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Shaking settings to reduce the breakdown of Entisol fragile particles in texture analysis

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ABSTRACT: Finding the proper shaking setting to ensure total particle dispersion without altering the content of sand, silt and clay in soils composed of fragile particles such as saprolite fragments is still a research gap. This study aimed to identify the best shaking setting to optimize particle dispersion and minimize particle change in samples of Entisol (*Neossolos*). Samples were dispersed using an electric mixer at 12,000 rpm for 5, 15, 30 and 60 min, a horizontal shaker at 150 cycles min⁻¹ for 1, 2, 4, 8 and 16 h with and without the use of nylon spheres, and an ultrasonic shaker at 70 and 110 J mL⁻¹ for approximately 20 and 30 min, respectively. All the treatments affected particle distribution. Coarse sand reduced up to 14 % and clay content increased up to 18 % with the horizontal shaker with nylon spheres and the electric mixer. These changes were attributed to the breakdown of saprolite fragments, which represented around 1/3 of the coarse sand. Our results showed that the best setting was the horizontal shaker at 150 cycles min⁻¹ for 1 to 2 h without using nylon spheres. This option allowed optimizing particle dispersion and minimizing changes in particle size distribution.

Keywords: saprolite fragments, physical dispersion, marginal soils, *Neossolos*.

INTRODUCTION

Soil texture is not only a basic characteristic to distinguish and classify soils, but it is also a predictor of several other soil properties. Soil texture is used as a predictor of the soil erodibility factor (K) of erosion models (Godoi et al., 2021), soil water retention by using pedotransfer function approaches (Saxton et al., 1986; Reichert et al., 2000; Schaap et al., 2001; Costa et al., 2013; Botula et al., 2014), the runoff curve number (USDA-SCS, 2007), and fertilizer supply to crops (CQFS-RS/SC, 2016). Thus, the more precise is the determination of soil texture, the more accurate the predicted soil properties and processes will be. This is crucial for farmers and decision-makers.

Soil texture analysis is based on the principle of dispersing all mineral particles without altering them. This principle is done by combining pre-treatments to dissolve cements such as oxides, carbonates and organic compounds and salts, followed by chemical solutions to disperse clays, and shaking for breaking remaining particle binding remaining after chemical dispersion (Gee and Bauder, 1986). It is well known that all these steps may not work flawlessly, but special care has been given to the shaking process, because it can fracture mineral particles (Suzuki et al., 2015). Different shakers have been evaluated, such as the electric mixer and horizontal reciprocating shaker (Kilmer and Alexandre, 1949; Tavares Filho and Magalhães, 2008; Miyazawa et al., 2011; Almeida et al., 2012), and ultrasonic shaker (Genrich and Bremner, 1972; Vitorino et al., 2001).

The official Brazilian methodology (Donagemma et al., 2011) recommends a shaking time of 5 min for sandy soils and 15 min for clayey soils when using an electric mixer at 12,000 rpm. If using a horizontal shaker at 150 cycles per min, a shaking time of 16 h is suggested for all soil types. However, changes in shaking times may be required depending on the presence of cementing agents and differences in intrinsic particle resistance of each soil. For Oxisols (*Latossolos*), a 16 h shaking time with a horizontal shaker and 5 to 20 min with an electric mixer has been recommended (Kilmer and Alexander, 1949; Tavares Filho and Magalhães et al., 2008). For some Oxisols with strongly cemented aggregates, adding coarse sand to force the breakdown of particle binding is also suggested (Tavares Filho and Magalhães et al., 2008; Corá et al., 2009; Rodrigues et al., 2011), but it may increase clay content. Nevertheless, Miyazawa et al. (2011) propose that a shaking time of 2 h in a horizontal shaker allows enough dispersion of Oxisols. Suzuki et al. (2015) subjected five soil classes (Paleudalf, Hapludox, Umbraqualf, Albaqualf and Palludert) to shaking times of 0.5 to 8 h in a horizontal shaker, adding nylon spheres to force the breakdown of particle binding, and recommended 1 to 2 h for kaolinitic soils and 4 h for soils with cementing agents. These studies show that shaking time to disperse all mineral particles (especially silt and clay) without altering them (typically sand) should be calibrated according to the intrinsic characteristics of mineral particles and their aggregation state.

The evaluation of the effect of different shaking settings is particularly scarce for Entisols (*Neossolos*) of hill or mountain terrains covering geological formations such as volcanic, plutonic and metamorphic rocks. In general, Entisols are spread worldwide (Poesen and Lavee, 1994; Ma and Shao, 2008; Throop et al., 2012), covering 13.4 % of the Brazilian territory (Teixeira et al., 2021) and 25 % of the state of Rio Grande do Sul (Lemos et al., 1973). Areas with diversified crop systems have been increasing over Entisols (Streck et al., 2008) in the last decades, especially in Rio Grande do Sul. This is merely a local example of the world trend of increasing crop areas over marginal soils (Bruinsma, 2003; Laurance et al., 2014). Thus, soil characterization also needs to be done in these areas to manage better and prevent degradation.

The particle size composition of Entisols of hill or mountain terrains commonly contains rock fragments with different sizes and degrees of weathering (Soil Survey Staff, 2014; Santos et al., 2018). A significant fraction of sand particles of these Entisols may be formed by fragile, weathered rock fragments instead of resistant minerals

such as quartz and magnetite. These fragile sand particles may be easily broken or eroded by impact and friction during the shaking procedure. Thus, using the current recommended shaking settings (5 or 15 min with an electric mixer at 12,000 rpm or 16 h with a horizontal shaker at 150 cycles per min) may result in the imprecise soil texture determination of these Entisols. Thus, using texture as a predictor in such cases may lead to further negative consequences, such as inaccurate estimates of soil properties and processes.

