



Revista Brasileira de Ciência do Solo

ISSN: 0100-0683

revista@sbc.org.br

Sociedade Brasileira de Ciência do Solo
Brasil

Tavares Filho, João; Moraes de Césare Barbosa, Graziela; Aparecida Ribon, Adriana
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Revista Brasileira de Ciência do Solo, vol. 34, núm. 5, 2010, pp. 1527-1534
Sociedade Brasileira de Ciência do Solo
Viçosa, Brasil

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WATER-DISPERSIBLE CLAY IN SOILS TREATED WITH SEWAGE SLUDGE⁽¹⁾

João Tavares Filho⁽²⁾, Graziela Moraes de Césare Barbosa⁽³⁾ & Adriana Aparecida Ribon⁽⁴⁾

SUMMARY

A by-product of Wastewater Treatment Stations is sewage sludge. By treatment and processing, the sludge is made suitable for rational and environmentally safe use in agriculture. The aim of this study was to assess the influence of different doses of limed sewage sludge (50 %) on clay dispersion in soil samples with different textures (clayey and medium). The study was conducted with soil samples collected from native forest, on a Red Latosol (Brazilian classification: Latossolo Vermelho distroférrico) loamy soil in Londrina (PR) and a Red-Yellow Latosol (BC: Latossolo Vermelho-Amarelo distrófico) medium texture soil in Jaguapitã (PR). Pots were filled with 3 kg of air-dried fine earth and kept in greenhouse. The experiment was arranged in a randomized block design with six treatments: T1 control, and treatments with limed sewage sludge (50 %) as follows: T2 (3 t ha⁻¹), T3 (6 t ha⁻¹), T4 (12 t ha⁻¹), T5 (24 t ha⁻¹) and T6 (48 t ha⁻¹) and five replications. The incubation time was 180 days. At the end of this period, the pots were opened and two sub-samples per treatment collected to determine pH-H₂O, pH KCl (1 mol L⁻¹), organic matter content, water-dispersible clay, ΔpH (pH KCl - pH-H₂O) and estimated PZC (point of zero charge): PZC = 2 pH KCl - pH-H₂O, as well as the mineralogy of the clay fraction, determined by X ray diffraction. The results showed no significant difference in the average values for water-dispersible clay between the control and the other treatments for the two soil samples studied and ΔpH was the variable that correlated best with water-dispersible clay in both soils.

Index terms: biosolids, flocculation, PZC, ΔpH, organic matter.

⁽¹⁾ Received for publication in December 2010 and approved in July 2010.

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RESUMO: ARGILA DISPERSA EM ÁGUA EM SOLOS TRATADOS COM LODO DE ESGOTO

As Estações de Tratamento de Esgoto Sanitário (ETES) geram um subproduto (lodo de esgoto) que, quando tratado e processado, adquire características que permitem sua utilização agrícola de maneira racional e ambientalmente segura. Este trabalho objetivou avaliar a influência de diferentes doses de lodo de esgoto calcado (50 %) na dispersão de argilas em amostras de solos com diferentes texturas (muito argilosa e média). O estudo foi realizado com amostras de solos coletadas sob mata nativa, em Latossolo Vermelho distroférico, textura muito argilosa, em Londrina (PR), e Latossolo Vermelho-Amarelo distrófico, textura média, em Jaguapitã (PR). Os vasos foram preenchidos com 3 kg de TFSa em casa de vegetação, em um delineamento de blocos ao acaso, com seis tratamentos – T1: controle; e os tratamentos com lodo de esgoto calcado (50%): T2 (3 t ha⁻¹); T3 (6 t ha⁻¹); T4 (12 t ha⁻¹); T5 (24 t ha⁻¹) e T6 (48 t ha⁻¹), com cinco repetições. O tempo de incubação foi de 180 dias; após esse período, os vasos foram abertos e coletaram-se duas subamostras por tratamento, para determinar: pH-H₂O, pH-KCl (1 mol L⁻¹), teor de matéria orgânica, argila dispersa em água, ΔpH (pH-KCl – pH-H₂O) e o PCZ estimado: PCZ = 2 pH-KCl – pH-H₂O, além da mineralogia da fração argila por difração de raios X. Os resultados permitiram concluir que não houve diferença significativa quanto aos teores médios de argila dispersa em água entre o controle e os outros tratamentos estudados para as duas amostras de solo; ΔpH foi a variável que melhor se correlacionou com a argila dispersa em água nos dois solos.

