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Multivariate analyses of the index properties of Diatomaceous Soils

Análisis multivariado de las propiedades índice de los Suelos Diatomáceos

[Daniel Alberto] [Zuluaga Astudillo]
[Programa de Doctorado en Ingeniería], [Universidad Militar Nueva Granada], [Colombia], [est.daniel.zuluaga@unimilitar.edu.co]
[Programa de Especialización en Diseño y Construcción de Vías y Aeropistas], [Escuela de Ingenieros Militares], [Colombia], [daniel.zuluaga@esing.edu.co]

[Carlos José] [Slebi Acevedo]
[Centro de Estudios Geotécnicos], [Escuela Colombiana de Ingeniería Julio Garavito], [Colombia], [carlos.slebi@escuelaing.edu.co]

[Juan Carlos] [Ruge Cárdenas]
[Programa de Doctorado en Ingeniería], [Universidad Militar Nueva Granada], [Colombia], [juan.ruge@unimilitar.edu.co]

ABSTRACT:

Overburden stresses, dissolution, and predation induce diatom frustules deterioration. The available literature focuses on characterizing some specific place's diatomaceous soils (DS) without delving into the simultaneous incidence of the frustules' content, characteristic shape, and deterioration level. This research analyzed three different origins of DS (Colombian COL, Mexican MEX, Peruvian PER) and morphological conditions (cylindrical, disk, elongated). The deterioration state was controlled by applying a mechanical crushing protocol. A numerical quantifier called "Conservation Index" was generated. It was concluded that the fossil shape affects the breakage susceptibility, particle size distribution, and surface area. The soil's Gs reduce with higher DS content (COL, MEX), the opposite of PER DS. The Liquid Limit (LL), Plastic Limit (PL), and Plasticity Index (PI) increase with a higher DS content for any origin. The highest LL and PL are observed in the MEX DS (flat disc shape), different from the initial water storage capacity expectation COL DS (cylindrical shape). As the deterioration condition increases, the PI is reduced in all species, and the COL and PER DS present a reduction in the LL and PL. A multivariate analysis was conducted using the Principal Component Analysis (PCA) method. The results showed that the LL and PL are significantly correlated, followed by the clay fraction content (CF) and, finally, by the Specific Gravity (Gs).

KEYWORDS: Diatomaceous soil, frustule deterioration, index properties, multivariate analysis, species characteristic shape.

1. INTRODUCTION.

Sedimentation processes of microalgae frustules generate Diatomaceous Soils (DS) and diatomites (Flower, 2013) (Arenaldi & Ovalle, 2019) (Mejía et al., 2016). During accumulation, these are affected by causes such as total or partial dissolution (Warnock & Scherer, 2015), fracturing due to transportation, pH variation, crushing due to geostatic stresses, proximity to geological fault zones, the activity of predatory species of diatoms (Finkel & Kotrc, 2010), water currents, thermal variation, reproduction rate, among others. Consequently, it is highly probable to find DS with frustules of different sizes and deterioration levels.

The proportion of diatomaceous skeletons that reach the seabed without being dissolved is minimal (<5%), given the susceptibility of amorphous silica (the main component of frustules) to seawater (Locat & Tanaka, 2001). The dissolution is the product of taphonomic and ecological processes and is enhanced in the most superficial part of the water bodies (Warnock & Scherer, 2015), given the exposure to hydrodynamic shear stresses (Karp-Boss et al., 2014). The preservation of frustules is possible for the larger species. Higher proportions could accumulate if the sedimentary basins were shallower or if the sedimentation rate were higher (H. Tanaka et al., 2001a).

In the superficial layer of some clayey deposits with traces of diatoms, partially dissolved fossils have been found with apparent reprecipitation on the surrounding aggregates, which could contribute to the structural cementation of the soil (H. Tanaka et al., 2001a). The shape of the frustules (whole, fragmented, elongated, rounded) influences the behavior of the soil (Arenaldi et al., 2019). Diatomaceous fragments contained in clayey matrices may behave differently from diatomites, which are composed almost exclusively of frustules.

The DS are mostly composed of pure biogenic opal, with some level of mechanical alteration or fragmentation. Due to natural variations, changes in the proportions of fractured and intact diatoms, organic matter, and sands are to be expected (Wiemer & Kopf, 2016). The microscopy technique is the direct method to validate the frustules' breakage state and their accommodation within a soil mass (Arenaldi et al., 2019).

The fracturing of the frustules is dependent on the order, specie, shape, and age of the diatoms. Ref. (Wiemer & Kopf, 2016) highlight the lack of understanding of the relationship between the fracture of the frustules and the increase in geostatic stresses. With minor changes in the geometry of the frustules, at a given geostatic stress, the amount of retained water is almost constant. The representative variation is evidenced with depth (H. Tanaka et al., 2001a). Investigations in marine geological processes have shown a higher content of diatoms as the strata deepen (Wiemer et al., 2017).

The intraskeletal pore space of the frustules is always connected to other voids in the containing soil (H. Tanaka & Local, 1999). The application of normal stresses causes individual breakage of the particles, resulting in a permanent decrease in the water storage capacity (Rajasekaran, 2006). The pore size distribution of the frustules walls is dependent on the specie and type of diatom. When the consolidation stress is greater than the yield stress, the frustules break, which implies that the skeletal pores collapse (Hong et al., 2006).

Hollow, rough, and interlocked microfossils alter soil index properties and behavior (Rajasekaran, 2006). The alteration degree depends on the extent, state, type of frustules, depth, and environment (ocean or lacustrine) at which the DS is located.

Water within skeletal and intraskeletal pores is retained by suction. With the breakdown of microfossils, the pores are reduced, therefore, the effects of the index properties vanish (H. Tanaka & Local, 1999). When fossil remains are fragmented, intraskeletal pore families are found to be minimal (H. Tanaka et al., 2001a).