In summary, it is well known that using different shaking settings may somewhat change sand, silt and clay contents in soils with resistant mineral particles. However, in soils with less resistant particles, the degree of alteration of sand, silt and clay contents by shaking is still understudied. Thus, this study aimed to identify and recommend the best shaking setting to optimize particle dispersion and minimize particle change in Entisols composed of fragile particles such as saprolite fragments.

MATERIALS AND METHODS

Location, soil and sampling

The study was carried out with soil samples collected in the A horizon of a 1.2 ha crop area located in the city of Ivorá, state of Rio Grande do Sul (latitude 29° 30’ 20.1” S, longitude 53° 37’ 34.6” W, and altitude of 477 m) (Figure 1a). The soil is representative of the geomorphological and pedological conditions of the upper part of the transition from the central plain to the plateau of the state of Rio Grande do Sul, Brazil. Entisols are the predominant soils in the region, formed from the weathering of volcanic rocks of the *Serra Geral* Formation. The sampled area has been cultivated with soybean (*Glycine max* L.) in the summer and black oat (*Avena strigosa* Schreb.) in the winter (Figure 1b). Trenches were opened at eight points in the crop field in which the soil profiles were described as Typic Udorthents and Lithic Udorthents (Entisols), according to Soil Taxonomy (Soil
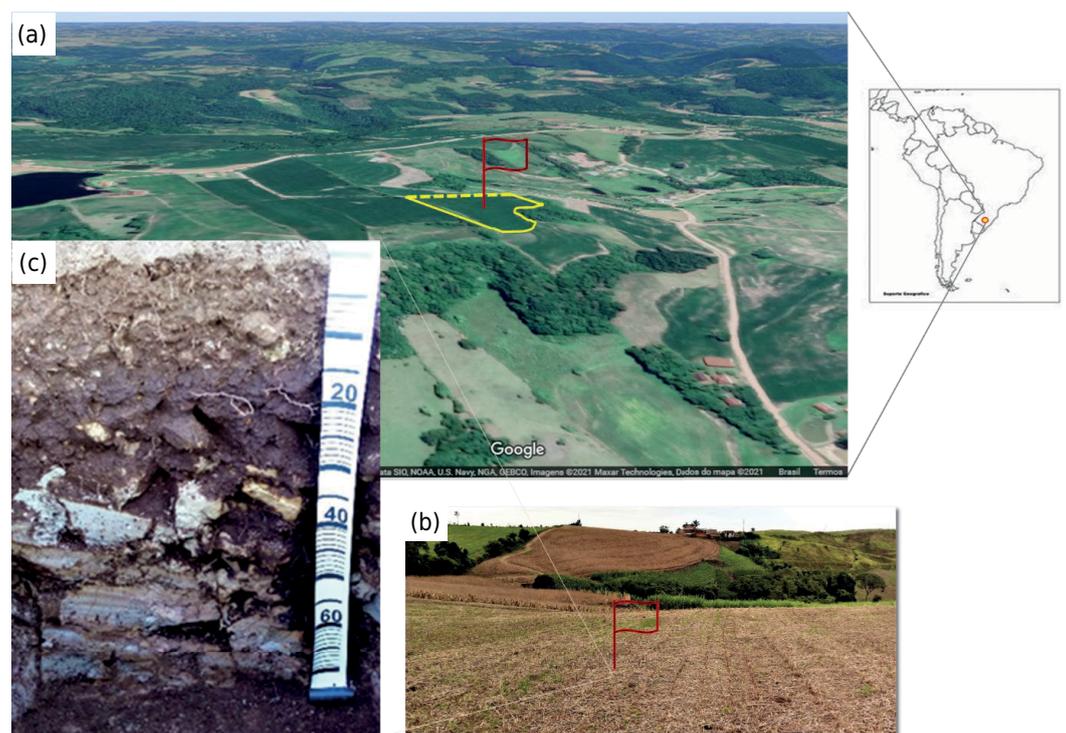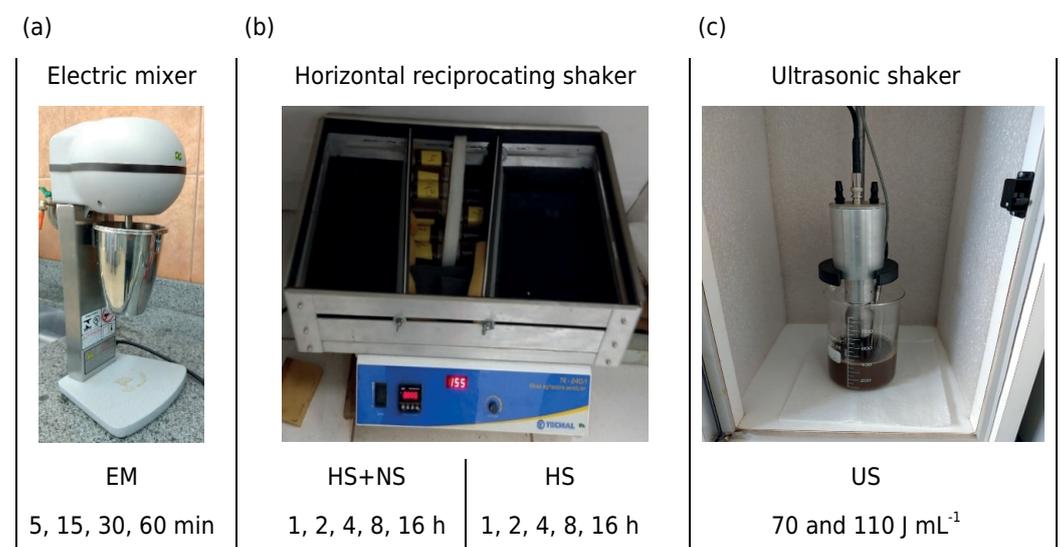

Figure 1. View of the region’s landscape (Google Earth) (a), a characteristic position of the crop area (29° 30’ 20.1” S; -53° 37’ 34.60” W) (b), and the profile of the predominant Entisoil (Typic Udorthents) (c).

