Characterization of the Granulometric Phases of Diatomaceous Soil

Caracterización de las fases granulométricas del suelo diatomáceo

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Abstract

The research proposes a methodology to separate the phases (particle size) of Colombian diatomaceous soil after placing it in columns with different aqueous media (water and hexametaphosphate) and subjecting it to agitation and sedimentation processes over periods of 3 and 7 days. Samples were extracted from each column at different heights, revealing distinguishable physical properties in terms of specific gravity, consistency, hydrometry, scanning electron microscopy (SEM), and oedometer consolidation tests. Some of the most efficient techniques for particle separation involve displacing solid particles within fluid media. Results showed a reduction in liquid and plastic limit values with deeper sections, that is, when the material was thicker. The liquid limit values for the columns at 3 days were, on average, higher than those at 7 days. The highest Gs values were found in the material at section S1 of each column, that is, the samples with the lowest liquid limit and largest particle size. In all cases, S1 of each column exhibited hydrometry curves with lower values than the other sections; that is, it reported larger particles. In the separation of particles by size, it was concluded that time and multiple agitation cycles had a greater impact, while the type of aqueous medium did not. In the samples treated with water, the void ratio increased, while the opposite occurred with the samples containing hexametaphosphate. The initial and final void variations did not exceed 10 %. SEM results did not allow for changes in particle size distribution among the different sections of each column to be recognized; the effect of sedimentation was more clearly evidenced in the hydrometry curves.

Keywords
aqueous media; diatomaceous soil; geotechnical characterization; particle size distribution; sedimentation column

Resumen

La investigación propone una metodología para separar las fases (tamaño de partícula) del suelo diatomáceo colombiano tras colocarlo en columnas con diferentes medios acuosos (agua y hexametafosfato) y someterlo a procesos de agitación y sedimentación durante periodos de 3 y 7 días. Se extrajeron muestras de cada columna a diferentes alturas, revelando propiedades físicas diferenciables en términos de gravedad específica, consistencia, hidrometría, microscopía electrónica de barrido (MEB) y ensayos de consolidación edométrica. Algunas de las técnicas más eficientes para la separación de partículas consisten en desplazar partículas sólidas dentro de medios fluidos. Los resultados mostraron una reducción de los valores de límite líquido y plástico en las secciones más profundas, es decir, cuando el material era más grueso. Los valores de límite líquido para las columnas a 3 días fueron, en promedio, mayores que los de 7 días. Los valores más altos de Gs se encontraron en el material de la sección S1 de cada columna, es decir, en las muestras con menor límite líquido y mayor tamaño de partícula. En todos los casos, la sección S1 de cada columna presentó curvas hidrométricas con valores menores que las demás secciones; es decir, reportó partículas de mayor tamaño. En la separación de partículas por tamaño, se concluyó que el tiempo y los múltiples ciclos de agitación tuvieron un mayor impacto, mientras que el tipo de medio acuoso no lo tuvo. En las muestras tratadas con agua, el índice de vacío aumentó, mientras que en las muestras con hexametafosfato ocurrió lo contrario. Las variaciones inicial y final de vacíos no superaron el 10 %. Los resultados de MEB no permitieron reconocer cambios en la distribución del tamaño de partícula entre las diferentes secciones de cada columna; el efecto de la sedimentación se evidenció con mayor claridad en las curvas de hidrometría.

Palabras clave:
medios acuosos; suelo diatomáceo; caracterización geotécnica; distribución del tamaño de las partículas; columna de sedimentación

Citation [IEEE]:
Pendiente

Introduction

This research develops a methodology to separate the phases (particle sizes) of Colombian diatomaceous soil (DS), applying the principle of sedimentation, in order to analyze the physical and mechanical characteristics of each phase. The properties of DS are little known [1] and are classified within the fossiliferous soils. DS is a microstructured natural material with particular properties resulting from its physical and mechanical relationships with clays and water. Therefore, it must be investigated in depth, as its use in geotechnical and environmental engineering is scarce [2]. In each phase, hydrometry, consistency, oedometric consolidation, and scanning electron microscopy (SEM) tests were conducted. The study variables were particle size, sedimentation time (3 and 7 days), and the aqueous medium (water, hexametaphosphate, or a combination of both).

Soil is the combination of elements of different sizes, and its behavior is a consequence of the interaction between these particles and the surrounding environment [3]. In a medium with particles of various sizes, densities and shapes, the separation of its fractions can occur by applying different type of processes (decantation, centrifugation) [4], taking advantage of the force of gravity acting on the particles suspended in a liquid, which allows the coarse fraction to precipitate and keep the finer one in suspension [5].

Diatoms are unicellular, photosynthetic, microscopic algae of oceanic or lacustrine origin, belonging to the phytoplankton population, whose sedimentation has led to the formation of diatom sediments and diatomites since the Cretaceous, Eocene, and Miocene periods [6], [7].

