

## TIMES OF AGITATION AND CONCENTRATION OF NaOH TO CHARACTERIZE THE SOIL TEXTURE

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### ABSTRACT:

The particle size distribution is an important index to assess the physical, chemical and biological attributes of a soil with practical applications in erosion and adsorption of nutrients. Thus, the aim of the present study was to test the effects of different concentrations of NaOH and agitation time for the particle-size distributions of four soils from the western region of Santa Catarina State, Brazil. Four soils were collected, being Humic Cambisol, Histosols, Red Ferralsols and Red Nitisols, by sampling "A" horizon of the Cambisol, "H" horizon of the Histosols, and "B" horizons of the Ferralsols and the Nitisols. The hydrometer method was used to evaluate the texture through the variation of the agitation time (4, 8, 12, 16, 24 and 30 hours) and variation in concentrations of the chemical dispersant (NaOH: 0.1; 0.5; 1 and 2 mol L<sup>-1</sup>) only at 16 hours of agitation. The clay content in the Cambisol was stabilized with the agitation time of 22.52h and 31.28h in the Ferralsols. An adequate agitation time was not observed for the Histosols and the Nitisols was not influenced by the agitation times. For the concentrations of the chemical dispersant (NaOH), the clay content of the Ferralsols was stable with 1.16 mol L<sup>-1</sup> and the Nitisols with 0.92 mol L<sup>-1</sup>. The Cambisol and the Histosols, however, showed no stabilization of the clay content.

**Keywords:** Cambisol, granulometry, Ferralsols, Nitisols, Histosols

## TEMPOS DE AGITAÇÃO E CONCENTRAÇÃO DE NaOH PARA CARACTERIZAÇÃO DA TEXTURA DE SOLOS

### RESUMO:

A granulometria é um atributo importante para as propriedades físicas, biológicas e químicas do solo, com aplicações práticas na erosão e adsorção de nutrientes. Desta forma, o trabalho teve por objetivo testar o efeito de concentrações de NaOH e tempos de agitação em quatro solos da região oeste de Santa Catarina, Brasil, sobre a granulometria. Coletou-se quatro solos, sendo Cambissolo Húmico, Organossolo Háptico, Latossolo Vermelho e Nitossolo Vermelho, amostrando-se os horizontes "A" do Cambissolo e H do Organossolo e os horizontes "B" do Latossolo e Nitossolo. O método para avaliação da textura foi o densímetro, onde se testou o tempo de agitação (4, 8, 12, 16, 24 e 30 h) e concentrações de dispersantes químicos (NaOH: 0,1; 0,5; 1 e 2 mols L<sup>-1</sup>) em 16 horas de agitação. Para o Cambissolo no tempo de agitação de 22,52h ocorreu à estabilização do teor de argila e para Latossolo foi de 31,28h. O Organossolo não se observou um tempo adequado e o Nitossolo não teve influência dos tempos de agitação. Para concentrações do dispersante químico NaOH, o Latossolo teve estabilização do teor de argila em 1,16 mol L<sup>-1</sup> e o Nitossolo em 0,92 mol L<sup>-1</sup>. No entanto, o Cambissolo e o Organossolo não apresentaram estabilização do teor de argila.

**Palavras-chave:** Cambissolo, granulometria, Latossolo, Nitossolo, Organossolo

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## INTRODUCTION

Particle size distribution analysis (PSDA) of a soil consists of determining the size distribution of individual soil particles in classes designated as sand, silt and clay, which are derived from factors of formation such as the material of origin (Klein et al., 2013). The analysis characterizes the soil and improves management practices by describing the compaction behavior, water retention, capacity of cation exchange, and the absorption of nutrients by plants (Klein et al., 2013).