The high Liquid Limit (LL) values presented by soils with the presence of diatom microfossils are explained by the large intraparticle pores inside the frustules, in which a large amount of water is stored. This volume is additional to the interparticle pore space and does not affect particle-particle interactions (Al Shatnawi & Bandini, 2019).

An increase in the consistency limits of different DS is observed as the fossil content increases, particularly in the LL (Caicedo et al., 2018). Although the volume of water stored in the pore networks of the frustules is considerable (with a higher percentage of diatoms), it does not alter the index properties, given the similar slopes presented by the LL and Plastic Limit (PL), that is no change in plasticity (Xu et al., 2022). The index properties of the DS are altered depending on the concentration of microfossils, due to the geometry and porosity of the frustules that enhance the amount of water stored (Locat & Tanaka, 2001) (Arenaldi et al., 2019) (Ruge et al., 2019) (Zhang et al., 2013). However, there is not enough literary report that allows to recognize the variations in the consistency limits depending on the diatom specie, its characteristic shape or its deterioration level.

Storage and absorption potential must be understood on two scales. The first, in the total volume generated by the fossil (micro dependent on the shape). The second, in the volumes of the distinctive pores of the frustules (nano dependent on the biological processes of the species) (Hong et al., 2006). Although the DS report great absorption, the water is encapsulated and does not affect the connectivity between frustules or their fragments. Certain diatomaceous deposits have low bulk density and high moisture content, due to the hollow structure of the frustules where water is stored, which leads to alter index properties (Wiemer et al., 2015).

The Casagrande chart is suitable for classifying kaolin (K) or diatomaceous (D) type soils, when they have a total concentration, that is, 100% for any of them. However, for mixtures of these two inputs (e.g. 20% D and 80% K), the classification below the “A line”, as silt, turns out to be inappropriate. There is disagreement with some values of high plasticity indices reported for artificial mixtures with diatom content greater than 60%, since the greater presence of frustules, although it leads to a greater water storage capacity, does not necessarily imply an increase in plasticity (Xu et al., 2022).

The water contained in the intraskeletal pores has little incidence on the index properties, that is, any relationship based on the proportion of water or on the void content (for soils that have the presence of microfossils) must be redefined to avoid calculation and classification errors (H. Tanaka & Local, 1999).

The determination of LL and PL in DS is complex using traditional Casagrande methods, given the non-plasticity of this type of deposit. The way to calculate this parameter has been done indirectly through the relationship between the cone drop method and the flow index (Xu et al., 2022) (Caicedo et al., 2019).

For different types of diatoms, the consistency limits do not strictly increase in parallel. Fractured diatom frustules are a cause of decreased Plasticity Index (PI). The diatom species can affect the limits, given the different shapes and microstructures. The PI in the Coscinodiscus species is higher than that associated with the Aulacoseira species (Xu et al., 2022) (Slebi et al., 2021).

With increasing diatom content, the shrinkage limit is increased and the volumetric shrinkage is decreased. This is due to the effect of water absorption in the interparticle pores, which reduces cracking caused by desiccation. Shrinkage tests indicate a strong water-holding capacity in diatom frustules (Xu et al., 2022).

Concerning the specific gravity of the solids (Gs) in artificial mixtures of Kaolin - DS, and depending on the concentration of fossils, it is concluded that the Gs decreases when the content of frustules increases; this is related to the high porosity of diatomaceous skeletons and to the volume they occupy, which is considerably more prominent compared to that of Kaolin (López, 2009). This trend occurs in DS of different origins, either for a monospecies or multispecies condition (Zuluaga, 2021).

This research evaluated the variation in the consistency, plasticity and particle size distribution of three morphologically distinguishable DS by gravimetrically adjusting the content of frustules within a kaolin-type clay matrix and modifying in a controlled manner the level of deterioration of the fossils, approximating the conditions of fracturing or dissolution inherent to its development environment. The state of deterioration was validated through observations in the Scanning Electron Microscope and by calculating the specific surface area.

The induced deterioration was verified using a quantitative "Conservation Index" criterion. Finally, the development of a multivariate analysis made it possible to understand, through graphic representations, the behaviour of the research variables and their relationship, as well as to determine the normality in the distribution and dispersivity of the data.

This study's novelty lies in the comparative analysis of different diatomaceous soils based on a controlled frustule content and level of deterioration. That novelty is supported quantitatively by the exposed "Conservation Index". Furthermore, the size variation is validated simultaneously with several criteria (hydrometry, specific surface area and microscopy). Generally, papers report the results of the Diatomaceous soils (a single species) in the condition granted by the extraction of the sample without assessing the effects of deterioration due to environmental agents.

This research evaluated only physical properties and did not cover chemical or mineralogical characteristics of the frustules, which could influence the behaviour of this type of soil. Although the main composition of these fossils is Silica, the presence of other elements and their interaction with the environment may affect the mechanical response due to dissolution or other phenomena.

2. MATERIALS AND METHODS

Diatomaceous soils DS from three different origins and species were analyzed _ Colombian (Boyacá), Peruvian (Ica), and Mexican (Jalisco), see Figure 1. A fine-grained soil Kaolin type (K) served as a mixing medium. The gravimetric proportions were 100%DS, 66%DS-33%K, 33%DS-66%K, and 100%K. The mixture of materials was made in a dry condition. The drying process in DS and K was at 60ºC until reaching constant weight. Three different controlled deterioration states were induced in each soil sample.

