility of clayey soils, whereas for high stresses the effect is the opposite as reported in [4,16]. The stress level should be considered when developing the relationships between water content and diatom content.

The coefficient of consolidation (Cv) was computed with the graphic method of the square root of time suggested by Taylor. Fig. 9 shows an increment of Cv at higher DC and higher vertical stresses. Comparing the Cv variation between DC of 0% to 20%, 20% to 40%, and 40% to 60%, the highest value corresponding to that of 20% to 40%. This is also seen in Fig. 5, with a higher increment of e_o associated with the sample of 40% DC. These data show that the tested fine-grained soil did not get higher fabric modifications at diatom contents up to 20%.

On the other hand, the increase of Cv at higher diatom content is supported by a faster pore pressure dissipation due to a higher porosity of the mixture. The high content of voids in diatom frustules influences the water carrying capacity of the mixture. Hence, the porosity of the mixtures, caused by the intraparticle and interparticle voids, aids the release of water pressure. In addition, this behavior makes the consolidation process faster at higher DC, considering that the behavior of uncracked frustules results in less volumetric deformations on the samples.

7. CONCLUSIONS

The increase of DC in the samples modified its microstructure, getting larger intraparticle porosities due to the voids within diatom frustules. Moreover, diatoms had a particle size bigger than clayey soils and micropores. Those characteristics justified the increased W_L, e_o, and the low dry specific gravity of samples at higher DC.

The maximum stress step applied in oedometer tests was lower than the one reported in the references of Caicedo et. al **[4]** and Sonyok & Bandini **[6]**. Consequently, the yielding stress of the frustules was not reached. The results suggest that C_c reduces as DC increases, which opposes the mentioned oedometer reports. Indeed, the compressibility index is 0.27 for a diatom content of 0% whereas 0.08 for a diatom content of 60%. In diatom-soil mixtures, an additional volumetric deformation is expected when the yielding stress of the frustules is reached.

The compressibility index of soil-diatom mixtures (S-D) had a reduction at higher diatom content (DC) due to an improvement in their compressive strength. The improvement depends on the uncracked condition of diatom frustules. An increment of diatom content resulted in lower compressibility due to the higher resistance of hard siliceous frustules.

The swell index of the tested samples had an increment at higher DC due to the recovering of elastic compression in some diatomite particles. Hence, some volumetric deformations in the unloading cycle were explained by short compressions of the interparticle and intraparticle porosities of the mixtures.

The counterintuitive properties of soil-diatom mixtures revealed a different behavior than in the research reported in [1-4]. For common soils, the compressibility increase as the liquid limit of the material does. This fact agrees with the compressibility measured in [1-4], but when the frustule yielding is achieved. The results of this research suggest that for stresses below the particle breaking the compressibility index reduces as the liquid limit of the material increase. Therefore, the correlations found in the literature had to be careful analysis when diatoms are in the soil.

The coefficient of consolidation had an increment at higher DC due to faster pore pressure dissipation. This could be explained by the diatom porosities, which had a bigger size than the ones in the fine-grained soil. That microstructure modification on samples resulted in more drainage that accelerates the consolidation process.

The results of this investigation showed a different behavior of the soil-diatom mixtures compared to other oedometer tests. The measured characteristics become an important contribution to the state of art of soil-diatom mechanical characteristics. For future investigations is important to consider the effect of diatom shape on the compressibility of the mixtures. Diatoms have different shapes with different stiffness that can lead to unlike behavior to the one reported in this document. Additionally, future works could analyze the effect of diatoms on the yield surface of the material, due to the frictional characteristics of diatoms particles

8. ACKNOWLEDGMENTS

This research was supported by the Universidad de los Andes and the Poligrant program of the Universidad San Francisco de Quito.

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