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DID WE UNDERESTIMATE SILT AND CLAY CONTENT IN THE TEXTURAL ANALYSIS?

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ABSTRACT

Particle size distribution (PSD) is a soil physical property influenced by organo-mineral complexes (or-mic) in silt and clay fractions. In Andosols, these fractions are underestimated due to the presence of or-mic in soil micro-aggregates. This study estimated quantitatively the organo-mineral fraction by comparing two methodologies of granulometric analysis (sequential dispersion and sieve-sedimentation) in Andosols and Fluvisols from southern Chile. Or-mic were calculated through specific dissolution extractions (Al, Si, Fe). In addition, pH_{NaF}, and soil organic carbon (SOC) were determined. Correlations between Al_o (0.74), Al_p (0.71), pH in NaF (0.41), and the or-mic were found. Or-mic were higher in Andosols, varying between 1.5 and 5.2%, which led to an underestimation of silt and clay fractions of the soil in PSD analysis and, thus, an overestimation of soil texture due to the continuous formation of micro-aggregates. Therefore, to properly determine soil PSD, it is essential to estimate or-mic, particularly in highly reactive soils, which are readily capable of binding organic compounds in their mineral fraction.

Key words: or-mic, granulometry, soil reactivity.

INTRODUCTION

Soil texture is the relative proportion of sand, silt and clay (mineral particles < 2 mm) and is determined after the physical disruption and chemical dispersion of the aggregates (USDA, 2004; Sandoval et al., 2012). Thus, particle size distribution (PSD) of soil is estimated based on the proportion and size of these mineral particles, often by sieving and sedimentation analyses (Carter and Gregorich, 2006).

PSD is a physical property that is often used to classify soils (Skaggs et al., 2001), and to estimate soil hydraulic properties through pedotransfer functions (Rabot et al., 2018), being also a relevant parameter for the efficient and sustainable management of soils (Kettler et al., 2001; Di Stefano et al., 2010). Consequently, PSD provides

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insight into soil behavior (Qi et al., 2018). PSD of a soil changes over time with soil formation processes, clay fraction mineralogy and soil organic carbon (SOC) (Kleber et al., 2015; Zúñiga et al., 2019). The chemical structure and type of organic ligands present in the soil determine the strength of the binding mechanisms of soil particles (Wagai et al., 2020), and thus the degree of stabilization of organo-mineral associations (Kögel-Knabner et al., 2008). The latter can be a problem when it comes to the determination of PSD of a highly reactive soil with a high SOC content. In this case, additional procedures must be implemented to reach soil sample dispersion since micro-aggregates are resistant to dispersing agents due to the binds of or-mic (mainly Al and Fe) distributed in the soil (Wagai et al., 2020). Moreover, under conditions of incomplete soil dispersion, the drag resistance of particles increases due to their irregular shapes, reducing the sedimentation rate and underestimating the soil clay fraction (Di Stefano et al., 2010). On the contrary, a prolonged dispersion to remove the cementing agents and separate the fractions can lead to erroneous PSD results due to the dissolution of the primary minerals (Velescu et al., 2010). Therefore, soil textural class is sometimes used instead of PSD (e.g. Haller et al., 2015 in Placaquands), which can be critical when pedotransfer functions based on the PSD are used.

The clay fraction (< 2 μm) is composed of layered aluminosilicates, metal oxides, hydroxides, and non-crystalline aluminosilicates (e.g., allophane and imogolite). This fraction influences soil aggregation, biological activity, and physico-chemical processes (Yudina et al., 2018; Wiesmeier et al., 2019). A high clay content increases the stability of aggregates (Six et al., 2002) because clay has a high specific surface area, which is capable of stabilizing organic compounds in the soil, influencing SOC sequestration (Wiesmeier et al., 2019; Clunes and Pinochet, 2021). A highly reactive mineral fraction generates strong or-mic (Kleber et al., 2015), which can alter the determination of clay and silt fractions and, consequently, of the degree of C stabilization (Plante et al., 2006).