Survey Staff, 2014), respectively, these classes corresponding to *Neossolos Regolíticos* and *Neossolos Litólicos* according to the Brazilian Soil Classification System (Santos et al., 2018). A single representative profile of Typic Udorthents was presented (Figure 1c) to illustrate the general soil composition of the area. This profile was characterized by the sequence of horizons A/Cr (0.00-0.12 m), Cr/A (0.12-0.33 m), Cr (0.33-0.60 m) and RCr (0.60-0.70$^+$ m). It is possible to notice coarse fragments of different sizes, some with characteristics and hardness similar to those of volcanic rock, but most in an advanced stage of weathering (easily cut with a shovel or knife) and reduced size, especially in the surface horizon.

Approximately 2 kg of undisturbed soil was collected in the 0.00-0.10 m layer of each point to represent the variability of particle size distribution and mineralogical composition of the A/Cr horizon. These sub-samples were subsequently mixed and homogenized to form a composite sample. In the laboratory, the samples were air-dried. After that, the coarser particles were removed and the remaining aggregated material was carefully disrupted by hand to avoid breaking the fragile particles of saprolite. Finally, the material was passed through a 2 mm mesh sieve to obtain the air-dried fine earth (ADFE).

Physical agents used for dispersing sand, silt and clay

Combinations of equipment with dispersion times and/or energy levels made up the 16 physical dispersion treatments used in this study (Figure 2), which were evaluated in a completely randomized design with six replications (N = 96). The equipment used was an electric mixer (EM), a horizontal reciprocating shaker (Tecnal TE - 240/1) with the use of nylon spheres (HS+NS) and without nylon spheres (HS), and an ultrasonic shaker (Vibra-cell Sonics & Materials VC 750) equipped with a probe (136 mm long and 13 mm ⌀) (US). The treatments consisted of shaking with an electric mixer at 12,000 rpm for 5, 15, 30 and 60 min; horizontal shaking with and without nylon spheres at 150 cycles per min for 1, 2, 4, 8 and 16 h; and sonication with an ultrasonic shaker at 70 and 110 J mL^{-1} for 20 and 30 min, respectively (Figure 2). The shaking times or energy levels used in this study were equal to, smaller and larger than the values suggested in analytical protocols (Gee and Bauder, 1986; Donagemma et al., 2011; Suzuki et al., 2015). This was done to assess the need for maintaining, reducing or increasing time and/or energy to disperse sand, silt and clay without breaking down the sand fraction of the study soil.

(a)	(b)	(c)
Electric mixer	Horizontal reciprocating shaker	Ultrasonic shaker
EM	HS+NS	HS	US
5, 15, 30, 60 min	1, 2, 4, 8, 16 h	1, 2, 4, 8, 16 h	70 and 110 J mL^{-1}

Figure 2. An electric mixer, a horizontal reciprocating shaker and an ultrasonic shaker. (a) Shaking at 12,000 rpm (EM); (b) Shaking at 150 cycles per min with nylon spheres (HS+NS) and without nylon spheres (HS); (c) Ultrasonication at 70 and 110 J mL^{-1} for 20 and 30 min (US), respectively.

Pretreatment to remove iron oxides, salts and organic matter was not necessary. There is a little content of iron oxides and salts in the Entisols of the sampled area (Lemos et al., 1973); and the organic matter content in the area was about 2 %. The sample mass and solution volume were different in some cases because we followed the recommendations of each technique used. For the EM treatments, 20 g of ADFE, 280 mL of distilled water and 10 mL of NaOH 1 mol L^{-1} were transferred to a dispersing cup (Donagemma et al., 2011) and shaken according to the time of each treatment (Figure 2). For the HS+NS treatments, 20 g of ADFE, two nylon spheres (3.04 g, 1.71 cm ø and 1.11 g cm^{-3}), 50 mL of distilled water and 10 mL of NaOH 1 mol L^{-1} was transferred to 100 mL flasks with snap caps (Suzuki et al., 2015) and immediately shaken according to the time of each treatment (Figure 2). The procedures for the HS treatments were the same as for HS+NS, except for the use of nylon spheres. For the US treatments, we used the same proportion of ADFE/dispersant aforementioned and an ADFE/water proportion of 1:40 (Sá et al., 1999). Thus, 10 g of ADFE and 390 mL of distilled water and 5 mL of NaOH 1 mol L^{-1} were transferred to an 800 mL beaker and subjected to 70 and 110 J mL^{-1}. The time for the equipment to reach 70 and 110 J mL^{-1} was approximately 20 and 30 min, respectively.

Separation and measurement of sand, silt and clay

The procedures for separating and measuring sand, silt and clay fractions were the same for all the dispersion treatments. Sand was separated by sieving and clay by the pipette method (Gee and Bauder, 1986). After dispersion, the samples were washed with distilled water over a 0.053 mm mesh sieve. In this procedure, the suspension containing silt and clay fractions was collected in a 1 L graduated cylinder. Sand retained in the sieve was transferred to a metal container and dried in an oven at 105 °C for 24 h. After drying, the total content of sand was measured and separated into coarse (> 0.2 mm) and fine (< 0.2 mm) sand. The fine, coarse and total sand contents were measured on a 2-decimal balance.

The graduated cylinder containing silt and clay suspension was filled with distilled water to a volume of 1 L. The suspension was homogenized for 60 s, and the temperature was measured. Subsequently, the suspension remained at rest in an air-conditioned room until there were no more silt particles from the surface to a depth of 0.05 m. The settling time for silt particles was calculated by Stokes' Law considering particle density of 2.65 Mg m^{-3} (general value suggested for soils with low oxide and organic matter contents), water density and viscosity as a function of temperature and gravitational acceleration of 9.8 m s^{-1} (Donagemma et al., 2011). After the settling time for silt particles, 50 mL of suspension was pipetted at a depth of 0.05 m (Gee and Bauder, 1986), transferred to a beaker with known weight and taken to the oven at 105 °C for 24 h. Finally, the clay content was measured on a 3-decimal analytical balance and the calculations of the percentages of sand, silt and clay were made according to Donagemma et al. (2011).