There are several systems currently in use to classify the sizes of soil particles. However, as suggested by Atterberg, only two systems have been recognized by the soil sciences as important, the North American System (United States Department of Agriculture - USDA) and the International System (IS). (Carvalho, 2011). According to the USDA, and the classification system adopted in Brazil, clay, silt, and sand fractions are identified as follows: Clay fraction is identified by the group of particles with a diameter of less than 0.002 mm; silt particles with a diameter between 0.002 and 0.05 mm, and the sand fraction is composed of particles measuring between 0.05 and 2.0 mm in diameter (Lier, 2010).

The hydrometer (Boyucos, 1951) and the pipette (Day, 1965) methods are the most common methods employed to determine the soil particle-size distribution (PSD). The pipette method is more accurate and is used to calibrate other methods. The hydrometer method is faster and more cost effective; however, it is based on the sedimentation of particles present in the soil (Veiga, 2011). Based on Stokes' Law, a fixed time is used to determine the suspension density, or total clay after a chemical dispersant is applied. The coarse fractions (fine and coarse sand) are separated by gravity decanting for three minutes. The clay is then measured by the density of the fluid with a densitometer, and the silt is obtained by the difference of the observations (Embrapa, 1997). The hydrometer method is the most used by commercial laboratories (that provide services) and the pipette method is mostly recommended for scientific research. Several laboratories from the western region of Santa Catarina State, however, employ either method. Ultimately, this may weaken reliable comparisons or conclusions of the results obtained. Furthermore, these laboratories apply the same methodology to different types of soils. Ideally, separate methodologies are calibrated for the clayey soils and the organic soils. The laboratories ultimately become more efficient to conduct research and perform services, to attain more accurate results by applying the most effective and

standardized methods. In turn, the standardization increases the trust of the producers in relation to the correction and the fertilization of the soil.

The quality of the PSDA has been questioned in previous studies Jorge, Paula and Menk (1985), for example, have emphasized that the standardization of analytical methods enables comparisons and facilitates the interpretation of data obtained by different institutions in Brazil. The authors compared the methods of particle-size analysis (PSA) of soils used by the National Survey and Conservation Service of Soils (SNLCS/Embrapa) and the Agronomic Institute of Campinas (IAC). They concluded the two methods produced the same results only for the clay fraction. Ruiz (2005) demonstrated that the additional collection of suspension (silt fraction + clay fraction), increases the accuracy of results. He also demonstrated that the calculation of silt fraction by subtracting the other fractions overestimates the silt fraction. This is because the calculation accumulates the total of all the errors of determination.

Thus, testing different concentrations of dispersants and agitation times to determine the texture of a soil more accurately is of relevance. In addition, the standardization of analytical methods would benefit the western region of Santa Catarina State, which comprises a wide variety of soils with high compositions of clay and organic material. The objective of this study was to standardize a method to determine the texture of the soils predominant in the western region of Santa Catarina State by testing concentrations of dispersants and agitation times.

## MATERIALS AND METHODS

The experiment was carried out in the soil laboratory of Western University of Santa Catarina (Universidade do Oeste de Santa Catarina-Unoesc), campus of Xanxerê, SC, Brazil. Initially, four different soils were selected and sampled from the region, with variations in the texture and the organic matter content. The soils were sampled on November 2014 in the following classifications and coordinates: Humic Cambisol (latitude 26°54'48" S and longitude 51°58'15.8" W); Haplic Histosols (latitude 26°51'30.3" S and longitude 52°23'23.5" W); Dystrophic Red Ferralsols (latitude 26°52'28.7" S and longitude 52°25'17.7" W) and Red Nitisols (latitude 26°52'59.6" S and longitude 52°25'56.4" W) Datum: Sirgas 2000.

The soils were sampled using an auger and a slice shovel in the A horizons of the Humic Cambisol, the O horizon of the Histosol, and the B horizons of the Ferralsols and the

Nitisols. The soils were then air-dried and passed through a 2 mm mesh sieve, and then the chemical properties (Table 1) were determined using the methodology of Tedesco et al. (1995).