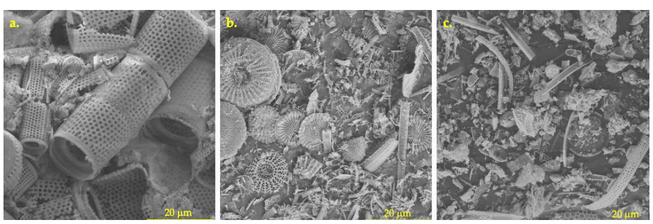

Figure 1. Different origin and morphology Diatomaceous Soils
a) Colombian origin, Aulacoseira granulata, centric, planktonic, avg. length: 13.58µm
b) Mexican origin, Cyclostephanos tholiformis, centric, planktonic, avg.diameter:11.44µm
c) Peruvian origin, thalassionema nitzschioides, araphid, benthonic, avg.width:2.43µm

2.1. Degradation process

A mechanical degradation process was developed tending to modify the size of the diatom frustules in a controlled manner. The production protocol considered a dried mass of Diatomaceous Soil of 5.000 gr. Later the sample was introduced into the "Los Angeles Machine" drum with an abrasive charge of twelve spheres. The Deterioration process (drum rotation quantity) resulted differently for each DS sample depending on the effective fragmentation state. For each DS origin, three different conservation states (high, medium, and low) were contemplated.

Two criteria validated the efficiency of the fragmentation process, the "Conservation Index CI" and "Particle Size distribution".

The CI quantitatively represents the sample deterioration state based on the particle size distribution by applying Scanning Electron Microscopy image processing (Image J software). As higher the CI, the more preserved the DS is. See figure 2. The CI calculations are supported in formula 1.

Although the conventional use of the "Los Angeles" machine is to evaluate abrasion in coarse aggregates, its application in this research was considered because it was the only method that allowed verifiable and significant changes in particle size to be induced in a controlled manner. Although the natural degradation mechanisms of a frustule are not by impact, but rather by dissolution or crushing, the final effect is a reduction in size and an increase in angularity of the fossil. The research group behind this article is currently studying the change in the size of frustules due to the time of exposure to waves, the dissolving agent and the diatom species.

2.2. Physical characteristics tests

For Consistency limits determination, the "Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils" (ASTM D4318-17e1) were applied in all DS samples, except the LL of Mexican soil, which was validated using the "Fall cone method" (GR-84-11). The Specific Gravity of the samples was determined by applying the ASTM D854 procedure. For Particle-Size Distribution Analysis of Fine-Grained Soils was used the Hydrometer Sedimentation method (ASTM D7928-21e1). The specific surface area was determined using the Blaine air-permeability apparatus and applying the procedure described in the ASTM C204-18e1 standard.

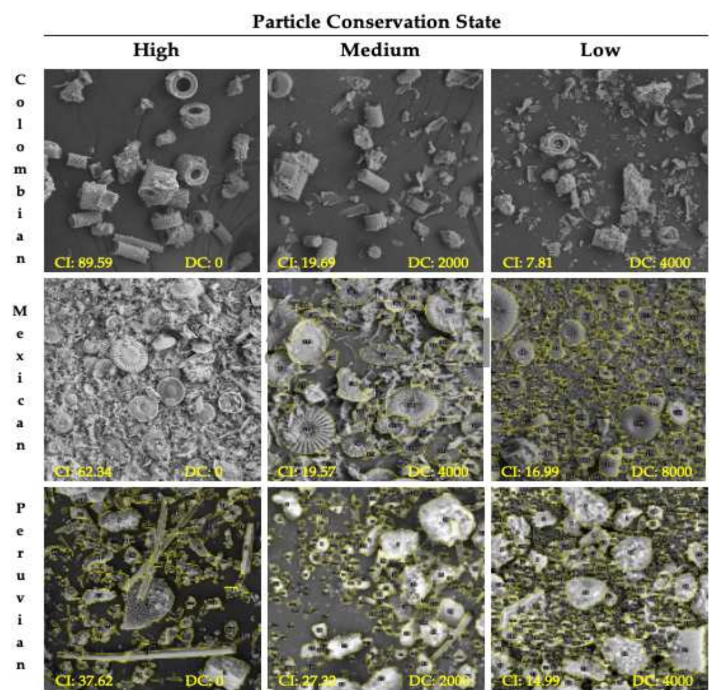

Figure 2. Evolution of the deterioration process of diatom frustules
CI: Conservation Index DC: Deterioration Cycles

$$CI = \sum \frac{Ai*FCi}{N} \quad (1)$$

CI: Conservation Index
A_i: Individual frustule area (μm^2)
N: Number of particles analyzed
FC_i: Individual Conservation factor
If intact frustule $FC_i = 1$

If affected frustule refer to formulas (2) and (3)

$$A_i \geq A_V IFS_{90}\ ; FC_i = CRF \tag{2}$$

$$A_i < AvIFS_{90}\ ;\ FC_i = CRF * \frac{A_i}{AvIFS_{90}} \tag{3}$$

A_vIFS: Average Intact Frustule Size approximately at 90% of Cumulative Relative Frequency COL 187.50 _ MEX 212.50 _ PER 87.50 (µm^2).

CRF: Cumulative Relative Frequency Factor (%) COL 88.79 _ MEX 90.58 _ PER 89.95.

3. RESULTS AND DISCUSSION

The results presented here concentrate on two aspects. The first is the effectiveness of the controlled degradation process. The second is the variation of the index properties depending on the simultaneous application of the study variables (concentration, species, and deterioration).

3.1. Controlled size variation

The particle size distribution for each specie is presented in Figures 3, 4, and 5. Each curve corresponds to the average behaviour of at least six hydrometrical tests. The curves' behaviour demonstrates the controlled crushing process's effectiveness, which reduces the particles' size as the number of cycles (DC) increases. The effect of the kaolin-type clay content on the size distribution can also be observed.