Although PSD analyses consider soil physico-chemical dispersion through laser diffraction, and ultrasonic or sieve-sedimentation methods (Dumbrovský et al., 2019), it is important to consider that, regardless of the dispersion method used, organo-mineral fractions remain stabilized. The objectives of this study were to: i) evaluate whether the degree of dispersion alters PSD in soils with contrasting reactivity, using two methodologies: the sequential method (chemical dispersion) proposed by Velescu et al. (2010), and the classic method (sieve-sedimentation), and ii) estimate an organo-mineral fraction within PSD through reactivity and soil condition indicators. We use 'organo-mineral complexes' (or-mic) to refer to the organo-clay and organo-mineral complexes distributed in the soil, assuming that these associations are due to adsorption, complexation and aggregation mechanisms between mineral particles and SOC.

MATERIALS AND METHODS

Description of the soils and sites

Three soil series were selected. The series correspond to different soil formation processes that took place under temperate (Cfb) and arid (BSk) conditions, according to the climate classification of Köppen-Geiger (Kottek et al., 2006). The disturbed material was sampled at surface (0-30 cm) and sub-surface (30-50 cm) depths. *The Valdivia Soil Series* (VAL) corresponds to a soil derived from volcanic ash, classified as a Petroduric Silandic Andosol (Salazar et al., 2005; WRB, 2014) or a Duric Hapludand (Luzio and Casanova, 2006). This soil was collected from the Estación Experimental Agropecuaria Austral (EEAA) (39° 47′ S, 73°13′ W) of the Universidad Austral de Chile. This soil has evolved at a mean temperature of 12 ºC and a rainfall of 2500 mm per year (González-Reyes and Muñoz, 2013). *Alerce Soil Series* (ALE) is located 17 km northwest of Puerto Montt city (41° 25′ S, 73° 8′ W). This soil developed from volcanic ash deposited over glacial till material under wet conditions. A thin iron cemented layer or placic horizon (Bsm) was formed in the contact zone between the cemented till material and the volcanic soil (Carrasco et al., 2017; Zúñiga et al., 2019), limiting drainage through the soil profile. This soil is classified as a Aluandic Andosol or Duric Histic Placaquand (CIREN, 2003; Salazar et al., 2005). In terms of climatic conditions, average annual temperature in this area is 10.7 °C, while rainfall ranges between 1783 and 2021 mm per year (Dörner et al., 2016). *Miramar Soil Series* (TDF) is located in the North of Tierra del Fuego Island (52° 39′ S, 69° 15′ W). This soil is characterized by a histic horizon developed on fluvial or lacustrine sediments. Soil textures vary from fine sand at the surface to clay at greater depths, restricting internal drainage, and locally known as 'vegas' (Filipová et al., 2010). In this area, the average annual temperature is 10 °C and the rainfall ranges between 350 and 450 mm per year (Díaz et al., 1960; Valle et al., 2015a). Based on their dominant clay, samples with a high reactivity (VAL and ALE) were compared with those with low reactivity but a high presence of carbonates (TDF) due to formation processes.

Soil chemical analysis

The reactivity indicators of the colloidal materials were determined in each soil using specific dissolution techniques (Parfitt and Wilson, 1985). Aluminum, silica and iron were extracted with Na dithionite-citrate-bicarbonate (Al_d-Si_d-Fe_d), 0.2 M acid ammonium oxalate (Al_o-Si_o-Fe_o), and 0.1 M sodium pyrophosphate (Al_p-Si_p-Fe_p). The pH_{NaF} was determined according to Fieldes and Perrott (1966), while the content of SOC was determined using the Walkley-Black method (Sadzawka et al., 2006).

Particle size distribution analysis

Soil samples (3 replicates x 3 soils x 2 depths) were dried at 30 °C, sieved at 2 mm and treated with H_2O_2 to remove organic matter. The PSD analysis was conducted using two methodologies: the sequential method proposed by Velescu et al. (2010) and the classic method (sieve-sedimentation).