**Table 1.** Chemical composition and particle density of the four soils used in the experiment.

Solis	pH water	pH SMP	P	K	MOS <sup>1</sup>	Al	Ca	Mg	V <sup>2</sup>	Dp <sup>3</sup>
			--mg dm <sup>-3</sup> --	%	----cmol <sub>c</sub> dm <sup>-3</sup> ----	%	g dm <sup>-3</sup>			
Cambisol	4.90	4.38	11.48	45.2	5.75	6.55	1.77	0.43	8.20	2.44
Histosols	4.40	3.94	88.34	54.2	9.29	10.45	2.39	1.49	9.18	2.18
Ferralsols	5.23	4.44	5.41	63.3	1.80	4.50	0.66	1.22	8.22	2.44
Nitisols	4.63	5.28	7.18	27.1	1.18	1.60	4.74	1.87	7.80	2.57

<sup>1</sup>MOS = Organic Material of the Soil.

<sup>2</sup>V = Saturation Percentage by Bases.

<sup>3</sup>Dp = Density of particles.

The tests were based on the hydrometer methodology (Bouyoucus, 1951) modified by Veiga (2011). The dispersion time was first tested, ranging at 4, 8, 12, 16, 24 and 30 hours for 1 mol L<sup>-1</sup> (methodological standard), in slow agitation (120 rpm) in a horizontal agitator. Then, the concentrations of chemical dispersant (NaOH) were tested at 0.1, 0.5, 1, and 2 mol L<sup>-1</sup> only for 16 hours of slow agitation (methodological standard). Thus, the study comprised six agitation times applied to 4 types of soils, and 4 dispersant concentrations for the standard time of 16 hours where each treatment had six replicates.

Soil samples of 50 g were weighed for each test and placed in plastic vials with 100 mL of distilled water, and 10 mL of chemical dispersant at the experimental concentrations. The solutions were then placed on the horizontal agitator and agitated at the experimental times. Each solution was then transferred to 1000 mL beakers and completed with distilled water. The solution was agitated manually for 3 minutes and observations were carried out with a hydrometer at 3 minutes (observation 1) and at 3 hours (observation 2). The clay, silt and sand contents were calculated from the two observations, where:

$$\text{Clay Content (\%)} = (\text{observation 2}) \times 2;$$

$$\text{Silt Content (\%)} = (\text{observation 2} - \text{observation 1}) \times 2;$$

$$\text{Sand Content (\%)} = 100 - \text{Clay Content} - \text{Silt Content}$$

The data was then subjected to a regression analysis using both simple linear regression and quadratic equation as follows:

$$\text{Linear regression: } Y = b_1 + b_2x, \text{ where } Y =$$

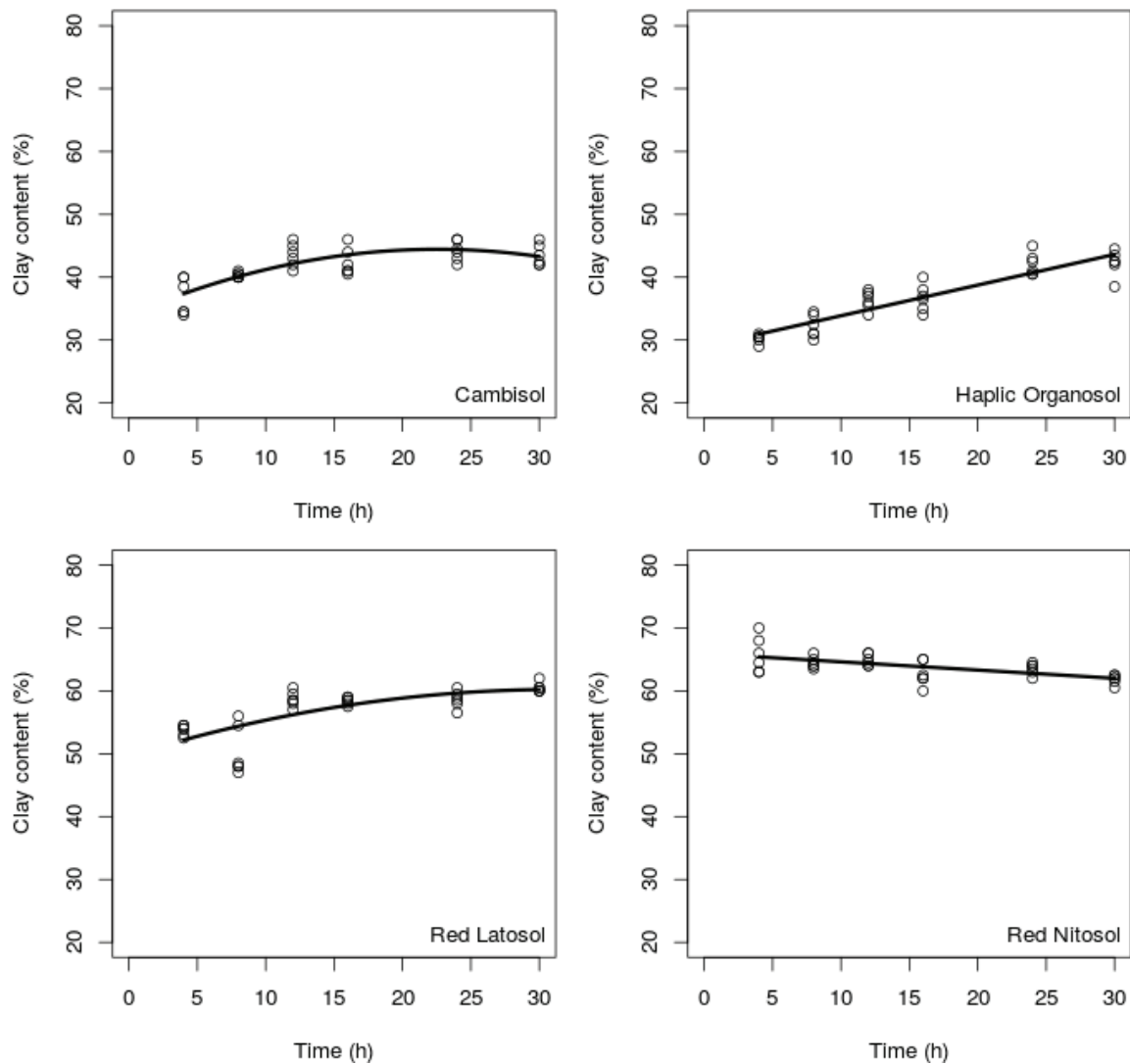
corresponds to the measured response,  $x$  is the time or concentration,  $b_1$  is the intercept (value of the response for  $x=0$ ) and  $b_2$  is the angular coefficient.

Quadratic equation used a parametrization proposed by Zeviani (2013):  $Y = y_m + c(x + x_m)^2$ , where  $y_m$  is the maximum response,  $x_m$  is the time or concentration that gives maximal response, and  $c$  is the parameter that controls the function form.

Mitscherlich model was also applied using a parameterization as proposed by Zeviani (2013):  $Y = b_1 * (1 - \exp\{\log(1 - 0.99) * ((x - b_1) / (b_2 - b_1))\})$ , where  $b_1$  is the maximum asymptote (maximum value) and  $b_2$  is the time or concentration that reaches 99% of the maximum asymptote. Data analyses were performed using the R statistical system (R Core Team, 2015) and the add-on package nls.