In the records of the Colombian (figure 3) and Peruvian (figure 5) species, the variation in particle size is observed (downward movement of the curves) depending on the higher content of frustules and the lower content of clayey particles (less than 2 um). When the DS contents are higher (DS 100%), the separation of the granulometric curves for different levels of deterioration is more evident. The curves present similar behaviors when the Kaolin content is representative (K 66%).

The size variation for Colombian species is evident when the analyses are based on the DS content rather than deterioration cycles (Figure 3). In the Peruvian species, particle size reductions can be seen in high concentrations (DS 66% and DS 100%), depending on the level of deterioration. This change reflects a greater homogeneity and susceptibility in the controlled crushing process (Figure 5).

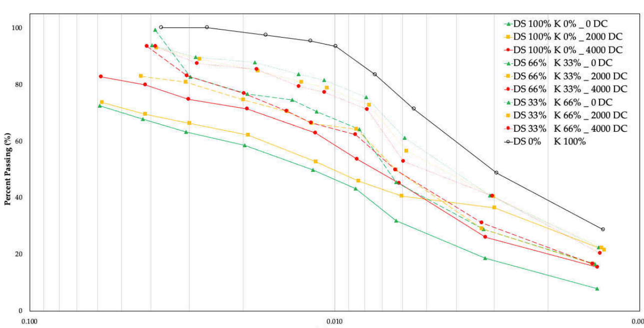

Figure 3. Colombian Diatomaceous Soil _ Particle Size Distribution variation as function of deterioration process.
DS: Diatomaceous Soil K: Kaolin DC: Deterioration Cycles.

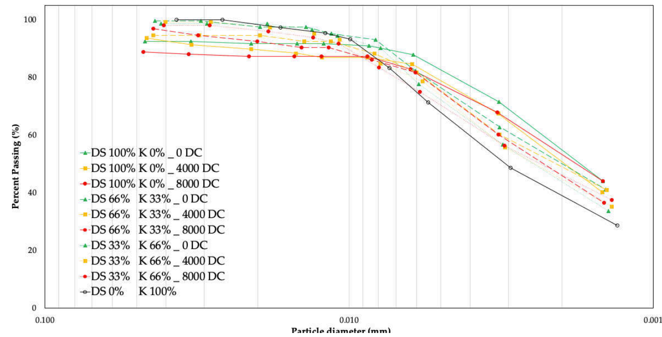

Figure 4. Mexican Diatomaceous Soil _ Particle Size Distribution variation as function of deterioration process.
DS: Diatomaceous Soil K: Kaolin DC: Deterioration Cycles.

This variation can be understood given the Peruvian particles' flat and elongated shape, which makes them more homogeneously exposed to the abrasive load. In contrast, with its greater volume and cylindrical shape, the Colombian frustule becomes more heterogeneous after breaking.

The Mexican species behaves differently, similar to the Kaolin curve. In this, the curves' organization is inverted (depending on DS content) when the size is close to 8 +/- 1 um. The variation in the size distribution is not clear when the deterioration condition is reviewed.

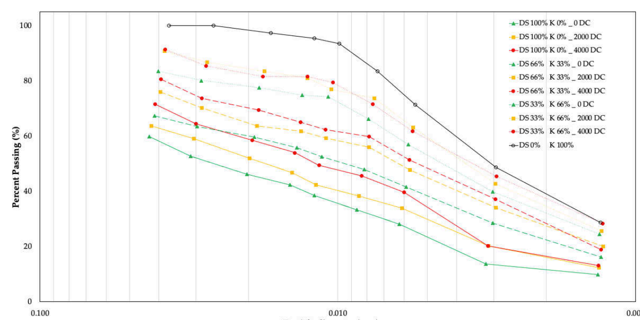

Figure 5. Peruvian Diatomaceous Soil _ Particle Size Distribution variation as function of deterioration process.
DS: Diatomaceous Soil K: Kaolin DC: Deterioration Cycles

3.2. Specific Surface

In addition to the particle size distribution, the effect of controlled crushing was validated by verifying the specific surface area. See Figure 6. In the samples from the three origins, an increase in cm^2/g was identified with more deterioration cycles. The Mexican species presents minor variability between the different levels of deterioration, while the Peruvian and Colombian species show a more marked change between 0, 2000 and 4000 cycles. This behaviour is consistent with the Conservation Index records presented in Figure 2.

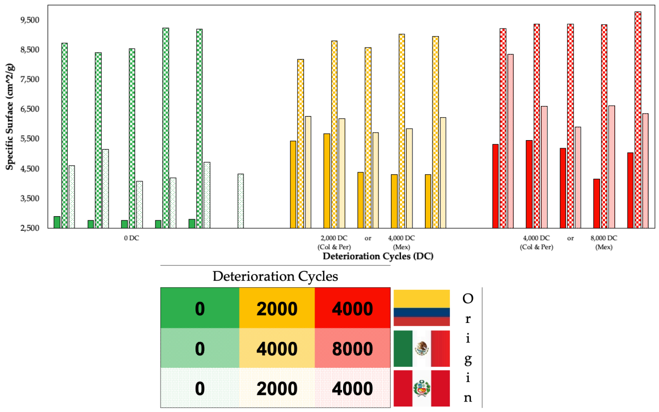

Figure 6. Specific Gravity variation as a function of species and deterioration process.

3.3. Specific gravity

The comparative analysis (Table 1) uses kaolin's Gs (2.65) as a contrasting element. For the Colombian and Mexican DS, a reduction of the parameter with a higher content of frustules is identified. The opposite situation is recognized with the Peruvian species, which increases the value of Gs with the highest content of diatomaceous soil.

Table 1. Diatomaceous Soils Specific Gravity variation as a function of concentration and deterioration process.