The method proposed by Velescu et al. (2010) was conducted as follows: 3 subsamples of soils (A, B and C) were prepared by weighing 10 g of dry soil, which were then treated with H_2O_2. Sample A was dried in a forced air oven at 105 °C to determine soil moisture content. Two hundred mL of 10.38% potassium oxalate solution were added to samples B and C, which were then heated in a water bath to 80 °C. Afterwards, 50 mL of a 9.5% oxalic acid solution was added at 90 °C and stirred with a rod for 5 minutes. Subsequently, the stirring rod was washed with 25 mL of oxalic acid, maintaining constant temperature for 3 minutes. Afterwards, samples B and C were centrifuged at 4000 rpm for 15 minutes, and then the supernatant was extracted. Subsequently, samples B and C were treated with 400 mL of a 0.2 M buffered oxalic acid NH_4 solution with a pH of 3.2, stirred in the dark for 16 hours, and then centrifuged at 4000 rpm for 15 minutes. The supernatant was then removed and centrifuged again to remove residues of the dispersants used. 25 mL of Na 0.1 M pyrophosphate were added to facilitate dispersion among the cemented particles in sample B. Sample C was dried in a forced air oven at 105 °C and the mass of the amount of dissolved material was estimated using the weight difference method. For samples B and C, the proportion of residual particles was weighed during each dispersion process. Thus, the proportion of or-mic present in each soil and depth was determined (Fig. 1).

For the PSD analysis using the 'classic method' (sieve-sedimentation), 40 g of sieved dry soil were digested with H_2O_2 (100 volumes) to remove organic matter (Sadzawka et al., 2006). Hydrogen peroxide was applied daily at 20 °C (room temperature), and then the samples were placed in a water bath (at about 80 °C) until the material stopped reacting. To ensure the removal of the cementing agents and adequate dispersion of the fine particles, the samples were pre-treated with sodium dithionite ($Na_2S_2O_4$), 0.3 M Na citrate ($Na_3O_7C_6H_5$), and 1 M Na acetate (CH_3COONa). After, a volume of 100 mL of sodium pyrophosphate (0.1 N) was added and the mixture was stirred for 10 minutes according to the Bouyoucos method. Silt and clay fractions were determined using the hydrometer method (Day, 1965). Finally, sand fractions were physically separated with a set of sieves (Forshythe, 1974).

Organo-mineral fractions (Or-mic)

Organo-mineral fractions were calculated using the following equation:

$$Or - mic = 100 - (PSD)$$

Where Or-mic correspond to organo-mineral fractions (%) and PSD is the sum of sand, silt and clay contents (%).

Statistical analysis

A correlation analysis (Pearson's coefficient) was performed to evaluate the degree of association or linear dependence between specific dissolution extractions and the presence of the or-mic obtained after each dispersant treatment ($p < 0.05$). A principal component analysis (PCA) was carried out to identify which reactivity indicators could explain the variation of the or-mic among the evaluated soils. The analyses were performed using R software, version 4.0.2 (R Core Team 2019) and RStudio software, version 1.3.1073 (RStudio, Inc., MA, USA).

RESULTS

Correlation between specific dissolution extractions and or-mic

Significant correlations ($p < 0.05$) were found between the Al_o (0.74), Al_p (0.71), Al_d (0.69) and or-mic content (Fig. 2A). This fraction is highly associated with aluminum binding to non-crystalline hydroxides and amorphous material (Al_o) organic compounds (Al_p), and paracrystalline materials (Al_d).

The principal components accounted for 93.6% of the cumulative variance (Fig. 2B). Aluminum fractioning (eigenvalue -0.401), pH_{NaF} (eigenvalue -0.319) and the or-mic (eigenvalue -0.304) had a high weight in PC1, and separated Andosols from Fluvisols (Fig. 2B). PC2 was explained by