Termos de indexação: biossólidos, floculação, PCZ, ΔpH, matéria orgânica.

INTRODUCTION

Wastewater Treatment Stations produce sewage sludge with an average composition of 99.9 % water and 0.1 % solids. Of the total solids, 70 % are organic (proteins, carbohydrates, lipids, etc.) and 30 % inorganic (sand, salts, metals, etc.). Their disposal is one of the greatest operational problems of wastewater treatment stations (Barbosa & Tavares Filho, 2006).

After treatment and processing, sewage sludge is referred to as “biosolids” and has characteristics that allow a rational and environmentally safe use in agriculture, since it contains some essential plant nutrients (N, P and micronutrients), is rich in organic matter and acts as a soil conditioner, improving the soil structure (aggregation of soil particles) (Barbosa & Tavares Filho, 2006). Agricultural recycling is therefore a more appropriate way of disposing of sewage sludge (biosolids) in technical, economic and environmental terms, given an appropriate application (Tsutiya, 1999). It is the cheapest method of recycling organic matter and nutrients (Matthews, 1998), and conditions and fertilizes the soil. In the United States, for instance, sludge has been used as fertilizer since 1927 (Neiva, 1999) and around 25 % of all biosolids produced (13,106 Mg year⁻¹) is used in agriculture (Tsutiya, 1999) and 41 % in forest soils (USEPA, 1993).

Sewage sludge requires treatment prior to application in agriculture. Since the sludge contains considerable concentrations of bacteria and parasites, it must be sanitized. One way of sanitation is to add

quicklime (in this case, the sludge is called *limed sludge*) (Barbosa & Tavares Filho, 2006).

When using limed sludge, the amounts of organic matter and Ca entering the soil are relatively high, and theoretically, together with other cations that may be present in the soil, can promote soil flocculation and aggregation. However, two important points must be borne in mind: (a) increasing the soil organic matter content can generate excessive negative electrical charges that affect colloid repulsion, raising the level of clay dispersion (Paiva et al., 2000); (b) limed sludge affects the soil pH and the presence of lime in the soil affects some of the electrochemical properties (net negative charge and surface negative electrical potential) of heavily weathered soils (acid soils with a predominantly variable charge), leading to possible changes in the soil physical characteristics.

The surface electrical potential is proportional to the difference between the pH and point of zero charge (PZC), which corresponds to the pH value at which the net variable charge is zero on variable-charge surfaces (Sposito, 1989). Clay tends to flocculate when the pH approaches the point of zero charge (PZC). This point corresponds to the pH value at the intersection point of potentiometric titration curves for soil samples at different electrolyte concentrations. Thus, if the pH is not close to the PZC, the soil will be either acid with predominantly positive charges or alkaline with a higher possibility of negative charges indicating an increase in CTC and a negative electrical potential of the soil surface. This phenomenon is common in more weathered soils and is the result of

chemical adsorption of hydroxyls on oxide and hydroxide surfaces, mainly of Fe and Al, on the broken edges of kaolinite, and of the ionization of functional groups of organic matter, the most important of which are carboxylates (Uehara & Gillman, 1981; Albuquerque et al., 2000).