Diatomaceous soils are of sedimentary origin. They are the product of the organic decomposition of diatoms and the precipitation of their skeletons, which reach the bottom of oceans and lakes [8]. Diatomite is a rock formed by the compaction of fossil sediment (frustules). These are porous and of low density [2]. Some authors report a frustule content of diatomite exceeding 50 % by weight and a porosity greater than 70 % [9].

Frustules are the skeletal structures of diatoms and are characterized by their high silica content (approximately 90 %) [9], unique morphology, and high water absorption capacity [2], [3], [7]. Soils combined with diatom frustules store significant amounts of water and simultaneously increase their resistance to vertical tension, proportionate to the diatomite content [3].

From a soil mechanics perspective, research has been conducted on soils with the presence of diatomaceous frustules to understand their physical characteristics and mechanical responses [3], [8]. A relationship exists between shear strength and compressibility, influenced by the size and content of diatomaceous particles within a soil mass. The high degree of compressibility of DS is due to the rupture of the frustules [8]. Expansive clayey materials influence the mechanical properties of DS [10]. DS have high values in geotechnical properties such as water content, void ratio, porosity, permeability, compressibility, consistency limits, and undrained shear strength [11].

Frustules are microfossils with a density lower than that of cement, with records as low as 767 kg/m^3 [12]. DS are mainly silts, originating from frustules or fractions thereof [2]. Some DS (Bogotá) with representative contents of frustules report high friction angles (close to 45°), simultaneously with high values of void ratio, compressibility index, and liquid limit (up to 200 %) [13]. This behavior is atypical. According to classical soil mechanics, as the plastic index increases, the friction angle decreases. The classification of DS under the Atterberg limits and according to the particle size is contradictory. Under constant stresses applied in consolidation tests, plastic deformations occur due to the breakage of the frustules, and compression indices similar to those of granular material are recorded. Diatomite, as a modifying agent, significantly increases the characteristics of soils in terms of texture and resistance [14].

The size and shape of soil particles influence their resistance to relative displacement. Generally, a greater mechanical response is present at scales greater than 0.075 mm. According to conventional standards, fine soils (nano- and micro-scales) have low resistance. However, DS (micro-scale) records high values of mechanical resistance. This response depends on the state of conservation, shape, and quantity of frustules [15].

Particle Separation Techniques

There are methods for separating fine particles at an industrial level, including those used for powders [16]. All granular material mixed with a liquid separates due to differences in size and density. The efficiency of segregation depends on the cohesion and conduction velocity of the particles, as well as the action of gravity [17]. Research has been conducted on the particle size distribution of kaolin minerals using settling techniques. Particles with sizes smaller than 38 µm exhibited colloid properties and were found to contain iron oxide. Additionally, kaolin improved its cleaning degree, and the contamination of salts absorbed by the fine particles was reduced [18].

Some of the most efficient techniques for particle separation in granular media involve studying the effects of solid particle displacement on fluid dynamics. It was found that larger particles are segregated toward the outside of the fluid, while smaller particles are segregated toward the center [17]. Some granular material separation methods utilize intermediate-specific-weight liquids as suspension media, which are efficient in extracting fine, floating materials [19].

In sedimentation processes for particle separation, a general first step is the induction of agitation and mixing through violent and irregular movements in the fluid mass, ensuring homogeneity, breaking up clumps, facilitating a precipitation route for the heaviest particles, and maintaining a constant concentration [20].

Sedimentation is a process used for the separation of suspended particles. The heaviest particles self-classify by the action of gravity [21]. Those with lower density can be removed by filtration [22]. Four types of sedimentation are recognized, depending on the interaction between particles, their concentration, physical properties, and deposition mode [21].

Type 1 sedimentation (discrete elements) is associated with a few particles in a suspension of solids of low concentration, characterized by the lack of interaction among suspended particles [21]. This type of sedimentation is characteristic of desanders and pre-settlers before filtration.

Type 2 sedimentation (flocculating particles) occurs in highly diluted suspensions, where particles agglomerate during sedimentation and settle more quickly as their mass increases [21].

Type 3 refers to suspensions with intermediate concentrations. The force among particles slows the settling of other particles around them [22].

Type 4 occurs when, due to a greater structural concentration of particles, compression settlement takes place. This phenomenon occurs due to the weight of the particles added during settlement and takes place in the lower, deep layer [21], [22].

