Aggregate breakdown and dispersion of soil samples amended with sugarcane vinasse

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ABSTRACT: Soil aggregation is a very complex issue related to important soil attributes and processes. The aggregate breakdown and dispersion of soil samples amended with sugarcane vinasse were evaluated using ultrasonic energy. Vinasse is an important byproduct of sugarcane industries, intensively applied to soils in Brazil as liquid fertilizer. Samples of two Oxisols and one Ultisol were used in this study. The physical and chemical characterization of soils was performed, and the 1 to 2 mm size aggregates (200 g) were packed in PVC columns (6.0 cm high and 4.0 cm internal diameter) and incubated with sugarcane vinasse under lab conditions for 1, 30 and 60 days. After incubation, aggregates were submitted to levels of ultrasonic energy, and the particle size distribution (53 to 2,000 µm, 2 to 53 µm, and < 2 µm fractions) was quantified. Mathematical equations were used to relate the mass of aggregates in each of these fractions to the applied ultrasonic energy, and parameters related to aggregate stability were then obtained. Soils showed an aggregate-hierarchy resulting in a stepwise breakdown under ultrasonic agitation. Considering this soil-aggregation hierarchy, vinasse contributed even in a short time to the bonding between and within 2 to 53 µm aggregates, mainly in the Oxisols. This may be related to organic compounds present in the vinasse, cementing soil particles. Potassium enrichment of soil samples did not contribute to soil dispersion.

Keywords: soil-aggregate hierarchy, soil structure, aggregate stability, dispersive energy

Introduction

Soil aggregate stability has been used to indicate soil resistance to erosive agents and soil quality (Nichols and Toro, 2011). Aggregate breakdown and soil dispersion, caused by raindrop impact and equipment used for soil tillage, may affect soil porosity, decreasing water infiltration and hydraulic conductibility and increasing surface sealing and susceptibility for erosion (Raine and So, 1993; Fuller et al., 1995). In addition, soil aggregates physically protect organic matter (Gregorich et al., 1989; Feller and Beare, 1997), which is important for carbon sequestration.

The use of ultrasonic energy to measure soil aggregate stability was proposed by North (1976), in cases where the amount of dispersive energy responsible for the aggregate breakdown is measurable. The results are normally expressed through soil dispersion curves, plotting, for example, the < 2 µm fraction released (dispersed) from aggregates against the respective levels of ultrasonic energy (Raine and So, 1993, 1994; Ribeiro et al., 2009). Ultrasonic energy irradiation on soil aggregate suspension allows an overview of the aggregate breakdown and dispersion process. Considering an aggregate hierarchy in soils (Perfect and Kay, 1991), a stepwise breakdown of different size aggregates during ultrasonic irradiation can be observed and modeled (Field and Minasny, 1999; Field et al., 2006; Zhu et al., 2009; Zhu et al., 2010).

Vinasse is an important byproduct from ethanol and sugarcane industries. In the past, Brazilian mills dumped the vinasse into the water courses, causing serious environmental impacts (Günkel et al., 2007). Nowadays, it has been intensively applied as liquid fertilizer to crops, mainly sugarcane crop. Thus, this work aimed to evaluate the aggregate breakdown and dispersion by using ultrasonic energy of soil samples that were amended with vinasse. It was hypothesized that the vinasse affects soil aggregation in two ways: i) due to the presence of monovalent cations (e.g., K^+) the vinasse disperses soil particles; ii) organic compounds contribute to the binding of soil particles and also to increase the microbial activity, which affects soil aggregation.

Materials and Methods

Topsoil samples (0 to 0.1 m layer) from an Acrudox and Hapludox from Lavras, state of Minas Gerais, Brazil (21º13′30″ – 21º14′60″ S; 44º58′30″ - 44º59′60″ W), and an Hapludult (21º09′03″ S; 45º00′29″ W), under native vegetation, were used in this study. Samples were air dried, carefully ground, and sieved in order to obtain 1 to 2 mm size aggregates. Chemical and physical characterization was performed according to Embrapa (1997). Selected attributes of soil samples are shown in Table 1. Soil texture (clay, silt and sand contents) was obtained by the pipette method. Soil organic matter was determined by the modified Walkley-Black method (Yeomans and Bremner, 1988). Contents of oxides were obtained after digestion with 9.4 M H_2SO_4 (Embrapa, 1997).