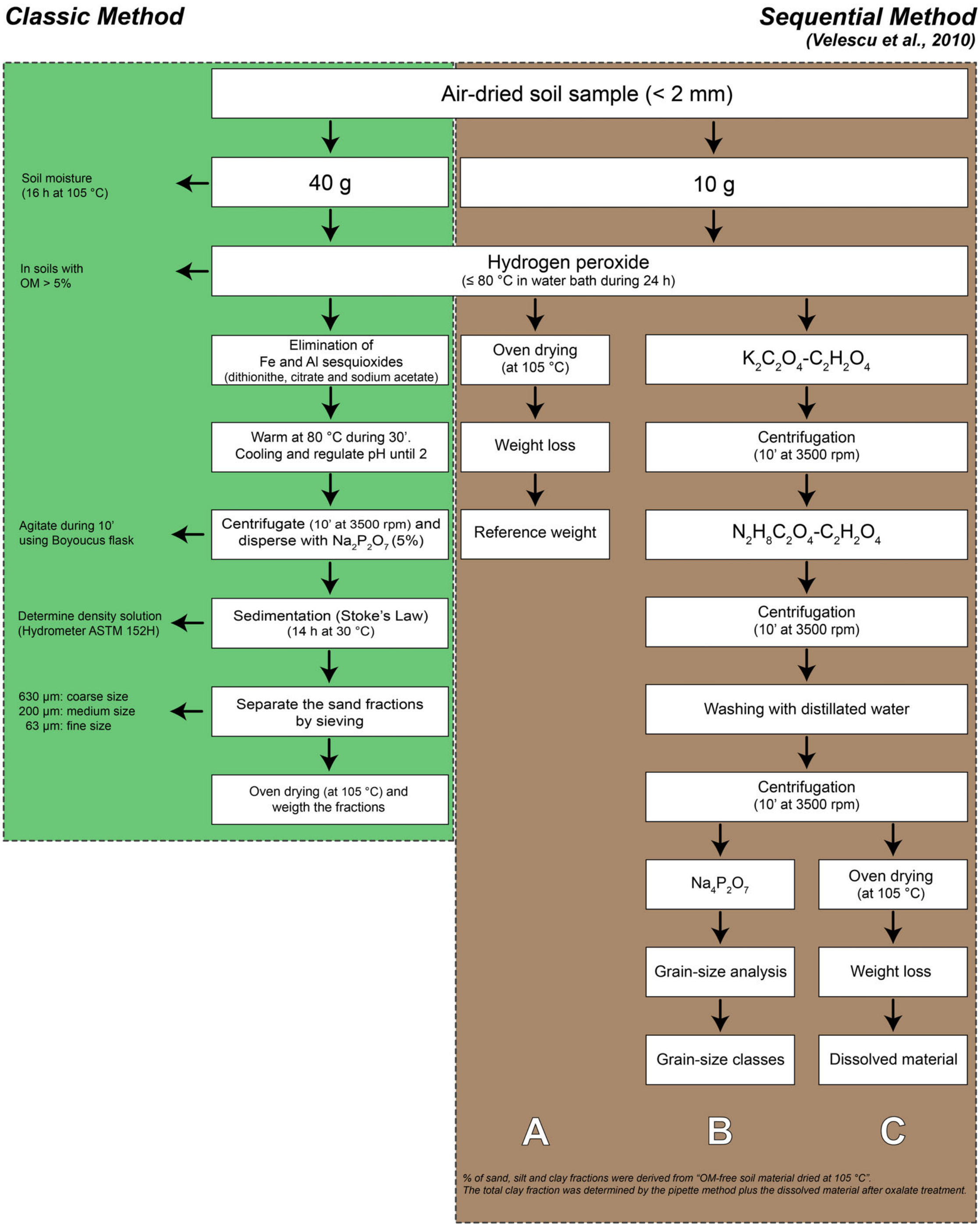


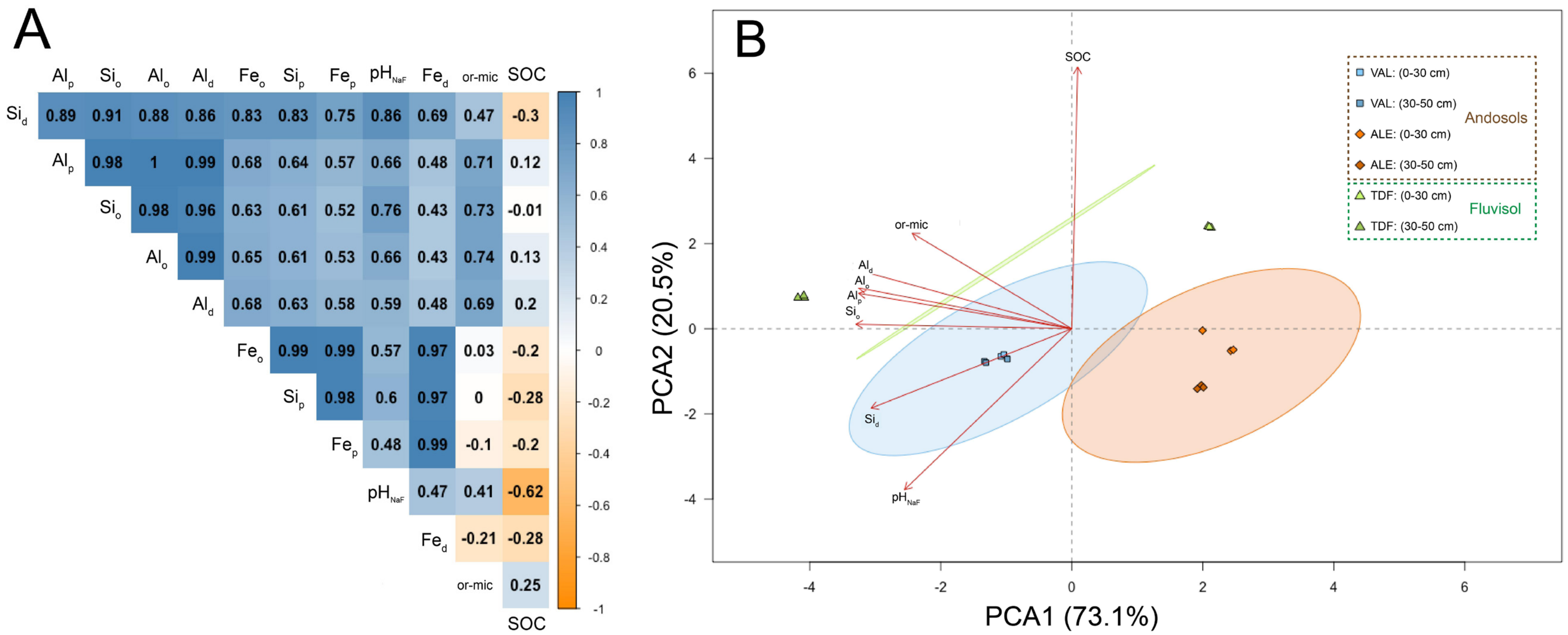
Fig. 1. Flowcharts for determining particle size distribution through the sieve-sedimentation or 'classic' method (green box), and the sequential method (brown box).

the SOC content, separating soils according to their reactive fraction (organic or mineral).

Changes in particle size distribution as a function of the methodology used

Soil particle size changes its distribution when it is subjected to a sequential method of dispersion (Velescu et al., 2010). In the present study, this resulted in the reordering of soil particles in each soil separate (Table 1).

The highest and lowest differences between the classic and sequential methods (ΔCM-SM) were found in the silt content in Aluandic Andosol (ALE), and in the surface sand content



	Al_p	Si_o	Al_o	Al_d	Fe_o	Si_p	Fe_p	pH_{NaF}	Fe_d	or-mic	SOC