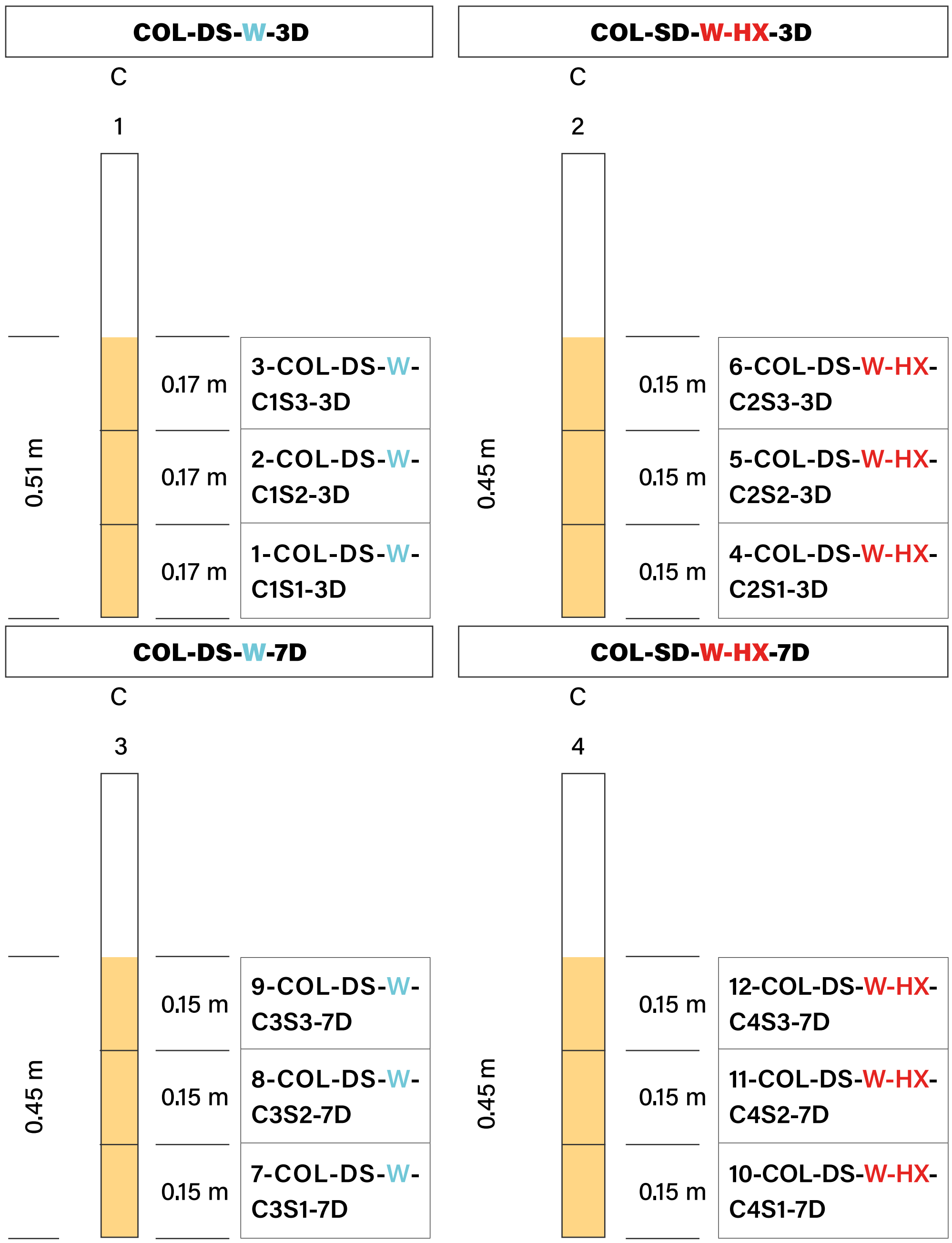
Materials and Methods

The study involved placing Colombian DS samples in separation columns under different aqueous media (water and hexametaphosphate) and subjecting them to agitation and sedimentation processes at various times (3 and 7 days). Samples were extracted from each column at different heights, which, due to the sedimentation process, exhibited distinct physical properties. Each fraction was subjected to hydrometry, SEM, and oedometer consolidation tests.

The samples were coded according to mixing and treatment variables, as follows: **COL** (Colombian species), **DS** (diatomaceous soil), **W** (Water), **C1-C2-CE-C4** (Columns No. 1, 2, 3, or 4), **HX** (Sodium hexametaphosphate), **S3-S2-S1** (section number in the column), **3D-7D** (3 or 7 days). See Figure 1.

Hexametaphosphate is a dispersant derived from Graham's salt and is obtained from phosphoric acid. These are white solids that dissolve in water. This is used to separate the different constituent particles of SD. It is a deflocculant that prevents agglomerations (lumps) when mixed with water [23]. Sodium hexametaphosphate was added in a proportion of 4 % according to the amount of water.