According to Gupta et al. (1984), the elevation of alkalinity in the presence of humic substances can increase clay dispersion. Although elevating the soil pH increases P availability, promotes microbial activity and reduces or eliminates Al and Mn phytotoxicity, with positive effects on water and nutrient absorption (Ernani et al., 1998), an increased level of clay dispersion in the soil can cause aggregate stability to drop, seal the soil surface, increase surface runoff and reduce water infiltration and soil hydraulic conductivity, affecting soil susceptibility to erosion (Roth & Pavan, 1991; Castro Filho & Logan, 1991; Levy et al., 1993; Fontes et al., 1995; Albuquerque et al., 2000).

Bearing in mind that wastewater treatment units have been providing rural producers with large quantities of sewage sludge at zero cost, and that the organic matter and calcium added by biosolids (limed sludge) improves soil aggregation, the aim of this study was to assess the influence of different doses of limed sludge (50 %) on clay dispersion in soil samples with different textures (very clayey and medium clayey).

MATERIAL AND METHODS

This study was carried out with native forest soil samples from the 0–20 cm layer of a Red Latosol (Brazilian classification: Latossolo Vermelho distroférico) with a very clayey texture (702 g kg⁻¹ clay) (A1) in Londrina (Paraná State, Brazil) (23° 19' 49" S/051° 08' 12" W) and a Red-Yellow Latosol (BC: Latossolo Vermelho-Amarelo distrófico) with medium texture (288 g kg⁻¹ clay) (A2) in Jaguapitã (Paraná State, Brazil) (23° 06' 34" S/051° 31' 58" W). According to the Köppen classification, the climate in this region is Cfa (subtropical humid).

In the laboratory, the samples were ground and passed through a 2 mm sieve (air-dried fine earth). Two sub-samples of the soils studied (A1 and A2) were separated to determine the mineralogy of the clay fraction by X ray diffraction, based on the deposits of a suspension of 10 g L⁻¹ clay (fraction < 2 mm) obtained by soil dispersion using ultrasound and successive washings with water on fine glass blades, using Philips equipment fitted with a copper anticathode, and the diffraction spectra interpreted according to Bragg's law.

In the greenhouse, the experiment was replicated twice with the same statistical scheme of a randomized

block design with six treatments (T1: control; T2: 3 t ha⁻¹ (1.5 g kg⁻¹) of limed sludge (50 %); T3: 6 t ha⁻¹ (3.0 g kg⁻¹); T4: 12 t ha⁻¹ (6.0 g kg⁻¹), T5: 24 t ha⁻¹ (12 g kg⁻¹) and T6: 48 t ha⁻¹ (24 g kg⁻¹) and five replications, for both soil samples (A1 and A2). The sludge to be added to the soil was limed at a proportion of 50 % (sanitized by mixing 50 % dry sludge with 50 % dry quicklime). Pots were lined with plastic and filled with 3.0 kg air-dried fine earth for the treatments. The pots were watered to maximum retention capacity and sealed, leaving an opening for gaseous exchange, for a period of 180 days (incubation time). At the end of this period, the pots were opened and two sub-samples per treatment (A1 and A2) and sample collected to determine: pH-H₂O, pH-KCl (1 mol L⁻¹) (according to Embrapa (1997)), organic matter content according to the *Anne* method (oxidation with potassium dichromate), water-dispersible clay using the pipette method of samples stirred with an orbital agitator at 30 rpm for 3 h, as well as ΔpH (pH-KCl – pH-H₂O, according to Mekaru & Uehara (1972)) and PZC, estimated by the following formula: PZC = 2 pH-KCl (1 mol L⁻¹) – pH-H₂O (according to Keng, *apud* Uehara (1979)).

The dry sewage sludge used in the experiment was provided by the Sanepar Wastewater Treatment Station, Londrina (Paraná State, Brazil) and had the following characteristics: pH 3.1 (CaCl₂); total N 2.3 %; P₂O₅ 1.4 %; K₂O 0.0 %; Ca 2.1 %; Mg 0.6 %; S 2.0 %; OM 37.1 %; C/N 8.7 and residual moisture content of 7.8 %. The lime (calcium oxide) used to disinfect the sludge had the following chemical characteristics: 40.60 % de CaO; 29.48 % de MgO, with Neutralization Power (NP) determined at 122.0 %.