Additional dispersion and analysis of silt and clay

Based on the results from the procedures described above, we found an increase in clay content correlated with a decrease in coarse sand. However, it was not possible to explain whether the increase in clay content was caused by the breakdown of sand or the greater dispersion of clay with increasing shaking time. For this reason, a complementary procedure was used in order to (i) apply shaking time just enough to separate sand from the other fractions, thus minimizing sand breakdown, and (ii) continue shaking silt and clay separately from sand to verify whether there would be an increase in clay with increasing shaking time.

For this complementary analysis, we only used the horizontal shaker. Initially, 60 samples of 20 g of ADFE were transferred to 100 mL flasks with snap cap in which no nylon

spheres were added. Then, 50 mL of distilled water and 10 mL of 6 % NaOH were added to each flask and the samples were shaken for 1 h. This time was defined based on two observations made in the first step of this study: (i) the water that dripped from the washing of the sand of the samples subjected to horizontal shaking for 1 h was clear, and (ii) the greatest content of sand was also observed in the samples subjected to a shaking time of 1 h. Therefore, one hour was enough time to separate sand from the other fractions, thus minimizing sand breakdown. After shaking, the suspension was washed with distilled water over a 0.053 mm mesh sieve. In this procedure, the suspension containing silt and clay fractions was collected in a 1 L glass flask, whose suspension volume was then adjusted to 750 mL. Sand retained in the sieve was transferred to a metal container and taken to an oven at 105 °C for 24 h. The measurement of total, fine and coarse sand contents was done as previously described.

Subsequently, the 60 suspensions of silt + clay inside the 1 L glass flask were separated into two groups of 30 samples (A and B). Each of these groups was separated once more into five subgroups of six samples. A shaking time was defined for each subgroup. Subgroups 1, 2, 3, 4 and 5 were subjected to shaking times of 1, 2, 4, 6, 8 and 16 h, respectively. As subgroup 1 samples had already been shaken for 1 h to separate sand, they were directly subjected to a settling time for silt and the measuring of clay content (described below). The subgroups 2 to 5 were subjected to the aforementioned shaking times. Samples of group A were shaken without using nylon spheres and those of group B were shaken by adding 15 nylon spheres to each flask. At the end of the shaking, the silt and clay suspensions were transferred to a 1 L graduated cylinder, and the volume of the suspension was adjusted to 1 L. We measured clay content using the pipette method and calculated the percentage of sand, silt and clay as previously described.

Characterization of the mineral particles of coarse sand

For a general characterization of the mineral particles present in coarse sand, replications of coarse sand determined using the treatments with the greatest potential for reducing more fragile particles and increasing the concentration of more resistant particles (EM and HS+NS) were excluded. Therefore, we used the six replications of coarse sand determined at all shaking times with HS (1, 2, 4, 8 and 16 h) and US (70 and 110 J mL^{-1}). Images of the coarse sand particles were taken with a Zeiss Stemi 508 stereo microscope (Carl Zeiss Microscopy GmbH, Jena, Germany) at 50 × magnification. The images were collected using an integrated camera connected to the Zeiss Zen software. Images of the fine sand particles were also taken, but the manual procedure described below only allowed a distinguishable general measurement of the mineralogical composition of the coarse sand fraction.

For measuring the mass ratio of saprolite, quartz, magnetite, rock fragments and aggregates of organic and mineral particles (agglomeration of organic material and small mineral particles formed during the drying process in the oven) in coarse sand, triplicates of 0.2 g of coarse sand were weighed on a 3-decimal balance. Initially, the sample was placed on a white porcelain plate, and the magnetite particles were removed with a magnet. Then, the remaining minerals were identified and separated based on particle color and texture, observed with a HL-410 bench magnifying glass with 8 × magnification (separation of large particles) and Biofocus stereo microscope with 40 × magnification (separation of small particles). Finally, the particle mass in each mineralogical class was measured on a 3-decimal balance and the results were expressed as percentage.

Statistical analysis

The effect of the physical dispersion treatments on particle size fractions was evaluated by analysis of variance. When the effect of treatments was significant (p<0.05), the average contents of silt, sand and clay were compared by the Duncan test (p<0.05).

RESULTS

The general mineralogical composition of coarse sand was 33 % saprolite fragments, 23 % rock fragments and 28 % magnetite fragments, 1 % quartz fragments, and 16 % aggregates of organic and mineral particles (Figure 3c). The predominance of saprolite in coarse sand is noticeable in the image taken with a stereo microscope (Figure 3a). A considerable presence of saprolite was also evident in fine sand with the stereo microscope, although the image reveals this less clearly (Figure 3b). Due to the difficulty in distinguishing and manually separating the minerals of the fine sand fraction, the mineral particles were only measured in the coarse sand fraction.

There was a difference in the content of all particle size fractions associated with shaking methods (Figure 4). The greatest differences were found in the coarse sand (Figure 4a) and clay (Figure 4d) fractions. The average content of the coarse sand fraction ranged from 3.8 % in the HS+NS with a shaking time of 16 h to 17 % in the HS with 1 h of shaking (Figure 4a). The average content of the clay fraction varied from 9.6 % in the US with at 70 J mL^{-1} to 28 % in the EM with a shaking time of 60 min (Figure 4d). There was a variation of 12 to 18 % and 44 to 65 % in the average content of fine sand (Figure 4b) and silt (Figure 4c), respectively.