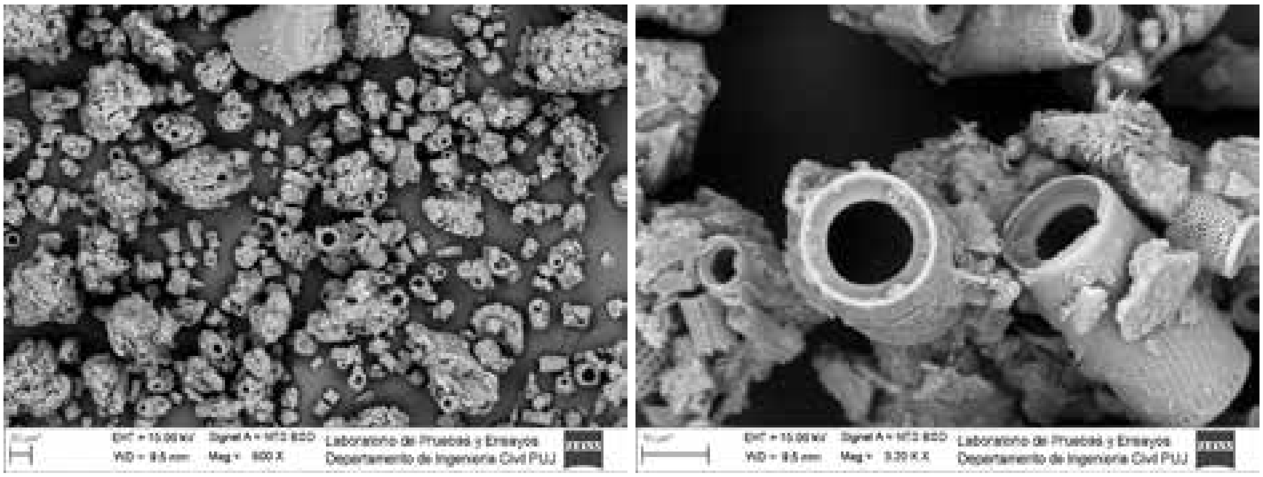
Figure 1. Soil Distribution Diagrams inside the Columns

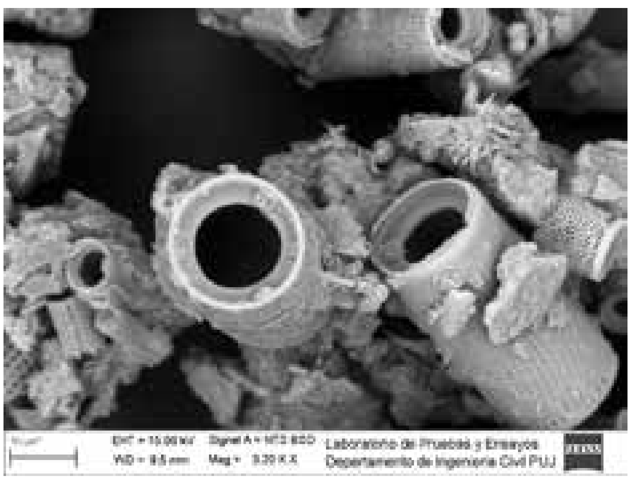


Diatomaceous Soil

The DS used is of Colombian origin, and its predominant species is Aulacoseira Granulata II. Figure 2 illustrates the morphological characteristics of the species, while Table 1 presents the results of the basic geotechnical characterization.

Figure 2. Morphological Characteristics of Diatomaceous Soil Frustules





Striae orientation:	Parallel
Medium:	Plantonic
Ecosystem:	Lacustrine
Organization:	Uniseriate
Pore structure:	Areola
Maximum length (µm):	38.66
Medium length (µm):	13.58
Minimum length (µm):	5.19
Maximum diameter (µm):	23.97
Medium diameter (µm):	11.51
Minimum diameter (µm):	4.04

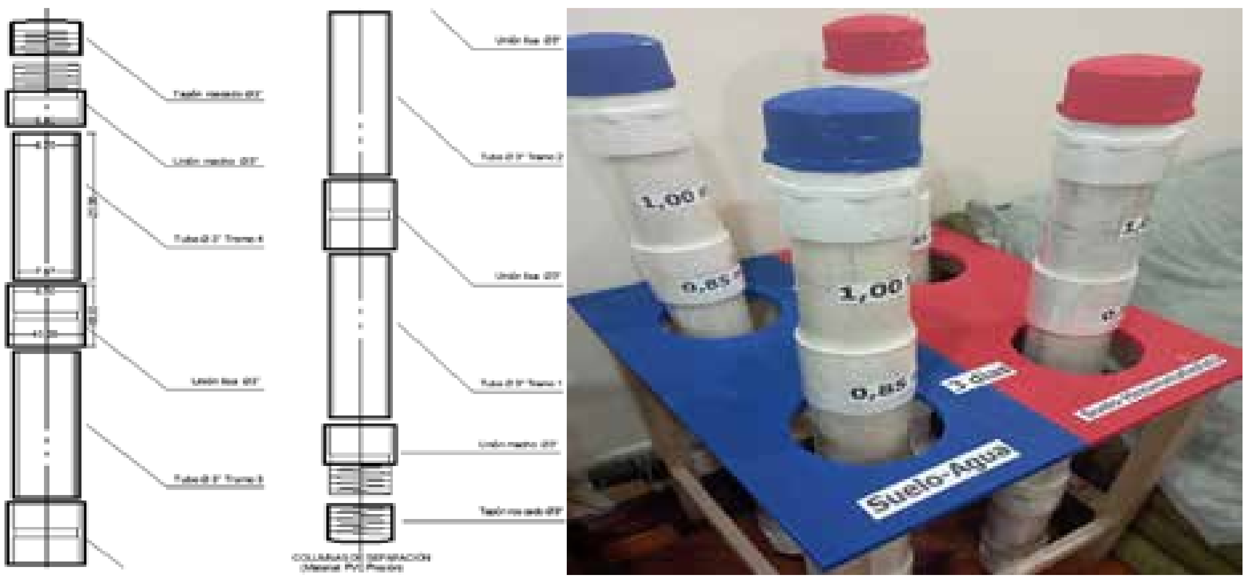
Table 1. Basic Geotechnical Characterization of Diatomaceous Soil