The results of the two experiments were presented as average values for 20 replications per treatment and the coefficient of variation and confidence interval were calculated at 95 %.

RESULTS AND DISCUSSION

The average values (for 20 replications per treatment), coefficient of variation and confidence interval (95 %) for water-dispersible clay in both soil samples studied (A1 and A2) after 180 day incubation in pots in the greenhouse are shown in figure 1.

It can be seen that, for the two soil samples studied (A1 and A2), the values of the coefficient of variation were lowest in T5, with values of 16.8 % for A1 and 6.34 % for A2, indicating an estimated experimental error in relation to the general average (experimental quality assessment) varying, according to Pimentel-Gomes (1990), from average (between 10 and 20 %) for sample A1 to low (less than 10 %) for sample A2. In contrast, the coefficients of variation were highest in T1, with values of 39.6 % for A1 and 30.3 % for A2, indicating a very high estimated experimental error,

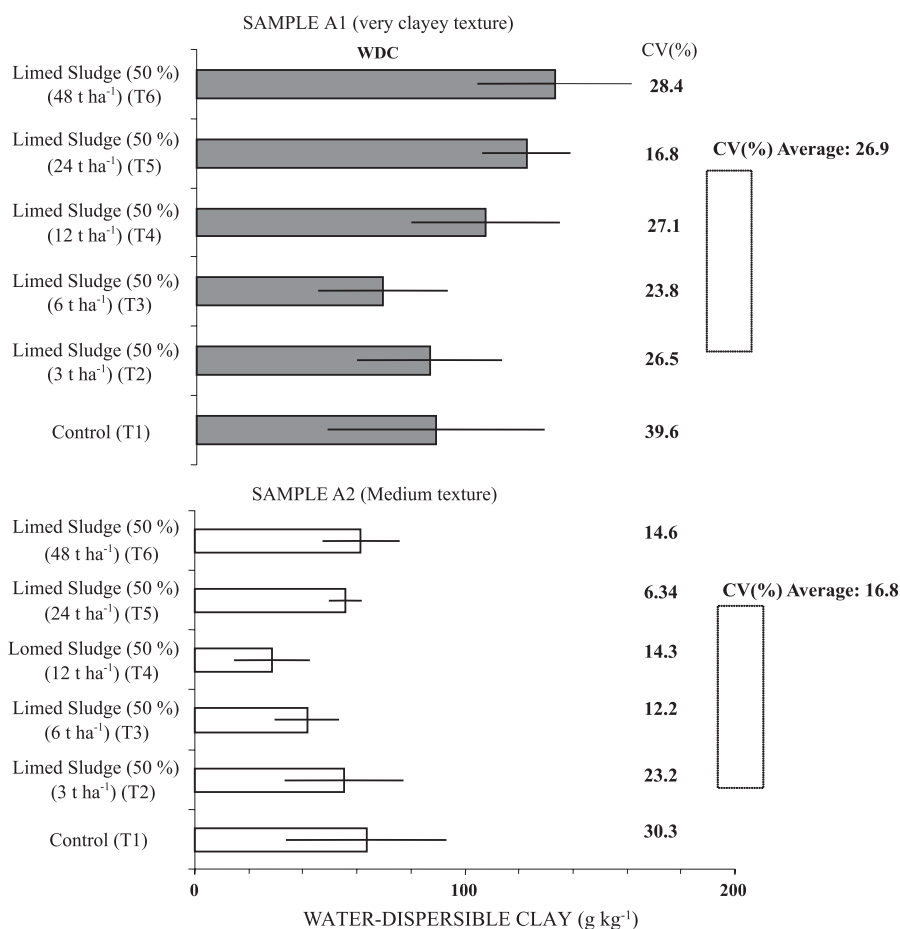


Figure 1. Average values for water-dispersible clay (WDC), coefficient of variation (CV %) and confidence interval, CI (95 %) for very clayey soil (A1) and medium texture soil (A2), after 180 days of incubation in pots kept in a greenhouse.