## RESULTS AND DISCUSSION

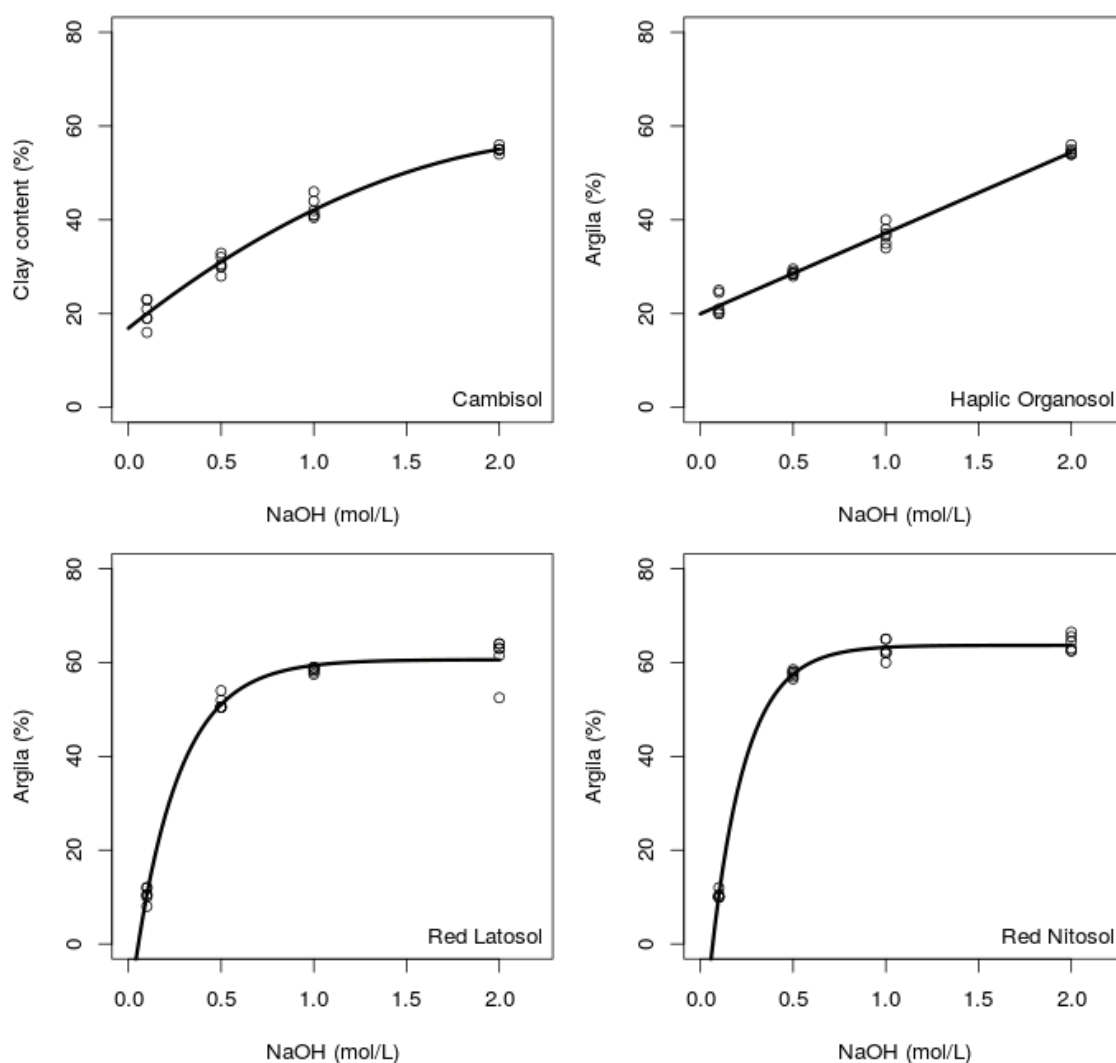
The agitation time to stabilize the clay content in the soil varied for each type of soil. The clay contents for Humic Cambisol, Haplic Histosols, Red Ferralsols and Red Nitisols, ranged from 4 to 30 hours of agitation (Figure 1). The concentrations of the chemical dispersants ranged from 0.1 to 2 mol L<sup>-1</sup> at the standard agitation time (16 hours) for each of the different orders of soil (Figure 2). The parameters estimated from the equations obtained in the different soils are presented in Table 2.