Criterion	Unit	Quantity
Specific surface area	cm^2/gr	2,600
Specific gravity	-	2.43
Liquid limit	%	134.97
Plastic Limit	%	75.21
Plasticity Index	%	59.76
Clay fraction <2µm	%	12.8

Particle Separation Column

Four separation columns were manufactured, each 3″ in diameter and 125 cm long. Each column has four sections that can be connected using fittings (connectors). Two columns were used for DS treatment with water (blue) and two for the hexametaphosphate treatment (red) (Figure 3).

Figure 3. Separation Column Characteristics



As part of the sedimentation process, it was necessary to define the volumetric conditions that would ensure the proper dispersion of the soil within the liquid medium (water or water + 4% hexametaphosphate). The volume ratio between soil and liquid was translated into column centimeters. Two ratios (soil: liquid) were considered: 1:2 and 1:3. Based on mixing homogeneity, separation efficiency, and ease of column operation, the optimal ratio was determined to be 1:2. The dosage applicable to each column is shown in Table 2.

Table 2. Basic Geotechnical Characterization of Diatomaceous Soil

	Column No.			
	1	2	3	4
Weight of diatomaceous soil (g)	1,780		1,570	
Height of diatomaceous soil (cm)	51		45	
Weight of diatomaceous soil + hexametaphosphate (g)		1,570		1,570
Height of diatomaceous soil + hexametaphosphate (cm)		45		45
Weight of water (g)	4,960	4,370	4,370	4,370
Height of water (cm)	102	90	90	90

Process Stages

With the previously defined dosage (1:2), the agitation stage commenced, consisting of manual rocking in thirty repetitions that simulated wave movements. Agitation was performed only once in the columns that would sediment for three days. The columns that were projected to sediment for seven days were agitated every twenty-four (24) hours, in the same number of repetitions, and were left to settle without movement for three additional days. Sedimentation began once agitation was completed in each of the columns. Once the sedimentation time was complete (3 or 7 days), the liquids were removed, and soil samples were subsequently extracted from the upper, middle, and lower sections (Figure 4) to proceed with separate characterization and analysis.

Figure 4. Visual Identification of the Diatomaceous Soil Phases Sedimented in the Columns