Si_d	0.89	0.91	0.88	0.86	0.83	0.83	0.75	0.86	0.69	0.47	-0.3
Al_p		0.98	1	0.99	0.68	0.64	0.57	0.66	0.48	0.71	0.12
Si_o			0.98	0.96	0.63	0.61	0.52	0.76	0.43	0.73	-0.01
Al_o				0.99	0.65	0.61	0.53	0.66	0.43	0.74	0.13
Al_d					0.68	0.63	0.58	0.59	0.48	0.69	0.2
Fe_o						0.99	0.99	0.57	0.97	0.03	-0.2
Si_p							0.98	0.6	0.97	0	-0.28
Fe_p								0.48	0.99	-0.1	-0.2
pH_{NaF}									0.47	0.41	-0.62
Fe_d										-0.21	-0.28
or-mic											0.25

Fig. 2. Correlation Matrix (A) and Principal Component Analysis (B) among aluminum, silica and iron fractions, pH_{NaF}, SOC and or-mic for Andosols and Fluvisol at surface (0-30 cm) and sub-surface (30-50 cm) horizons.

Table 1. Particle size distribution determined at surface (0-30 cm) and sub-surface (30-50 cm) horizons for Andosols and Fluvisols using two methodologies: Sequential dispersion (A) and sieve-sedimentation (B).