according to Pimentel-Gomes (1990) (above 30 %) for both soil samples. In terms of averages, the estimated experimental error for A1 is considered high (CV = 26.9 %) and average (CV = 16.8 %) for A2. According to Cargnelutti Filho & Storck (2007), there seems to be a consensus among researchers concerning the use of CV as a measurement of experimental accuracy, where results indicating average, high or very high accuracy are acceptable.

Confidence intervals (Figure 1) were relatively high for both soil samples. In sample A1, there is no significant difference at 5 % between T1 and the other treatments studied. However, between T3 (6 t ha⁻¹ limed sludge) with the lowest average dispersed clay value, and T5 and T6 (highest dispersed clay average values), there is a difference at 5 %. In sample A2, there is also no significant difference at 5 % between T1 and the other treatments studied. However, between T4 (12 t ha⁻¹ limed sludge) with the lowest average dispersed clay value, and T5 and T6 (highest average dispersed clay values), there is a difference at 5 %. These differences between treatments T3 and

T5, T6 (sample A1) and T4 and T5, T6 (sample A2), are probably due to the fact that the soil pH is higher than the PZC when the doses of limed sludge increase significantly, causing greater electrostatic repulsion and a drop in clay flocculation (Mitchell, 1976; Uehara, 1979; Uehara & Gillman, 1980; 1981).

In addition, we expected the application of biosolids to facilitate the aggregation of soil particles, with favorable effects on soil structure and the flocculation process due to the addition of both organic matter and calcium to the soil, since according to Babcock (1963) and Roth & Pavan, (1991) the presence of Ca in the soil and the high ionic strength could promote soil flocculation across cationic bridges and reduce the diffuse double layer. However, this did not occur in treatments T4, T5 and T6 of soil sample A1 and in T2, T5 and T6 of soil sample A2, in which the increased doses of limed sludge also increased clay dispersion in the soil.

The effects of this Ca-organic matter interaction, influencing the reactions in soils, should vary according to soil type and its mineralogical

composition, since, according to Albuquerque et al. (2000), the effect of Ca on soil organic matter content can affect the point of zero charge (PZC), ie the pH, at which the net variable surface charge is zero, since its variables are inversely related.

The sample texture is markedly different and the mineralogical analysis of the clay fraction indicated that kaolinite, total free Fe oxides and gibbsite are predominant in both samples (Table 1). For sample A1, there are also 2:1 clay minerals, probably with hydroxy-Al in the interlayers as demonstrated by Assouline et al (1997). These mineralogy results indicate a predominance of pH-dependent variable charges of both soil samples (A1 and A2).

The values of pH_{KCl} , pH_{H_2O} , ΔpH and PZC (point of zero charge) and organic matter (OM) content for samples (A1) and (A2) after 180 days of incubation in pots in a greenhouse are given in table 2. For both

samples, the use of limed sludge increased the soil pH ($pH-H_2O$) from 5.10 to 5.47 (soil sample A1) and from 5.01 to 5.41 (sample A2), depending on the dose applied. Values for $pH-KCl$ also increased invariably, depending on the dose of limed sludge, and were always lower than $pH-H_2O$ values, resulting in negative ΔpH values (less than zero) for all treatments of both soil samples. This result indicates that there is a net negative charge for all treatments of both soils, even the control, favorable to increased cationic exchange capacity when limed sludge is applied, since (as shown in Table 1) the variable charge was predominant in the soil mineralogy, depending on the pH.