The most significant trends were a decrease in coarse sand (Figure 4a) and an increase in clay (Figure 4d) with increasing energy in the US and increasing shaking times in the
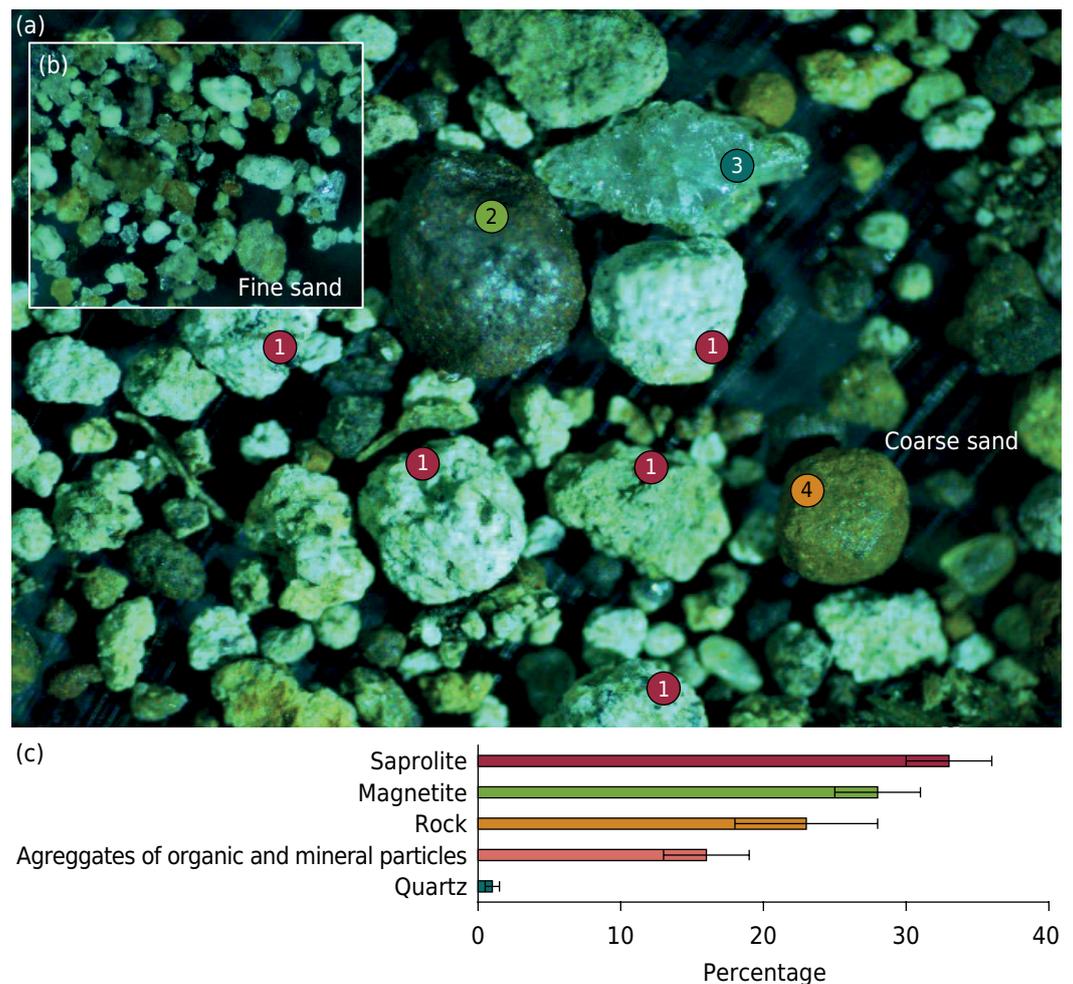
Figure 3. A representative image of the coarse sand components captured with a Zeiss Stemi 508 stereo microscope (Carl Zeiss Microscopy GmbH, Jena, Germany) at 50× magnification (a) and fine sand (b). The numbers 1, 2, 3 and 4 indicate saprolite, magnetite, quartz and rock fragments, respectively. (c) Percentage of mineral fractions in coarse sand dispersed with US at 70 and 110 J mL^{-1} and HS for 1, 2, 4, 8 and 16 h. N = 42. Bars in the columns represent the standard deviation.

HS+NS, HS and EM. In comparing the averages within the same method (US, HS+NS, HS and EM), there were differences between one or more comparisons of each method, both for coarse sand and clay.
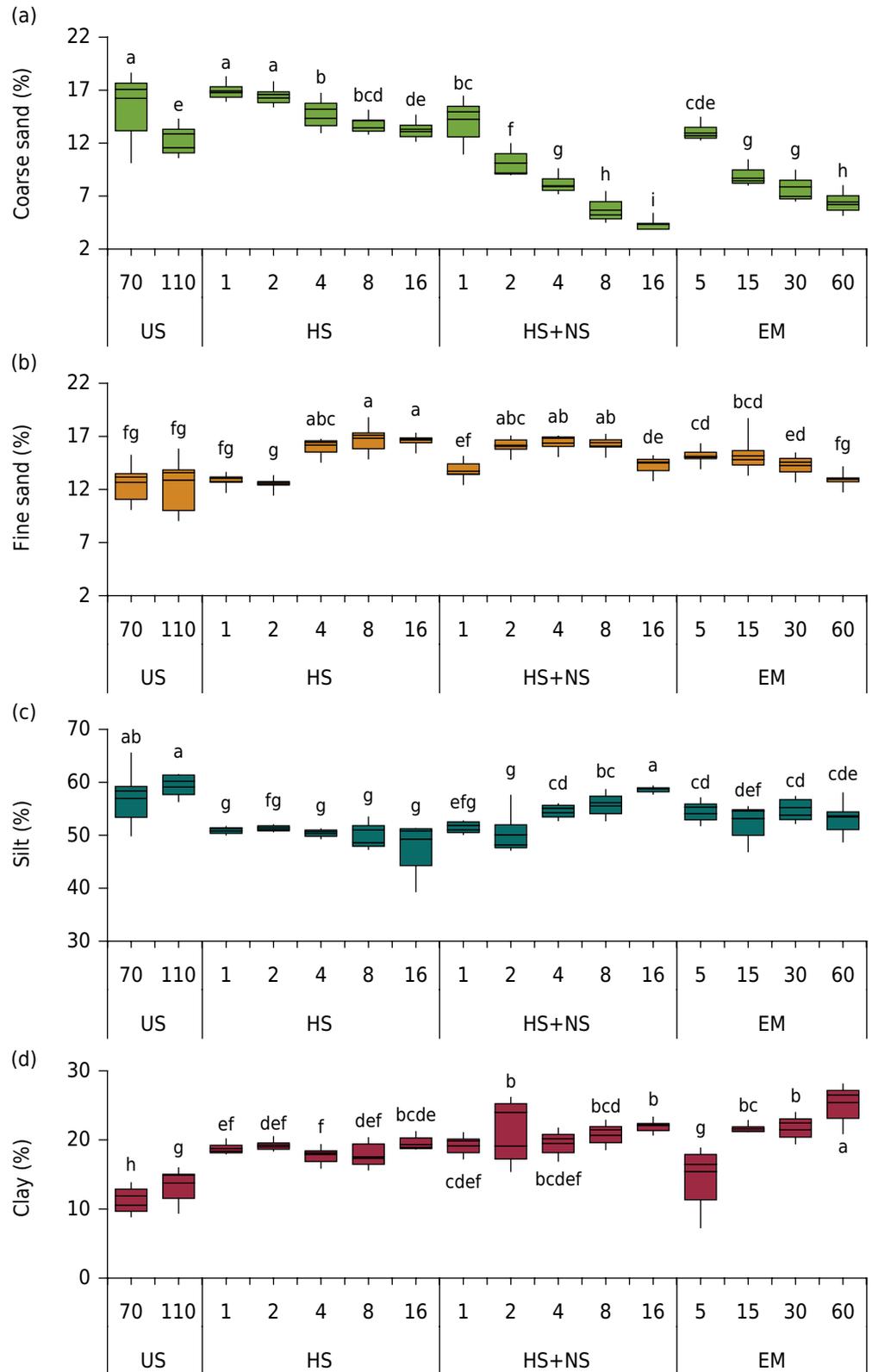