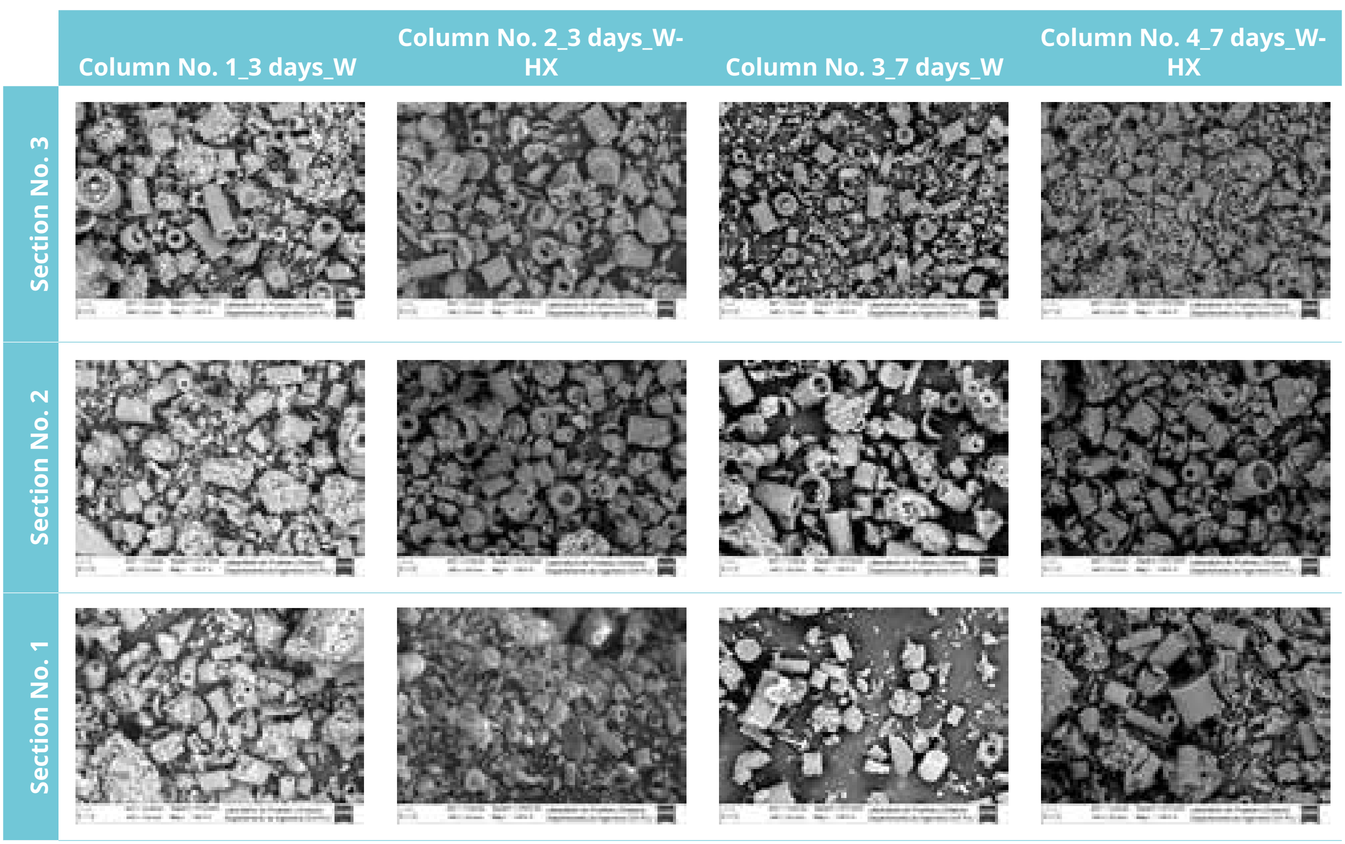
Laboratory Tests and Trials

In this study, twelve (12) diatomaceous soil samples were analyzed and selected based on the liquid medium (water or water + hexametaphosphate), the settling time (3 or 7 days), and the location within the columns. The following procedures were applied to each sample: soil liquid limit (INV-E-125-13), soil plastic limit and plasticity index (INV-E-126-13), particle size analysis using a hydrometer (INV-E-124-07), determination of the specific gravity of solid soil particles and mineral filler using a pycnometer with water (INV-E-128-13), and one-dimensional soil consolidation (INV-E-151-13). Additionally, observations were carried out using SEM at zooms ranging from 1600× to 6400×, in conjunction with the analysis of chemical composition by energy-dispersive X-ray spectroscopy (EDS).

Results and discussion

First, the results of the microscopy tests are presented, allowing the geotechnical properties to be associated with the observable physical characteristics (see Figure 5).

Figure 5. SEM Results for the Different Phases of Each Column



Images of the different phases of each column are presented; for comparison purposes, a zoom level of 1600× was selected. From the results, it can be observed that in most cases, when comparing a sample treated with water and one treated with hexametaphosphate, for the same time and section, the sample treated with water reveals larger clusters. Therefore, the dispersing effect of hexametaphosphate is efficient in DSs. The records of the columns treated with water (Columns 1 and 3) exhibit greater brightness and sharpness in the microscopy details. It is understood, then, that the presence of hexametaphosphate affects the surface reflection, even when the surface has been coated in gold. The SEM results of this research do not allow for the recognition of changes in the particle size distribution among the different sections of each column; that is, the effect of sedimentation is validated more clearly in the hydrometry curves. A clear cleaning pattern is not recognized (absence of fossil fragments). In all sections, whole frustules of different sizes were identified, as well as remains inside the cylindrical structures.

Consistency Limits

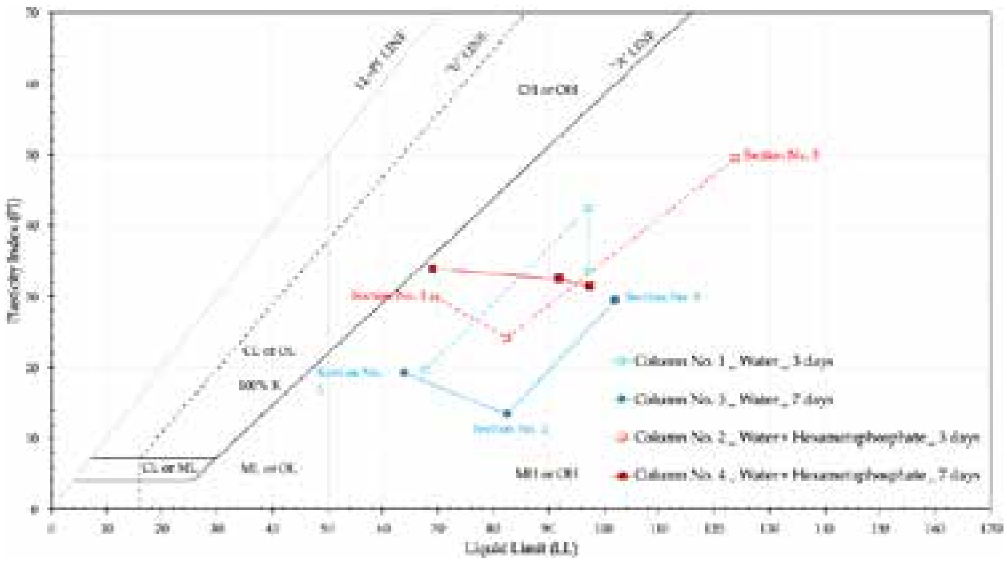
In all cases, a reduction in the liquid limit (LL) and plastic limit (PL) values is observed as the section is located deeper; that is, lower values are reported when the material is thicker. However, the plasticity index (PI) does not follow this trend (see Table 3). The average LL values are higher in the columns that include hexametaphosphate. The average LL value is higher in the columns settled for three (3) days. The highest average PL value (62.13) was obtained in the column settled for seven (7) days with water, and the lowest (53.62) was obtained at the same time but with hexametaphosphate. The lowest average PI value (20.66) was obtained in the seven-day sample with water. Regarding sedimentation time, the LL values for the 3-day columns are, on average, higher than those for the 7-day columns. The same behavior is observed in the PI.