		Colombian Diatomaceous Soil Content			
		0% (*)	33.33%	66.66%	100%
DC	0	2.65	2.56	2.49	2.43
	2000		2.61	2.48	2.41
	4000		2.58	2.47	2.29

		Mexican Diatomaceous Soil Content			
		0% (*)	33.33%	66.66%	100%
DC	0	2.65	2.42	2.26	2.09
	4000		2.49	2.29	2.11
	8000		2.45	2.29	2.08

		Peruvian Diatomaceous Soil Content			
		0% (*)	33.33%	66.66%	100%
DC	0	2.65	2.69	2.79	2.77
	2000		2.72	2.82	2.78
	4000		2.70	2.78	2.77

DC: Deterioration Cycles (*):Kaolin 100%

3.4. Consistency limits

The Liquid Limit (LL), Plastic Limit (PL) and Plasticity Index (PI) values increment with a higher DS content. See Table 2. The highest reports of LL and PL are observed in the Mexican species, mainly associated with a flat disc shape. Contrary to the results, the initial expectation was that the Colombian species, which has a cylindrical shape, would have a greater water storage capacity. The LL and PL are similar in the Colombian and Peruvian species. As expected, the elongated Peruvian particles with less pore density reported less storage potential.

At this point, it is worth remembering the difference in the behaviour of the Mexican species in terms of particle size distribution. Likewise, it is denoted that the Colombian and Peruvian species present a more yellowish coloration, while the Mexican species stands out for its whiteness. In the same sense, it is worth clarifying that it was precisely the Mexican species that required greater exposure to deterioration cycles (0, 4000 and 8000) to reach a differentiable condition in terms of CI, while the Colombian and Peruvian species presented representative variations with fewer cycles (0, 2000 and 4000).

This aspect invites verifying the chemical composition and specific surface of the DS at the different levels of deterioration since the particle's shape or size only partially defines the soil's consistency.

Regarding the influence of the level of deterioration, it is identified that in the Colombian and Peruvian DS, there is a decrease in the LL and PL as the deterioration cycles increase. There is no clear pattern in Mexican soil for any deterioration condition.

The PI in Peruvian and Mexican DS tends to decrease as the level of deterioration increases. The Colombian sample reports the highest PI values.

Table 2. Diatomaceous Soils consistency limits variation as a function of concentration and deterioration process.

	COLOMBIAN DIATOMACEOUS SOIL					MEXICAN DIATOMACEOUS SOIL					PERUVIAN DIATOMACEOUS SOIL				
		DS Content					DS Content					DS Content			
	DC	0% (*)	33.33%	66.66%	100%	DC	0% (*)	33.33%	66.66%	100%	DC	0% (*)	33.33%	66.66%	100%
LIQUID LIMIT	0	42.07	64.10	92.10	134.97	0	42.07	102.30	140.30	160.30	0	42.07	52.61	62.50	71.78
	2000		60.80	83.40	120.07	4000		106.20	138.20	163.13	2000		50.67	57.89	65.75
	4000		59.30	78.20	106.13	8000		103.00	146.40	159.67	4000		48.08	57.00	63.46
PLASTIC LIMIT	0	30.52	36.80	53.10	75.21	0	30.52	64.30	108.10	114.00	0	30.52	30.22	32.15	33.03
	2000		36.00	51.00	55.44	4000		77.70	99.00	128.00	2000		29.17	29.16	29.31
	4000		39.10	47.50	48.63	8000		78.90	119.10	123.83	4000		27.48	28.31	28.87
PLASTICITY INDEX	0	19.4	27.30	39.00	59.76	0	19.4	38.00	42.20	46.30	0	19.4	22.39	30.35	38.75
	2000		24.80	32.40	64.63	4000		28.50	29.20	35.13	2000		21.50	28.73	36.43
	4000		20.20	30.70	57.50	8000		24.10	27.30	35.83	4000		20.60	28.69	34.59

DC: Deterioration cycles (*):Kaolin 100%

Different documentary sources (Locat & Tanaka, 2001) (Xu et al., 2022) (M. Tanaka et al., 2012) from 2011, 2012 and 2022 indicate that the liquid and plastic limits increase as the microfossil content increases. However, such increases in LL and PL occur at a similar rate, so the Plasticity Index remains more or less constant. The references explain that this phenomenon occurs due to the water absorption capacity within the pores of the frustules, but this does not imply changes in the soil's plasticity. Despite the above, the present research showed that the LL and PI slopes are not so similar that DS can report changes in the Plasticity Index as the frustules' contents increase.

Figure 7 shows the increasing trends in LL and PL for the multi-origin DS under investigation. When there is no relationship with any level of deterioration, the growth trends are similar between the LL and the PL; that is, the "0 DC" condition. The most significant differentiation is observed in the Colombian and Peruvian samples when “2000 and 4000 DC” are applied.

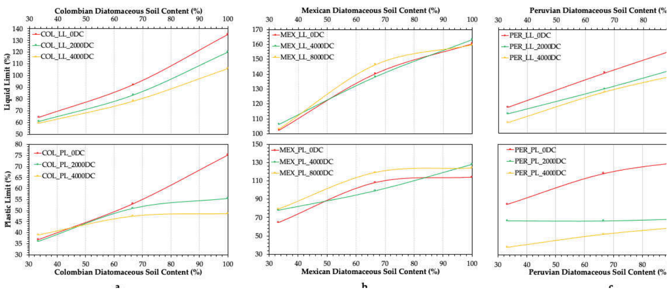

Figure 7. LL and PL rate increment in different origin DS as a function of concentration and deterioration process.
a) Colombian DS b) Mexican DS c) Peruvian DS. For all figures, above: LL, below: PL. DC: Deterioration cycles.

Figure 8 represents the behavior of the DS on the Plasticity Chart. Very well-defined trends were identified for each of the soils.