Figure 4. Coarse sand (a), fine sand (b), silt (c) and clay (d) determined with different physical dispersion methods. US: ultrasonication at 70 and 110 J mL^{-1}; HS: horizontal shaking at 150 cycles per min without nylon spheres for 1, 2, 4, 8 and 16 h; HS+NS: horizontal shaking at 150 cycles per min with nylon spheres for 1, 2, 4, 8 and 16 h; EM: shaking with an electric mixer at 12,000 rpm for 5, 15, 30 and 60 min.

In treatments without nylon sphere (HS), the increase in shaking time from 1 to 16 h caused a slight decrease of 5.2 % of coarse sand, while shaking with spheres (HS+NS) caused 11.9 % of reduction of coarse sand with the increase in shaking time from 1 to 16 h (Figure 4a). Consequently, the content of coarse sand after 16 h of shaking without nylon spheres was greater than that of coarse sand after 2 h of shaking with nylon spheres (Figure 4a).

Coarse sand was inversely correlated with fine sand, silt and clay (Figure 5a, 5b and 5c, respectively). The degree of correlation increased in that same order, with Pearson's r going from -0.23 to -0.39 and -0.60. Fine sand was also inversely correlated with silt (r = -0.33). However, its correlation with clay was not significant (r = 0.14) (Figure 5d).