Table 3. Consistency Results for the Different Phases of Each Column

Column No.	Medium	Section No.	Sedimentation time (days)	Liquid limit	Plastic limit	Plasticity index
1	W	3	3	97.28	63.81	33.50
		2	3	97.27	54.67	42.60
		1	3	67.84	48.27	19.60
2	W+HX	3	3	123.67	74.15	49.50
		2	3	82.47	58.26	24.20
		1	3	69.57	39.68	29.90
3	W	3	7	102.09	72.70	29.40
		2	7	82.52	69.09	13.40
		1	7	63.80	44.61	19.20
4	W+HX	3	7	97.52	66.01	31.50
		2	7	92.11	59.61	32.50
		1	7	69.02	35.24	33.80

The representation of the fractions in each column on the plasticity chart indicates that all samples are categorized as MH or OH, with some parallelism to the "A-line." In all cases, an inflexion point is observed associated with Section No. 2 of each column. See Figure 5.

Figure 6. Location of the Phases of Each Column in the Plasticity Chart



Specific Gravity

It is observed that the highest Gs values correspond to the material in section S1 of each column (Table 4), that is, the samples with the lowest liquid limit and largest particle size. For mixtures with water, a decrease in Gs values is evident as the settling time increases. The opposite behavior occurs in the mixtures that include hexametaphosphate. The average values for the different columns do not vary by more than 3.6 %.

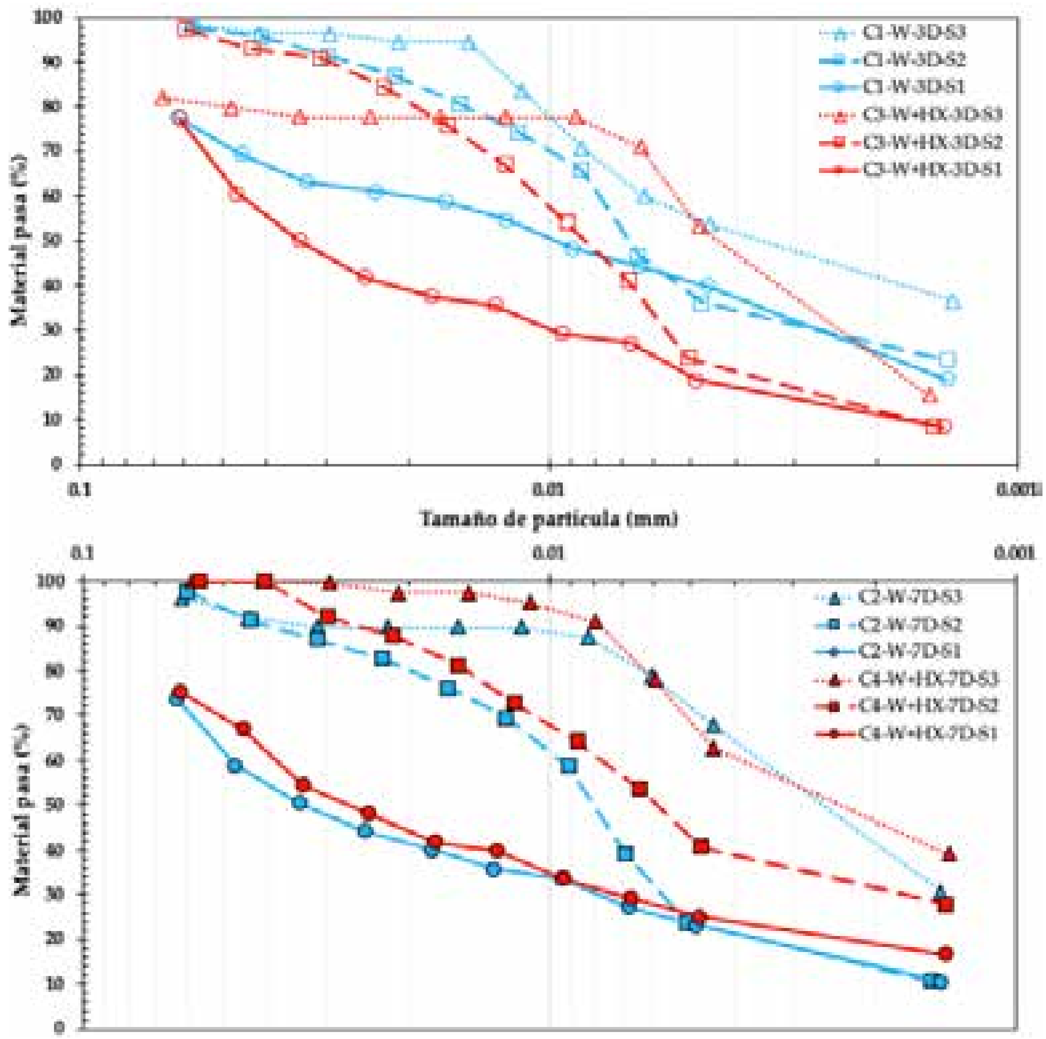
Table 4. Specific Gravity Results for the Different Phases of Each Column