Knowing that the PZC (point of zero charge) indicates the pH at which the net surface charge on variable charge surfaces is zero, causing higher soil flocculation, the results obtained for $PZC_{estimated}$ (Table 2, Figure 2) show that a variation tendency of

Table 1. Texture and mineralogy of the clay fraction (< 2 mm) of samples (A1) and (A2) collected under native forest, in the 0–20 cm horizon

Sample	Texture			Mineralogy
	Clay	Silt	Sand	
	g kg ⁻¹			
A1 - Very clayey texture	702	163	135	Kaolinite, total free iron oxides, gibbsite; trace minerals 2:1
A2 - Medium texture	288	206	506	Kaolinite, total free iron oxides, gibbsite

Table 2. Average values (for 20 replications per treatment) for WDC (water-dispersible clay), $pH-KCl$ (1 mol L⁻¹), $pH-H_2O$, ΔpH and $PZC_{estimated}$ (point of zero charge) OM (organic matter) for samples (A1) and (A2) after 180 days of incubation in pots kept in a greenhouse. Pearson linear correlation coefficient (r) between WDC and other parameters analyzed

Treatment		Parameters analyzed					
		WDC	$pH-KCl$	$pH-H_2O$	ΔpH	ZPC Estimated	OM
		g kg ⁻¹				g kg ⁻¹	
Sample A1 (Very clayey)	T1 (Control)	86.00	4.95	5.10	-0.15	4.80	33.9
	T2 (Limed Sludge (50 %) (3 t ha ⁻¹))	83.75	4.97	5.12	-0.15	4.82	35.0
	T3 (Limed Sludge (50 %) (6 t ha ⁻¹))	66.75	5.06	5.15	-0.09	4.97	37.4
	T4 (Limed Sludge (50 %) (12 t ha ⁻¹))	103.75	5.10	5.27	-0.17	4.93	56.9
	T5 (Limed Sludge (50 %) (24 t ha ⁻¹))	118.50	5.19	5.38	-0.19	5.00	55.5
	T6 (Limed Sludge (50 %) (48 t ha ⁻¹))	128.75	5.25	5.47	-0.22	5.03	60.5
		Correlation – values of r					
Water-dispersible clay (WDC)		1	0.82	0.93	-0.97	0.58	0.89
Sample A2 (Medium)	T1 (Control)	60.50	4.90	5.01	-0.11	4.79	18.3
	T2 (Limed Sludge (50 %) (3 t ha ⁻¹))	55.25	4.97	5.07	-0.10	4.87	13.8
	T3 (Limed Sludge (50 %) (6 t ha ⁻¹))	41.50	5.01	5.10	-0.09	4.92	31.2
	T4 (Limed Sludge (50 %) (12 t ha ⁻¹))	28.75	5.09	5.15	-0.06	5.03	35.8
	T5 (Limed Sludge (50 %) (24 t ha ⁻¹))	55.75	5.17	5.32	-0.15	5.02	0.5
	T6 (Limed Sludge (50 %) (48 t ha ⁻¹))	61.50	5.23	5.41	-0.18	5.05	46.9
		Correlation – values of r					
Water-dispersible clay (WDC)		1	0.03	0.24	-0.80	-0.30	-0.28

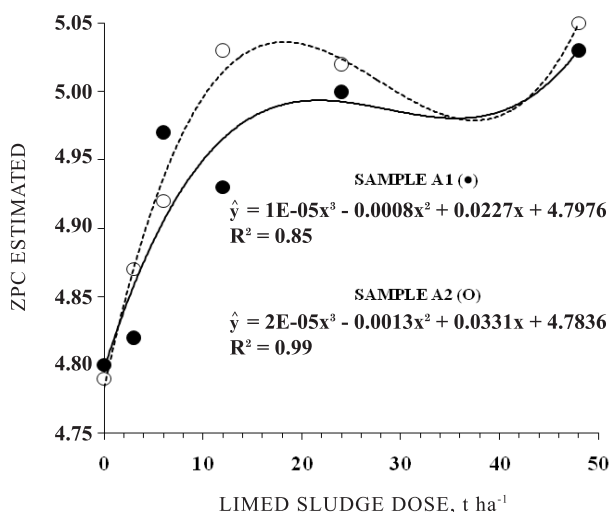


Figure 2. Relationship between the doses of limed sludge (50%) and the estimated point of zero charge ($PZC_{estimated}$) for soil samples A1 (very clayey texture) and A2 (medium texture), used in this study.