The complementary analysis of silt and clay dispersion with and without spheres in the horizontal shaker carried out after removing the sand (also in the horizontal shaker without the use of spheres and with shaking time of 1 h) showed that the silt (Figure 6c) and clay (Figure 6d) contents were not associated with the increased in shaking time from 1 to 16 h. The minimum and maximum contents of silt were 49 and 55 %, and the minimum and maximum clay contents were 16 and 21 %. The average content of silt at different shaking times varied only 2 % (51 to 53 %) and only 1 % in that of the clay fraction (18 to 19 %). The results of silt and clay contents were not skewed by systematic heterogeneity in coarse (Figure 6a) and fine (Figure 6b) sand. This is because the differences between the replications were no more than 3 % for coarse sand and 1 % for fine sand and were not correlated with the silt and clay fractions.
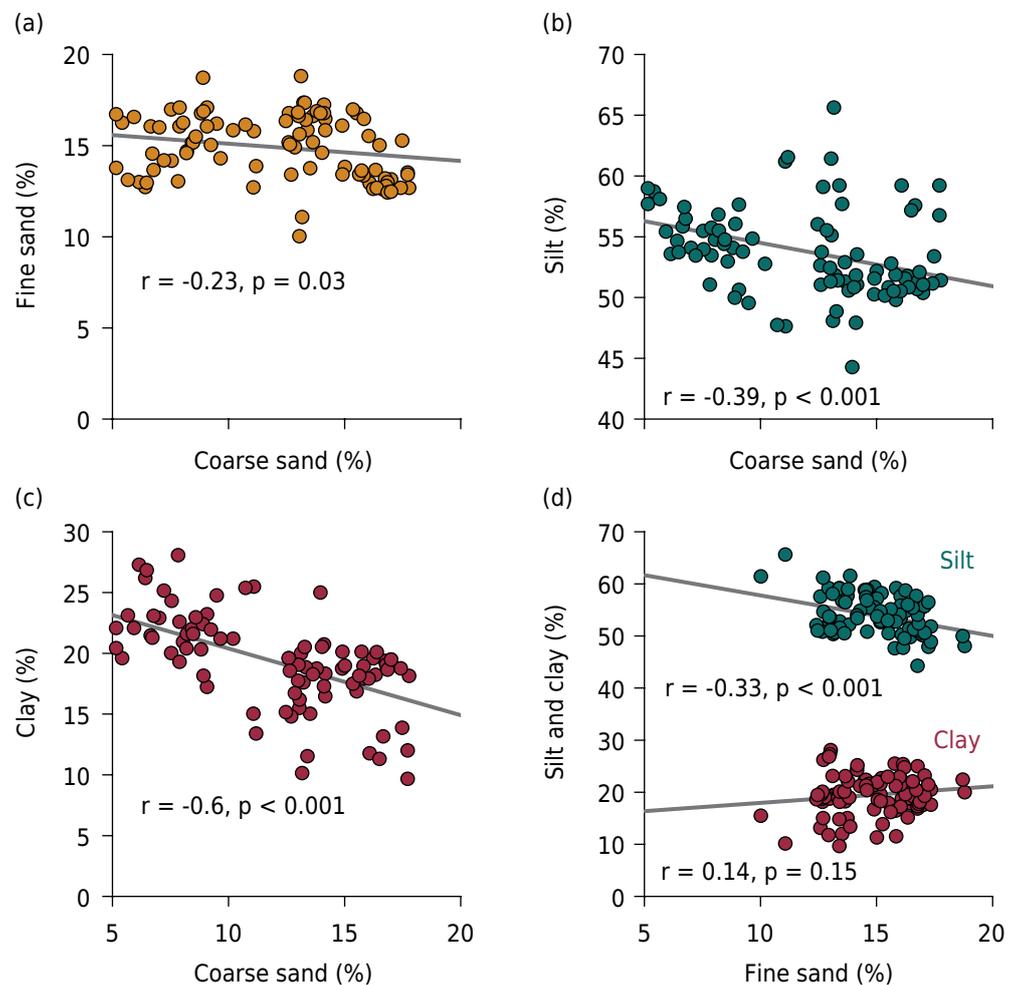
Figure 5. Correlation between particle-size fractions determined with all the treatments. N=96.
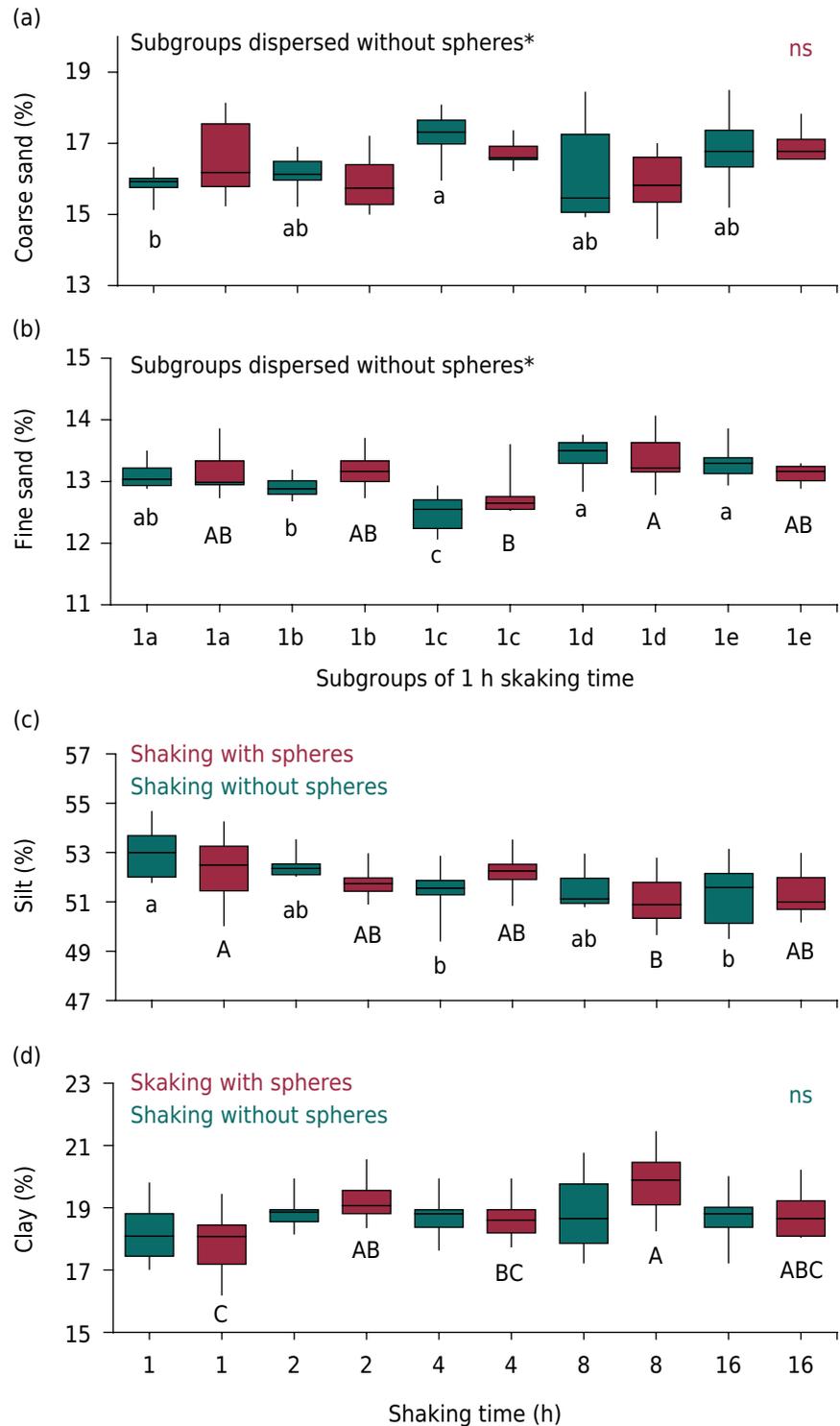
Figure 6. Coarse (a) and fine (b) sand determined after horizontal shaking for one hour without the use of spheres; silt (c) and clay (d) determined after horizontal shaking of up to 16 h with and without the use of spheres. * Sub-samples of coarse and fine sand dispersed without using spheres, corresponding to sub-samples of silt and clay shaken with spheres (red) and without spheres (green).