**Figure 1.** Clay Content (%) obtained in different soils subjected to agitation times with the chemical dispersant NaOH at  $1 \text{ mol L}^{-1}$ .

Simple Linear regression ( $Y=b_1+b_2x$ ), where  $Y$  corresponds to the measured response,  $x$  is the time or concentration,  $b_1$  is the intercept (value of the response for  $x=0$ ) and  $b_2$  is the angular coefficient. For the quadratic equation, a parametrization proposed by Zeviani (2013):  $Y=y_m+c(x+x_m)^2$ , was used. Where  $y_m$  is the maximum response,  $x_m$  is the time or

concentration that gives maximal response and  $c$  is the parameter that controls the function form. In Mitscherlich's model, was also used a parametrization proposed by Zeviani (2013):  $Y=b_1*(1-\exp\{\log(1-0.99)*((x-b_1)/(b_2-b_1))\})$  where  $b_1$  is the maximum asymptote (maximum value) and  $b_2$  is the time or concentration that reaches 99% of the maximum asymptote.



**Figure 2.** Clay Content (%) obtained in four soils subjected to the standard agitation time (16 h) with different concentrations of the chemical dispersant NaOH at  $1 \text{ mol L}^{-1}$ .

The clay content of the Cambisol was observed to stabilize at 44.44% over 22.52 hours (Table 2). For the Ferralsols, the clay content stabilized at 31.28 hours at 60.22%. The clay content of the Histosols increased with the increasing

agitation times. This suggested a greater agitation time was needed to stabilize the clay content. The Nitisols did not demonstrate a sufficient agitation time, suggesting that the agitation times did not influence the variation of clay content.

**Table 2.** Parameters estimates from the equations in the different soils subjected to times of agitation with the chemical dispersant NaOH at 1 mol L<sup>-1</sup> and from the soils subjected to concentrations of chemical dispersant (NaOH) only agitation time of 16h.

	Soil Agitation time (hours)		NaOH Concentration (mol L <sup>-1</sup> )	
	Ym	Xm	Ym	xm
<b>Quadratic Linear Model</b>				
Cambisol	44.44	22.52	56.93	2.55
Ferralsols	60.22	31.28	-	-
<b>Simple Linear Model</b>				
	b <sub>1</sub>	b <sub>2</sub>	b <sub>1</sub>	b <sub>2</sub>
Histosols	28.99	0.48	28.99	0.48
Nitisols	65.92	-0.13	-	-
<b>Mitscherlich Model</b>				
	b <sub>1</sub>	b <sub>2</sub>	b <sub>1</sub>	b <sub>2</sub>
Nitisols	-	-	63.65	0.92
Ferralsols	-	-	60.60	1.16

Menezes et al. (2010) evaluated the influence of different agitation times on the dispersions of particles in soils from the Brazilian Cerrado. He perceived a significant reduction in the proportion of silt and an increase in the clay fraction occurred for the Ferralsols and Argisol orders when the agitation time was greater than 8 hours. This study found similar results, but the increase in clay content was significant greater up to 30 hours of agitation (Figure 1 - Ferralsols).

Oliveira et al. (2002) observed that the mechanical dispersion method combined with a horizontal helical movement for 3 hours, and with the addition of 30 g of coarse sand, improved the efficiency of the dispersion of clay in Ferralsols, when compared to the jigger method. Miyazawa and Barbosa (2011) observed that two hours of agitation was sufficient to dissociate the particles in the studied Ferralsols (Dystrophic Red Ferralsols, Dark-Red Ferralsols, and Dystrophic Una Ferralsols). This study differs from the previous study, showing a time significantly greater, of 31.28 hours for the Ferralsols (Table 2).