In the Peruvian case, a displacement almost comparable to "A line" is evidenced. No clear difference is observed as function of levels of deterioration. The Peruvian curve prolongation (LL Vs PI) is the smallest.

These projections are broader for Colombian and Mexican soils (greater LL and PI). As the representation of the results becomes horizontal (Mexican soil), a more pronounced separation of the curves based on the level of deterioration is recognized.

The representation of the Peruvian soil with a concentration of 100% frustules for any level of deterioration (DC 0, 2000, 4000) classifies it as (CH - OH) even though its fraction below 2 µm is only 13%.

The Colombian and Mexican samples, with kaolin contents of 66%, are categorized as MH-OH in the Plasticity Chart, contrary to the classic classification criteria.

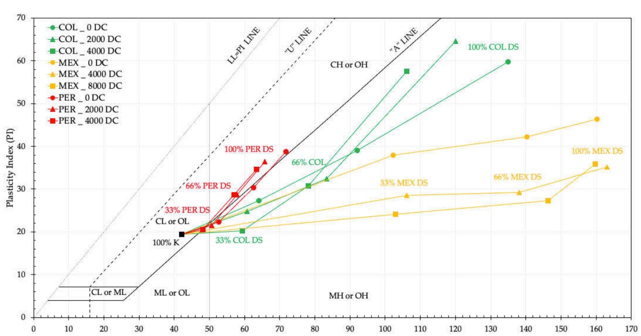

Figure 8. Diatomaceous Soils and Plasticity Chart
DC: Deterioration cycles

3.5. *Soil Activity*

For soils dosed with Colombian and Peruvian material, a higher clayey material (<2um) is evident when there is a lower proportion of DS; that is, when the concentration of Kaolin is higher. The opposite is observed in the Mexican sample, in which the material (<2um) increases with the more significant presence of fossils (Table 3).

For all soils, lower activity values are associated with higher levels of affectation (DC deterioration cycles).

In Colombian and Peruvian soils, the activity variation is more evident depending on the fossil content, reporting its maximum records with concentrations of 100%. In the Mexican case, the maximum magnitudes are obtained when deterioration is not induced (0 DC).

Soil activity (PI / Clay Content) is modified by the presence of microfossils. As previous reference, the clayey soils of Ariake (Japan) have high diatom content and report activity values between 1.0 and 2.0. Other low-fossil content clays, e.g., Singapore and Bangkok, report activity ranges between 0.5-0.8 and 0.9-1.4 respectively (H. Tanaka et al., 2001b).

Table 3. Diatomaceous Soils Activity

	COLOMBIAN DIATOMACEOUS SOIL					MEXICAN DIATOMACEOUS SOIL					PERUVIAN DIATOMACEOUS SOIL				
		DS Content					DS Content					DS Content			
	DC	0% (*)	33.33%	66.66%	100%	DC	0% (*)	33.33%	66.66%	100%	DC	0% (*)	33.33%	66.66%	100%
PLASTICITY INDEX	0	19.4	27.3	39	59.76	0	19.40	38.00	42.20	46.30	0	19.40	22.39	30.35	38.75
	2000		24.80	32.40	64.63	4000		28.50	29.20	35.13	2000		21.50	28.73	36.43
	4000		20.20	30.70	57.50	8000		24.10	27.30	35.83	4000		20.60	28.69	34.59
CLAY FRACTION	0	39.2	31.20	21.60	12.8	0	39.20	43.60	50.20	54.80	0	39.20	32.20	22.60	11.60
	2000		31.20	21.60	28.80	4000		44.80	48.80	50.80	2000		34.60	27.40	15.80
	4000		30.40	22.40	20.40	8000		46.20	46.00	53.20	4000		37.40	28.20	16.60
SOIL ACTIVITY	0	0.49	0.88	1.81	4.67	0	0.49	0.87	0.84	0.84	0	0.49	0.70	1.34	3.34
	2000		0.79	1.50	2.24	4000		0.64	0.60	0.69	2000		0.62	1.05	2.31
	4000		0.66	1.37	2.82	8000		0.52	0.59	0.67	4000		0.55	1.02	2.08

3.6. *Multivariate analysis*

Figure 9 illustrates the interaction between the number of deterioration cycles and the diatomite content for each species studied. Following these findings, a multivariate statistical analysis was conducted. The "response variables" considered were specific gravity (Gs), liquid limit (LL), plastic limit (PL), and clay fraction content (CF). "Response variables" such as plasticity index (PI) and activity were excluded to avoid direct codependence with the other variables.

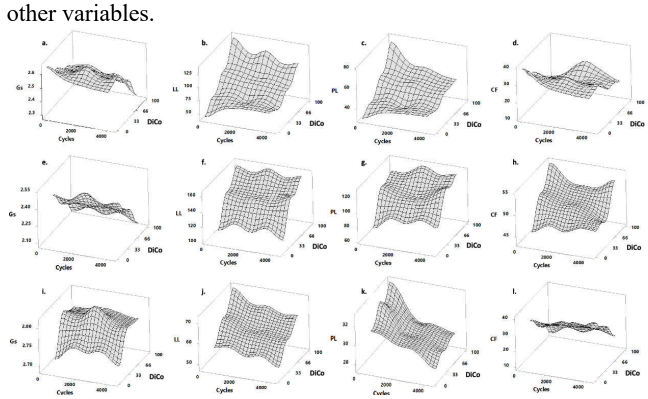

Figure 9. Surface Plot _ Deterioration Cycles and Diatomite Content (DiCo) Vs Each Response.

a,b,c,d: Colombian Species. e,f,g,h: Mexican Species. i,j,k,l: Peruvian Species.