DISCUSSION

Presence of approximately 1/3 of saprolite in the coarse sand mass (Figure 2c) revealed that there was a considerable amount of fragments with low resistance and subject to breakdown and abrading by friction or impact occurring in the physical dispersion processes used in texture analysis. The change in the sand fraction caused by shaking

is not typically a major problem in soils with sand predominantly made up of crystalline mineral particles with high hardness, such as quartz and magnetite. In such soils, the main problem is promoting efficient dispersion of the clay and silt fraction, which is strongly aggregated by cementing agents such as iron and aluminum oxides (Ferreira et al., 1999; Donagemma et al., 2011). In such cases, an increase in energy or shaking time is generally required to improve the dispersion of the clay and silt fractions (Miyazawa et al., 2011). However, shaking with nylon spheres can fracture even quartz sands after 4 h (Suzuki et al., 2015). As the saprolite in the coarse sand (33 %) and fine sand of the samples used in this study (Figure 2) is less resistant to friction and impact during shaking, there was a considerable reduction in coarse sand content with increasing shaking time or energy, as will be discussed below.

Decrease in coarse sand associated with increasing clay contents (Figures 4a, 4d and 5c) allowed two suppositions. One is that there was a breakdown of coarse sand with the increase in energy and/or shaking time, and the resulting particles of the abrading and breakdown of coarse sand increased the clay content. Another interpretation is that there was no breakdown of coarse sand, and the increase in clay content was a result of better particle dispersion with increasing energy or shaking time. The consequences of the first interpretation would be to decrease energy and shaking time and use the US and HS methods (as they fracture coarse sand and overestimate clay less). As for the second interpretation, the consequences would be the opposite, that is, to increase energy and shaking time and use the HS+NS methods (as they disperse clay more and overestimate coarse sand less).

The fact that there was no difficulty in washing the sand favors the reduction of energy and shaking time and recommends the use of the US and HS methods. In all the cases, the water that passed through the sieve at the end of the washing of sand was not dirty. Visually, the transparency of the water flowing from the sieve was equal to that of the water added to the sieve. Thus, we were not able to notice residual particles of poorly dispersed silt and clay that continued to detach from the sand particles. Therefore, the significant linear decrease in the average content of coarse sand with increasing shaking time with HS+NS (15 % in 1 h to 4 % in 16 h) and with EM (14 % in 5 min to 7 % in 60 min) (Figure 4a) must have been caused by the breakdown and abrading of sand.

However, it is difficult to recommend a shaking method for dispersing clay and silt based on the data in figure 4. Part of the particles produced by the breakdown and abrading of coarse sand may have accumulated in the fine sand fraction, as there was a negative correlation between fine sand and coarse sand (Figure 5a) as well as a significant increase in fine sand with 4, 8 and 16 h of shaking in the HS and with 2, 4 and 8 h of shaking in the HS+NS (Figure 4b). There was also a negative correlation between silt and coarse sand (Figure 5b) and an increase in silt content with 4, 8, and 16 h of shaking in the HS+NS (Figure 4c). If all the particles of the breakdown of coarse sand accumulated in the fine sand and silt fractions, then clay-sized particles were not produced by shaking. Consequently, the increase in clay content with increasing energy and/or shaking time shows that clay fraction requires a greater energy and/or shaking time than those required to disperse sand.

The lack of correlation between shaking time of silt + clay separated from the sand as well as the amount of clay or silt from the complementary analysis (Figure 6) revealed that a shaking time of 1 h in the horizontal shaker without using spheres was enough to disperse clay and silt. Therefore, the increase in clay content with increasing shaking time in HS and HS+NS, in which all the fractions remained together until the end of the shaking (Figure 4c), was due to the breakdown and abrading of the sand fraction. These phenomena must also have been responsible for reducing coarse sand and increasing clay content in the US and EM methods (Figures 4a and 4d).

We noticed in our study that many of the physical dispersion treatments changed the sand, silt and clay contents of Entisols with the presence of saprolite fragments in the fine earth fraction. These physical dispersion treatments could possibly affect Leptosols and Cambisols, in which saprolite fragments may be present. Thus, for the particle size analysis of soils with mineralogical composition similar to the one used in this study, we recommend the HS method with a shaking time of 1-2 h to minimize the breakdown of sand and allow enough dispersion of silt and clay.

Other settings can be used depending on operational preferences, equipment availability and application of the results. If there is no need to discriminate total sand into coarse and fine sand and if changes of up to 5 % in total sand, silt and clay fractions are acceptable, shaking times of 2 to 16 h in HS, 2 to 4 h in HS+NS, 5 to 30 min in EM could be used (Figure 4). It can be noticed that in all treatments the differences in sand (total), silt and clay fractions were at most 5 %. However, we evaluated a composed sample (see methodology section) from which only an average frangibility of sand could be assessed. Evaluating apart each location of the sampled area may show a higher degree of sand breakdown in some spots. Besides knowing how many sand particles are broking while shaking, it is important to take into account the effect of changing sand, silt and clay contents on predicted soil properties, as the soil erodibility factor (K) (Godoi et al., 2021), soil water retention estimated by pedotransfer functions (Saxton et al., 1986; Reichert et al., 2000; Schaap et al., 2001; Costa et al., 2013; Botula et al., 2014), the runoff curve number (USDA-SCS, 2007), and fertilizer supply to crops (CQFS-RS/SC, 2016). This is a more rational way to set an operational physical dispersion option. However, if total sand should be discriminated into coarse and fine sand, we recommend only using the HS method with a shaking time of 1-2 h for Entisols with fragile particles.

CONCLUSION

Most shaking times used in the horizontal shaker with and without nylon spheres (1, 2, 4, 8 and 16 h) and in the electric mixer (5, 15, 30 and 60 min), and energy levels used in ultrasonic shaker (70 and 110 J mL^{-1}) change the particle size fractions of Entisols with saprolite fragments. The main changes were the decrease in coarse sand caused by abrading and breakdown with a resulting increase in clay content due to the deposition of such particles.

For particle size analysis of soils with mineralogical composition similar to the one used in this study, we recommend horizontal shaking without nylon spheres for 1-2 h to minimize the breakdown of sand and ensure enough dispersion of silt and clay.

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