Column No.	Medium	Section No.	Sedimentation time (days)	Specific gravity
1	W	3	3	2.31
		2	3	2.36
		1	3	2.41
2	W+HX	3	3	2.13
		2	3	2.26
		1	3	2.44
3	W	3	7	2.21
		2	7	2.24
		1	7	2.41
4	W+HX	3	7	2.26
		2	7	2.32
		1	7	2.43

Hidrometry

The particle size distribution results are categorized based on the settling time (3 and 7 days). In both conditions, the samples treated with water are differentiated from those that included hexametaphosphate. The curves for the sections of each column are also distinguished. See Figure 7.

Figure 7. Particle Size Distribution by Hydrometry. Top: three-day distribution in water and water + hexametaphosphate. Bottom: seven-day distribution in water and water + hexametaphosphate

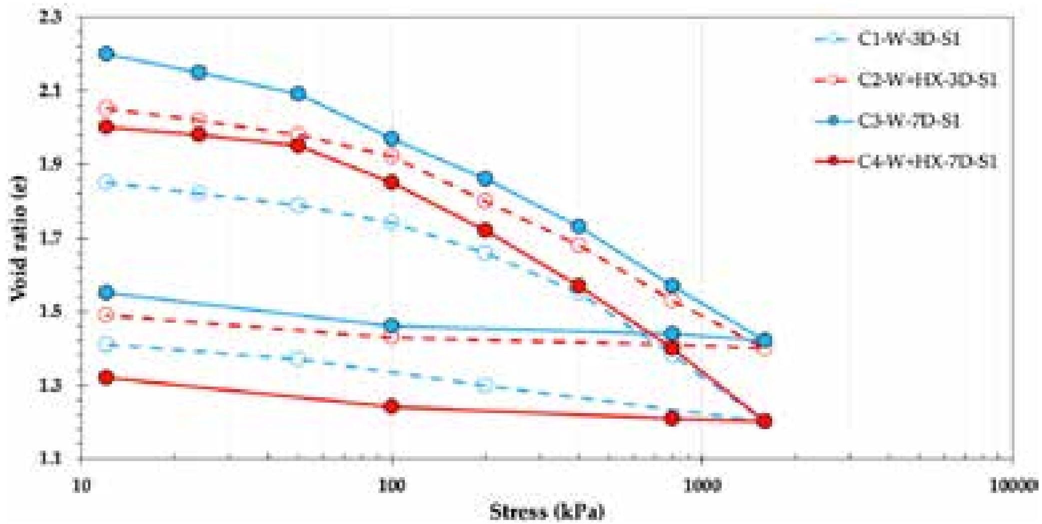


In all cases, Section No. 1 of each column is observed to be located below the curves of the other sections, meaning it presents larger particles. In the 7-day sedimentation, the curves of the different sections are clearly differentiated, and the behavior of both columns (W and W+HX) is quite similar. In the 3-day sedimentation, a crossover between the curves is observed. From this, the effect of time and multiple agitation cycles is more pronounced, while the type of aqueous medium has a lesser influence.

Compressibility

Consolidation tests were performed only on samples from Section 1 of each column, that is, on the deepest sections and those with the lowest material content below 2 μm. This was done to demonstrate the volumetric variation due to the breakage of a greater number of well-preserved frustules. See Figure 8.

Figure 8. Compressibility Curves in Section No. 1 of Each Column



A shift in the curves is observed over time, ranging from 3 to 7 days, for both aqueous media. In the case of water (W), there is an increase in the void ratio values; the opposite is true for the hexametaphosphate samples. Even though the curves shift, the initial and final void variations do not exceed 10 %. It is concluded that the samples from the sections with thicker material, obtained by different methods, exhibit similar compressibility behavior. The values of the compression (Cc) and swelling (Cs) indices for the different conditions are presented below (Table 5).

Table 5. Compression and Swelling Indices for Each Section and Column

Condition	C_c		Condition
C1-W-3D-S1	0.41	0.43	C2-W+HX-3D-S1
C3-W-7D-S1	0.43	0.64	C4-W+HX-7D-S1

Condition	C_s		Condition
C1-W-3D-S1	0.12	0.04	C2-W+HX-3D-S1
C3-W-7D-S1	0.04	0.05	C4-W+HX-7D-S1

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