Twenty-eight experimental designs, including the reference sample, were employed to conduct the multivariate analysis. The Anderson-Darling normality test assessed whether the data distribution conformed to normality. Figure 10 demonstrates that variables such as Gs and CF were normally distributed, whereas the LL and PL exhibited non-normal distributions. Consequently, the Pearson correlation coefficient was computed for variables following a normal distribution, while the Spearman correlation coefficient was utilized for those that did not. The identified trends and corresponding values are depicted in Figure 11.

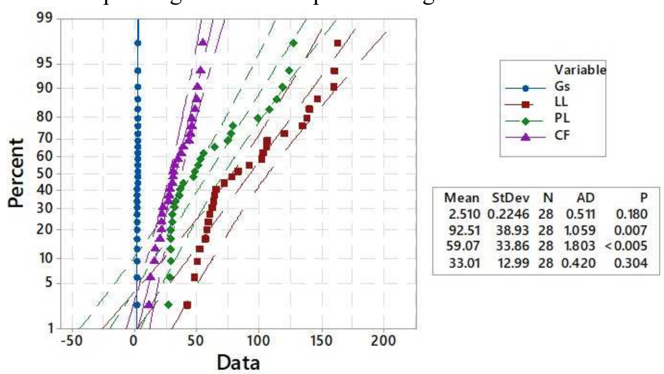

Figure 10. Anderson-Darling normality test conducted on each response variable

Figure 11 exposes a substantial correlation between the variables, all statistically significant at a 95% confidence level with corresponding p-values. The graph demonstrates strong negative correlations between Gs and LL-PL, as steep downward trends indicate. This behaviour suggests that the LL and PL values increase as the specific gravity decreases. Conversely, a strong positive correlation between the LL and PL is highlighted by the upward trend in the graph, implying that they increase together, a typical characteristic of cohesive soils. The CF has a moderate to strong positive correlation with LL and PL, suggesting that an increase in the clay content tends to be associated with higher liquid and plastic limits. The p-values close to zero across all correlations confirm the significance of these relationships, reinforcing the reliability of these insights into soil behaviour for the origins marked as Colombia (COL), Mexico (MEX), Peru (PER), and the reference sample (REF).

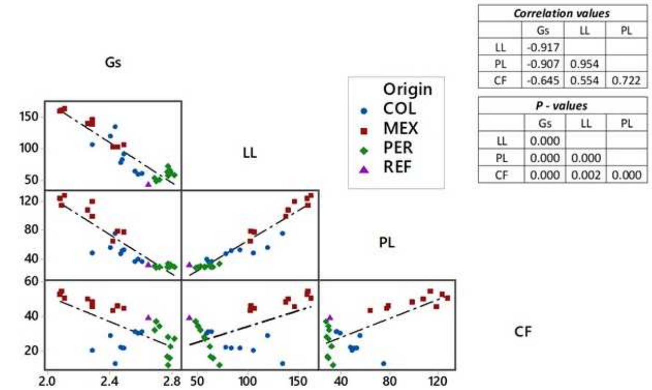

Figure 11. Correlation plot between response variables

A "principal component analysis" (PCA) was conducted to identify the most significant factors and reduce dimensionality in the dataset. The results are displayed in Figure 12.

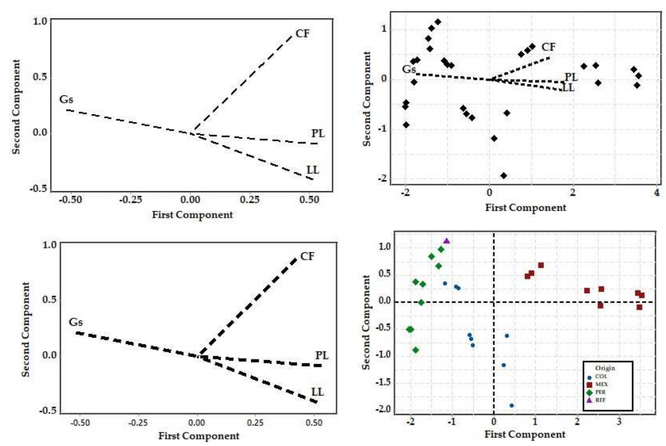

Figure 12. Summary results obtained from PCA

The eigenvalues indicate the magnitude of the variance captured by each principal component. The first "principal component" (PC1) has an eigenvalue of 3.3719, accounting for a substantial 84.3% of the variance within the data, suggesting that this component alone captures most of the variability observed. The cumulative proportion of variance accounted for by the first two components (PC1 and PC2) is 97%, with subsequent components contributing minimally to explaining the data's variance. The variable loadings on each principal component reveal the relationship between the variables and the components (see Figure 12). All PC1 variables have significant loadings, with Gs being inversely related to LL, PL, and CF. This inverse relationship may be attributed to higher plasticity limits typically associated with increased porosity, leading to a lower specific gravity. Conversely, through their fossilization processes, certain species of diatoms tend to form slightly heavier minerals, which increases their specific gravity compared to clay fractions, whose minerals are generally less dense. PC2 has the highest loading for CF, indicating that this component may represent the clay content's influence on soil properties.

The provided PCA biplot (see Figure 13) visualizes the distribution of samples based on the first two principal components derived from the multivariate analysis. The scatter of points across the plot corresponds to different experimental designs characterized by their Diatomaceous Content (DiCo) and the number of cycles they have undergone.