Soil type	Depth[1] (cm)	Sand[2] CM*	Sand[2] SM	Silt CM	Silt SM	Clay CM	Clay SM	Or-mic SM	ΔSand	ΔSilt	ΔClay
		------ % ------							ΔCM-SM		
Aluandic Andosol (ALE)	1	6.5	11.3	58.5	64.9	35.0	22.2	1.7	-4.8	-6.4	12.8
	2	35.4	8.9	36.4	77.1	28.2	8.8	5.2	26.5	-40.7	19.4
Silandic Andosol (VAL)	1	10.2	11.4	62.9	50.9	26.8	36.6	1.0	-1.2	12.0	-9.8
	2	11.7	8.9	60.0	73.1	28.3	16.4	1.5	2.8	-13.1	11.9
Histic Fluvisol (TDF)	1	32.3	11.6	50.5	75.4	17.2	11.9	1.1	20.7	-24.9	5.3
	2	24.0	28.0	59.5	30.7	16.5	40.6	1.3	-4.0	28.8	-24.1

[1]: 1 = 0-30 cm; 2 = 30-50 cm.

[2]: Sand = 2000-63 μm; Silt = 63-2 μm; Clay = < 2 μm.

*: CM: Classic method; SM: Sequential method. All variables in %.

in the Silandic Andosol (VAL), respectively. The sequential method was able to disperse the micro-aggregates, increasing silt content in ALE (-6.4%; 40.7%), VAL (-13.1%) and TDF (-24.9%), while clay content increased in VAL (-9.8%) and TDF (-24.1%). The highest values of sand were found in ALE (26.5%) and TDF (20.7%) by the classic method.

The clay content obtained by sequential extractions was higher than that determined by the classic method in VAL (0-30 cm) and TDF (30-50 cm). On the contrary, it was lower when measured with the sequential method of dispersion in VAL (30-50 cm), ALE (0-30 cm), ALE (30-50 cm) and TDF (0-30 cm) (Table 1). This variation in the clay fraction as a result of the methodology used was also observed in the silt content, where VAL (0-30 cm) and TDF (30-50 cm) were lower and VAL (30-50 cm), ALE (0-30 cm), ALE (30-50 cm) and TDF (0-30 cm) were higher by the sequential method (Table 1). With respect to the sand fraction, this increased in VAL (0-30 cm), TDF (30-50 cm) and ALE (0-30 cm), but decreased in VAL (30-50 cm), TDF (0-30 cm) and ALE (30-50 cm) by the sequential method.

Organo-mineral fraction

As observed in the previous fractions, when using a methodology with a sequential dispersion period, PSD was rearranged within the soil separates and the remaining material in each dispersion represented an org-min fraction within the granulometric analysis of the soil. The highest or-min fraction was obtained in ALE (5.2%) (Table 1). In all the soils, or-mic content tended to increase with depth (ALE > VAL > TDF), suggesting that a translocation process could be involved in the distribution of or-mic in the soil profile.

DISCUSSION

Soil reactivity is generally evaluated through soil chemical indicators (Kleber et al., 2015), which are useful to determine the physicochemical associations between mineral and organic compounds (Dwivedi et al., 2019).

Although Al_d showed high correlation with or-mic, it was not possible to ensure that the Al came from non-crystalline aluminium silicates or gibbsite (García-Rodeja et al., 2004; Valle et al., 2015b; Porras et al., 2017). In fact, Al_o and Al_p have proved to be more reliable to estimate the reactive fraction in soils (Wagai et al., 2020). Specific dissolution Al extractions have been widely used to determine different reactive pools in soils derived from volcanic ash (Porras et al., 2017; Valle and Carrasco, 2018; Zúñiga et al., 2019), as well as the pH_{NaF} values that represent the reactivity of the OH^- edges present in the soil colloidal fraction capable of binding soluble organic compounds (Clunes and Pinochet, 2021). Moreover, these indicators associated with the mineral fraction have been widely used to determine the ability to protect SOC (Matus et al., 2014; Valle et al., 2015b; Panichini et al., 2017; Valle and Carrasco, 2018; Zúñiga et al., 2019; Clunes and Pinochet, 2021), and for differences between soils with crystalline or short order mineralogy, presenting pH_{NaF} values from 9.7 to 12.0 (Valle et al., 2015b).