third polynomial order, with high determination coefficients for both soil samples studied ($R^2 = 0.85$ for A1; $R^2 = 0.99$ for A2), indicating that the variation in the doses of limed sludge significantly affected the estimated PZC, so that for the model used, the “doses of limed sludge” variable explained 85% of the variance in $PZC_{estimated}$ for sample A1, and 99% of $PZC_{estimated}$ variance for sample A2. Values of $PZC_{estimated}$ increased almost linearly between 0 and 12 $t\ ha^{-1}$ limed sludge, dropped between doses of 12 and 38 $t\ ha^{-1}$, and increased again as from 38 $t\ ha^{-1}$ until the highest value was reached for T5 (48 $t\ ha^{-1}$ limed sludge) (Figure 2).

For sample A1, the soil flocculation value (Figures 1, 2 and Table 2) was highest in treatment T3, with ΔpH at -0.09 and $PZC_{estimated}$ at 4.97 and for sample A2 in treatment T4, with ΔpH at -0.06 and $PZC_{estimated}$ at 5.03. In all cases, the value of $PZC_{estimated}$ is higher than the value for kaolinite (4.6) (Sposito, 1989), and lower than the values for goethite (6.4) and gibbsite (5.1) (Varadachari & Chattopadhyay, 1997), which are the predominant clay minerals in these Latosols (Table 1).

Note also (Table 2, Figure 3) that the correlation between WDC and ΔpH for sample A1 is high in terms of the Pearson linear correlation coefficient ($R^2 = 0.94$) and for sample A2 ($R^2 = 0.64$), indicating that the ΔpH is the variable that most influences the dispersion of both soils studied. Therefore, when ΔpH tends towards zero, charges are balanced, with less dispersion. Dispersion is directly proportional to the excess number of negative charges. Therefore, the surface electrical potential (proportional to the difference between pH and PZC) corresponds to the

pH value at which the net variable charge is zero on variable charge surfaces (Sposito, 1989). In this case, the clay tends to flocculate when the pH approaches the point of zero charge (PZC). In other words, the use of limed sludge (50%) at doses of 6 $t\ ha^{-1}$ (T3/sample A1) and 12 $t\ ha^{-1}$ (T4/sample A2) in these oxidic weathered soils helps reduce the surface net charge (Table 2: $\Delta pH = -0.09$ for T3/sample A1 and $\Delta pH = -0.06$ for T4/sample A2) and, due to the drop in repulsion between the double electrical layers of clay particles, they interact freely, attracting each other through van der Waals forces and rapidly flocculating according to Raji & Peech (1972).

In addition, there was no clay flocculation in treatments T2, T4, T5, and T5 (sample A1) and T2, T5 and T6 (sample A2). According to Mitchell (1976) and Uehara (1979), dispersion occurs when the soil pH (in our study, this is pH- H_2O) is higher than the PZC. In this case, there is greater electrostatic repulsion, reducing clay flocculation (Uehara, 1979; Uehara & Gillman, 1980, 1981). Therefore, in these weathered oxidic soils, doses of limed sludge (50%) higher than 6 to 12 $t\ ha^{-1}$ caused the Ca concentration in the soil to increase, which could have displaced exchangeable aluminum to the soil solution, and lower valency cations occupied the exchange complex, causing the diffuse double layer to expand, increasing the distance between colloids. Thus, since the attraction between colloids is based on Van der Waals-London forces, the increase in the distance between colloids prevents these forces from acting efficiently, causing the clays to disperse (Castro Filho & Logan, 1991; Albuquerque et al., 2000).