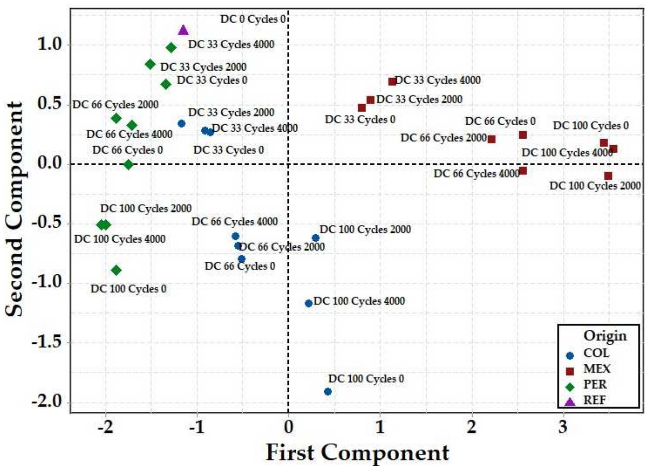

Figure 13. PCA biplot.

In the PCA biplot, we observe that species with a Mexican origin (MEX) are predominantly positioned towards the positive side of the first principal component, indicating a distinctive pattern or set of characteristics that separate them from species of other origins. This report could imply that Mexican species have unique attributes or have responded differently from other DS (COL, PER) and the reference sample (REF) to the degradation cycles application.

Species from Colombia are more dispersed but with a slight concentration towards the negative side of both principal components, suggesting a variance in response, yet generally different from the Mexican species. Peruvian species appear slightly more clustered and are spread across the center of the plot, indicating a moderate response to the degradation cycles without a strong bias towards any specific principal component.

A combined approach using "Principal Component Analysis" (PCA) and cluster analysis provides a multifaceted view of the data structure in understanding the relationships between different response variables. The PCA serves as a precursor to clustering by simplifying the complexity of multidimensional data, thereby allowing for identifying patterns that might not be readily apparent.

The dendrogram (figure 14) generated from the cluster analysis illustrates a notable variable division. At the outset, Gs is significantly divergent from the rest, implying a unique response characteristic distinct from the other variables. Moving further up the similarity scale, LL and PL are observed to form a cluster, indicating a moderate level of correspondence between them. Such a relationship suggests that these variables could capture similar aspects or are mutually influenced by the same conditions.

Lastly, CF joins the cluster with LL and PL at a higher level of similarity but is less closely related than LL and PL are to each other. This positioning in the dendrogram hints at a shared connection between CF and the other two variables, yet it also maintains a certain level of individuality.

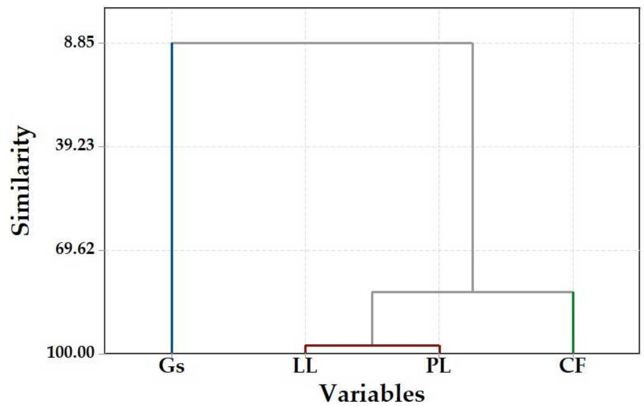

Figure 14. Cluster analysis of response variables

4. CONCLUSIONS

The effectiveness of the controlled deterioration process could be validated with the particle size variation. Applying the study variables allowed the recognition patterns in the granulometric distribution curves, whose characteristic shapes were differentiable between the species. The changes are more evident due to the effect of the concentration of fossils and not so much due to the impact of induced deterioration. The behaviour of the Colombian and Peruvian species is similar to each other and very different from the behaviour of the Mexican species.

The variation in particle sizes can be understood by the characteristic shape of each species and the susceptibility to breaking. The Peruvian particles have a flat and elongated shape, which makes them more exposed to breakage. The Colombian frustule becomes more heterogeneous after breaking, considering its greater volume and cylindrical shape. The dish shape of the Mexican species implies a disposition for horizontal accommodation and a greater surface area to distribute the impact forces, which restricts breakage.

In the three origins samples, an increase in cm^2g^{-1} was identified with cumulative deterioration cycles. This behaviour is consistent with the Conservation Index records. The Gs present clear patterns of change depending on the concentration and not so much due to the level of deterioration. Although the composition of the frustules is similar between the three species, the Gs values are different. For all soils, lower activity values are associated with higher levels of affectation (DC deterioration cycles).

The LL, PL and PI values increase with a higher DS content. The highest reports of LL and PL are observed in the Mexican species (flat disc shape). The initial expectation was that the Colombian species, which has a cylindrical shape, would have a greater water storage capacity. As expected, the elongated Peruvian particles with less pore density reported less storage potential.

From the results obtained, it was possible to validate that implementing the classification system based on the "plasticity chart" is inappropriate for DS. Combinations of soils with high clay content, close to 66% by weight, are classified as silt. DS concentrations of 100% are categorized as clays, even when the granulometric distribution shows that they are silts.

The Mexican species presents marked differences with the Colombian and Peruvian species in aspects such as the soil colouration (COL and PER are yellowish, MEX is white), the shape of the granulometric curves, and LL and PL as a function of deterioration cycles. This aspect invites verifying the chemical composition and specific surface of the DS at the different levels of deterioration since the particle's shape or size only partially defines the soil's consistency.

The principal component analysis (PCA), accounting for 97% of the total variance within the first two components, has effectively depicted the complexity of the multivariate data, highlighting significant relationships among the examined soil properties. The spatial distribution in the PCA biplot revealed that the Mexican species exhibited distinct characteristics compared to the Colombian and Peruvian species. Cluster analysis, complemented by PCA, enhanced the understanding of the dataset by revealing its underlying structures. The resulting dendrogram clearly illustrated a divergence of specific gravity (Gs) from other variables. Similarly, a close relationship between liquid limit (LL) and plastic limit (PL) was observed, with clay fraction (CF) sharing a connection to both while retaining a distinct influence.

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