In study, it was observed that the indicators fulfill the capacity of Andosols to bind organic compounds and form or-mic in both surface and subsurface horizons. The high variability in reactivity indicators observed in ALE (Fig. 2B) is due to the fact that Aquands have soil depth limitations (50-80 cm) because of the formation of a placic horizon (iron-cemented layer), which is originated by the chelation processes between SOC and metallic cations such as iron, manganese, and aluminium (Zúñiga et al., 2019; Bravo et al., 2021). The high levels of pH_{NaF} can be explained by the presence of carbonates in soils formed over calcareous materials, such as in TDF, where fluorine reacts by releasing OH^-, which is associated with calcium carbonate.

The percentage of clay determined through the PSD analysis was not a direct parameter of reactivity, but an indirect parameter of organic carbon storage, which agrees with Wiesmeier et al., (2019). Therefore, a prolonged dispersion allowed for the separation of SOC from the micro-aggregates and the colloidal fraction (López-Sangil and Rovira, 2013). Moreover, SOC is an appropriate indicator of organic C stabilized and stored in the soil because its association with fine soil particles is highly stable (Six et al., 2002; Wiesmeier et al., 2019; Wagai et al., 2020). Therefore, it is also a useful indicator of the or-mic fraction associated with silt and clay fractions. Soils of non-volcanic origin, such as TDF, do not show a direct relationship between their incipient content of organo-mineral complexes and soil reactivity parameters despite having a dominant 2:1 clay (montmorillonite), which is highly reactive and expandable (Diaz et al., 1960). The latter is due to the fact that the aluminum present in these soils is precipitated.

Particle size distribution and or-mic

The presence of organo-mineral associations in volcanic ash soils overestimates their PSD, particularly of the sand and silt fractions of the soil. This is due to the strong aggregation of volcanic ash soils as a consequence of organo-mineral complexes that permit the formation of micro-aggregates.

An incomplete dispersion underestimates clay content because particles lower than 2 mm can form micro-aggregates with organic materials or or-mic. This can inaccurately estimate part of the silt fraction, depending on the size of the aggregate, and alter the textural classification of soils (Plante et al., 2006). Andosols easily form micro-aggregates due to their colloidal fraction (Valle et al., 2015b), and have the capacity to bind organic compounds (Matus et al., 2014). In addition, they can expand the aggregation hierarchy by increasing the range among scales, depending on the order of particles and strength of the bonds (Asano and Wagai, 2014; Kleber et al., 2015; Bravo et al., 2021). This can alter results when soil particle size is determined through the principle of sedimentation according to Stokes' Law, since organo-mineral complexes present a similar behavior to the silt fraction during sedimentation (Velescu et al., 2010). The mass distributions of the PSD change after chemical and/or physical dispersions, i.e., the particles are relocated into coarse (>250 µm) and fine (53-250 µm) fractions, due to the dispersion of micro-aggregates formed by carbon compounds associated with the clay-silt fractions, which increase clay and silt contents (Plante et al. 2006). Therefore, the carbon content found in the compounds associated with clay and silt needs to be estimated in order to reliably quantify the C content that is potentially stored in the soil (Matus, 2021).

Furthermore, the soil profile distribution of or-mic could change depending on the soil horizon where they are accumulated, content of soil clay and SOC, and historical management under environmental changing conditions. Moreover, the results reveal the importance of proper particle dispersion not only for volcanic ash and highly reactive soils, but also question the accuracy of the method proposed by Day (1965) in soils originated from calcic sediments in Tierra del Fuego.

CONCLUSIONS

The determination of PSD varies depending on the methodology used. This variation highlights the importance of establishing a procedure to measure the fraction associated with organo-mineral complexes for soils that are highly reactive due to their mineralogical or organic composition. This fraction could be detected by sequential soil dispersion and measured directly on each residual fraction. The methodological issues identified in PSD analysis could be overcome by incorporating a fraction associated with organo-mineral complexes. Therefore, particle dispersion techniques need to be considered when evaluating soils capable of forming organo-mineral complexes as Andosols.

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