In contrast to the results found in this study, Suzuki et al. (2004) perceived that the necessary agitation time is greater than 4 hours in samples of Dystrophic Red Nitisols.

Rodrigues et al. (2009) compared the contents of sand, silt and clay in soils from the state of Ceará State/Brazil (Chromic Luvisol, FluvicNeosol, Quartzarenic Neosol, Regolithic Neosol, Red Argisol, Haplic Cambisol, Argilluvic Plinthosol, Red Ferralsols) and observed that the horizontal

agitation for a minimum time (of 6 hours) was more efficient than the rapid agitation for these soils.

The clay content of Cambisol has a tendency to stabilize, which did not occur in the Histosols regardless of the NaOH concentrations (Figure 2). For the Histosols, the best fit was a simple linear regression equation (Table 2), demonstrating that the higher concentration of NaOH further disseminates the clay particles attached to silt and sand. In the Ferralsols and the Nitisols, the clay was stabilized at the concentrations of 1.16 mol L<sup>-1</sup> and 0.92 mol L<sup>-1</sup> NaOH, respectively (Table 2). Thus, the concentration recommended by the literature (1 mol L<sup>-1</sup>) for the Ferralsols is not ideal and is over estimated for the Nitisols. Miyazawa and Barbosa (2011) affirm that the clay aggregates in the Ferralsols become completely fragmented with mechanical agitation and the application of NaOH at mol L<sup>-1</sup>, thus over-estimating the clay content.

Similar results for the Red Ferralsols were observed by Sousa Neto et al. (2009), affirming that the pretreatment with HCl 1 mol L<sup>-1</sup> + NaOH 1 mol L<sup>-1</sup> is more efficient in the complete dispersion of the soil samples than the treatment with basic dispersants (NaOH 0.1 mol L<sup>-1</sup>; NaOH 1 mol L<sup>-1</sup> and calgon 1 mol L<sup>-1</sup>).

The soils evaluated in the present study showed different responses to the various agitation times and the concentrations of the chemical dispersant. The Nitisols did not demonstrate a sufficient agitation time perhaps due to having a well-formed structure (Figure 1 - Nitisols). An ideal

agitation time was not determined for the Haplic Histosols perhaps due to the high content of organic material in this soil. Further studies, however, are recommended to observe the influence of greater agitation times for this soil order.

## CONCLUSIONS

The ideal agitation time for Cambisol and the Ferralsols is 22.52 hours and 31.28 hours, respectively.

The Histosols did not exhibit an adequate agitation time and the Nitisols was not influenced by the agitation time.

The results of this study suggest there should be an increase in the concentration of the chemical dispersants in the standard methodology for Ferralsols and a decrease for the Nitisols. The clay content in the Cambisol and the Histosols did not stabilize with the different concentrations of NaOH.

It was verified that there is a different agitation time and different concentration of the dispersant for each type of soil. This is more likely a result of high soil organic matter content as well as clay content and mineralogy.