Bearing in mind that, according to Siqueira et al. (1990), soil organic matter (OM) can promote chemical adsorption of organic and inorganic compounds on its surfaces since it is one of the main sources of negative

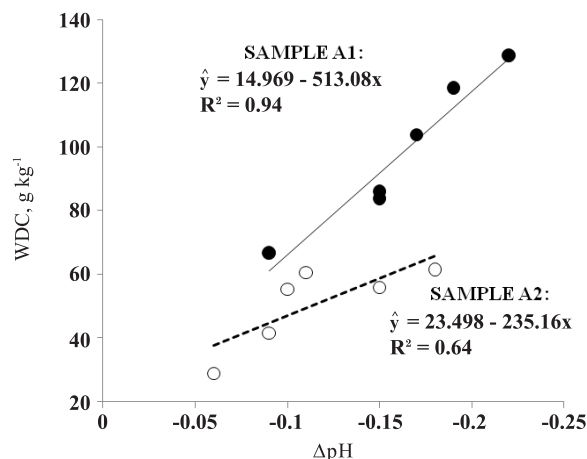


Figure 3. Correlation between water-dispersible clay (WDC) and ΔpH in soil samples A1 (very clayey texture) and A2 (medium texture), after 180 days of incubation in pots kept in a greenhouse.

charge in weathered soils with 1:1 mineralogy (Raij & Peech, 1972), table 2 and figure 4 show that there is a second-order polynomial tendency variation between the doses of limed sludge and organic matter content, with high determination coefficients for both samples studied ($R^2 = 0.87$ for both A1 and A2), indicating that varying doses of limed sludge affected OM contents. The variable limed sludge dose explains 87 % of OM content variance in the soil for both soils studied.

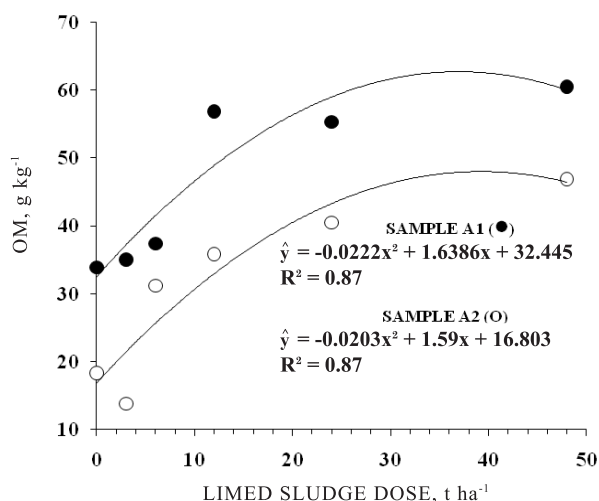


Figure 4. Organic matter (OM) content as related to the dose of limed sludge (50 %) applied to soil samples A1 (very clayey texture) and A2 (medium texture) in this study.

In addition, according to Paiva et al. (2000), a significant increase in soil organic matter can generate excess negative charges that affect colloid repulsion, increasing clay dispersion. According to Gupta et al. (1984), raising soil alkalinity (limed sludge affects soil pH) in the presence of humic substances can increase clay dispersion. According to Siqueira et al. (1990), OM can promote chemical adsorption of organic and inorganic compounds. The reason is, according to Raij & Peech (1972), that OM is one of the main sources of negative charges in weathered soils with 1:1 mineralogy and, according to Oades (1984) and Levy et al. (1993), organic anion action is produced by OM decomposition and root exudates in the dispersion of kaolinite, the normally predominant clay mineral in Latosols (Table 1) at the sample site of A1 and A2.

CONCLUSIONS

1. There was no significant difference between the average content values of water-dispersible clay (WDC)

in the control and other treatments of both soil samples studied.

2. Δ pH was the variable that correlated best with WDC in both soils.

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