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## REFERENCES

- Bouyoucos, G. J. A recalibration of the hydrometer method for making mechanical analysis of soil. Am. (1951). Sociedade Nacional de Agricultura 43(9): 434-438.
- Carvalho, A. A. A. A. (2011). **Avaliação das áreas de preservação Permanente de curso d'água na área de proteção de manancial do córrego quinze, Distrito Federal**. Brasília-DF: Universidade de Brasília. 145p. Dissertação de Mestrado.
- Day, P.R. (1965). Particle fractionation and particle-size analysis. In: BLACK, C.A. (Ed.). **Methods of soil analysis**. Vol. 1. Madison: American Society of Agronomy. p.545-566.
- EMBRAPA (1997). **Manual de Métodos de Análise de Solo**. 2. ed. Rio de Janeiro: (EMBRAPA-CNPS. 212p.
- Jorge, J.A.; Paula, J.L. & Menk, R.F. (1985). Comparação de dois métodos de análise granulométrica de solos utilizados pelo SNLCS/EMBRAPA e IAC. **Pesquisa Agropecuária Brasileira** 20(7): 865-871.
- Klein, V. A.; Maldoso, T, Reichert, J. M.; Suzuki, L. E. A. S.; Veiga, M.; Albuquerque, J. A. & Pauletto, E. A. (2013). Metodologias de controle de qualidade de análises granulométricas do solo. **Ciência Rural** 43(5): 850-853 2013. Disponível em: <http://www.scielo.br/pdf/cr/v43n5/a14913cr6870.pdf>. Acesso em: 22 jun. 2018.
- Lier, Q. J. V. (2010). **Física do solo**. Viçosa: Sociedade Brasileira de Ciência do Solo. 298p.
- Menezes, M. M. M.; Viana, J. H.; Costa, A. M.; Neto, M. M. G. & Chagas, P. G. (2010). **Avaliação da composição granulométrica de diferentes solos do Cerrado submetidos a diferentes tempos de agitação**. Documento Embrapa. Disponível em: <https://ainfo.cnptia.embrapa.br/digital/bitstream/item/29553/1/Avaliacao-composicao-granulometrica.pdf>. Acesso em: 27 mar. 2018
- Miyazawa, M. & Barbosa, G. M.C. (2011). Efeitos da agitação mecânica e matéria orgânica na análise granulométrica do solo. **Revista Brasileira de Engenharia Agrícola Ambiental** 15(7): 680-685. Disponível em: <http://www.scielo.br/pdf/rbeaa/v15n7/v15n07a05.pdf>. Acesso em: 22 jun. 2018.
- Oliveira, G. C; Dias Junior, M. S.; Vitorino, A. C. T.; Ferreira, M. M.; Sá, M. A. C. & LIMA, J. M. (2002). Agitador horizontal de movimento helicoidal na dispersão mecânica de amostras de três Latossolos do sul e campos das vertentes de Minas Gerais. **Ciência Agrotecnologia** 26(5): 881-887.
- R Core Team R. (2015). **A language and environment for statistical computing**. R Foundation for Statistical Computing, Vienna, AT. Disponível em: <https://www.R-project.org/>. Acesso em: 09 nov. 2017.
- Rodrigues, W. S.; Lacerda, N. B. & Oliveira, T. S. (2009). Análise granulométrica em solos de diferentes classes por agitação horizontal. **Revista Ciência Agronômica** 40(4): 474-485.
- Ruiz, H.A. (2005). Incremento da exatidão da análise granulométrica do solo por meio da coleta da suspensão (silte + argila). **Revista Brasileira Ciência Solo** 2(29): 97-300.
- Tedesco, M. J.; Gianello, C.; Bissani, C.A.; Bohnen, H. & Volkweiss, S.J. (1994). **Análises de solo, plantas e outros materiais**. Porto Alegre: UFRGS-Departamento de Solos. 174p.
- Sousa Neto, E. L. de; Figueiredo, L. H. A. & Beutler, A. N. (2009). Dispersão da fração argila de um Latossolo sob diferentes sistemas de uso e dispersantes. **Revista Brasileira de Ciência do Solo** 33(3): 723-728.
- Veiga, M. (2011). **Metodologia para coleta de amostras e análises físicas do solo**. Florianópolis: Epagri. 52 p.
- Zeviani, W. M. (2013). **Parametrizações interpretáveis em modelos não lineares**. Lavra: Universidade Federal de Lavras. 146p. Tese de Doutorado. Disponível em: <http://repositorio.ufla.br/bitstream/1/917/1/TESE%20Parametriza%C3%A7%C3%B5es%20interpret%C3%A1veis%20em%20modelos%20n%C3%A3o%20lineares.pdf>. Acesso em: 01 dez. 2017.