Aggregates with 1 to 2 mm were packed in PVC columns (12.0 cm high and 4.0 cm internal diameter), containing 200 g of aggregates (density 1.00 ± 0.04 g cm^{-3} and total porosity 0.59 m^3 m^{-3}), and incubated

with sugarcane vinasse. The vinasse (from sugarcane spirit-brandy production) was applied at the following equivalent rates: 0 (control), 150 and 300 m^3 ha^{-1}; and then, the samples were kept at field capacity (Acrudox: 0.32 m^3 m^{-3}; Hapludox: 0.30 m^3 m^{-3}; Hapludult: 0.27 m^3 m^{-3}) during 1, 30 and 60 days. For each soil, the treatments followed a 3 × 3 factorial scheme (three vinasse doses and three incubation times), in a randomized design with three replicates. The main properties of the vinasse were: pH (3.5); electrical conductivity (7.5 dS m^{-1}); organic carbon content (9.6 g L^{-1}); potassium (2.1 g L^{-1}); sodium (0.74 mg L^{-1}); and water content (98.5 %).

After each incubation time, the aggregates were carefully removed from the columns and air-dried for 48 h; a sub-sample was used for chemical characterization. For measuring aggregate disruption, 5 g of aggregates (oven-dried basis) were placed into a 250-mL beaker and pre-moistened by slow dropping of distilled water along the walls of the beaker (approximately 30° inclination) using a burette. After all the aggregates were immersed in water, the volume was completed to 200 mL (soil: water ratio 1:40).

The soil suspension was submitted to levels of ultrasonic energy: 0; 210; 420; 840; 1,680; 3,360; 6,720; and 13,440 J g^{-1}, based on calorimetric techniques described by Raine and So (1993, 1994). The equipment used was a probe-type Misonix, XL 2020 model, with an output power of 70 W, with a standard solid 19.1mm

Table 1 – Some physical and chemical properties of the 1 to 2 mm aggregates from the 0 to 0.1 m soil layer of the Acrudox, Hapludox and Hapludult.

Attribute	Acrudox	Hapludox	Hapludult
Clay (g kg^{-1})	594	481	390
Silt (g kg^{-1})	222	157	233
Sand (g kg^{-1})	184	362	377
pH	6.0	5.8	5.9
K (mg dm^{-3})	73.0	28.0	33.0
Ca ($mmol_c$ dm^{-3})	20	28	21
Mg ($mmol_c$ dm^{-3})	5	4	6
Al ($mmol_c$ dm^{-3})	2	2	2
H+Al ($mmol_c$ dm^{-3})	39	48	29
SB ($mmol_c$ dm^{-3})	27	33	28
$CEC^{1/}$ ($mmol_c$ dm^{-3})	29	35	30
$CEC^{2/}$ ($mmol_c$ dm^{-3})	66	81	57
V (%)	41.0	41.0	49.1
m (%)	7.0	5.7	7.0
SOM (g kg^{-1})	30.0	31.0	22.0
$SiO_2^{3/}$(g kg^{-1})	167	135	177
$Al_2O_3^{3/}$(g kg^{-1})	257	91	194
$Fe_2O_3^{3/}$(g kg^{-1})	219	90	71
Al_2O_3/Fe_2O_3	1.84	1.59	4.29
Ki	1.11	2.53	1.54
Kr	0.72	1.55	1.25

pH in water (relation soil:water 1:2.5); SB: sum of bases; $CEC^{1/}$: cation exchange capacity at field pH; $CEC^{2/}$: cation exchange capacity at pH 7.0; V: Base saturation; m: Al saturation; SOM: soil organic matter; $^{3/}$Indexes of total oxides after digestion with 9.4 M H_2SO_4; Ki and Kr: respectively molar ratio (SiO_2/Al_2O_3) and (SiO_2/Al_2O_3 + Fe_2O_3).

probe immersed 2.5 cm into the soil suspension.

After each level of applied ultrasonic energy, the 53 to 2,000 µm fraction was gently separated by wet-sieving. The aggregates and the soil suspension that passed through the sieve (53 µm) were transferred to 500-mL measuring cylinders. After adequate settling-times, the < 2 µm fraction was determined using the pipette method. The 2 to 53 µm fraction was determined by difference (1 – 53 to 2,000 µm plus < 2 µm fraction). Amounts of 53 to 2,000, 2 to 53 and < 2 µm fractions were adjusted to the equations proposed by Field and Minasny (1999) (Equations 1 to 3), and the energy to complete dispersion and the one required for aggregate releasing and dispersion of the 2-53 µm fraction ($E_{crit.}$) (Equation 4) were calculated.

The soil-dispersion characteristic curve (SDCC) can be represented by the equation:

$$SDCC = a[1 - exp(-b . x)] \qquad (1)$$

where SDCC is the clay fraction (< 2 µm) dispersed using a given ultrasonic energy level (x); a and b are the fitting parameters; b represents a dispersion constant (k_2) (g J^{-1}), according to Field and Minasny (1999) and Field et al. (2006).

SDCC represents the amount of clay dispersible at different ultrasonic energy levels as consequence of aggregate breakdown and dispersion of aggregates. As the ultrasonic energy increases the clay increases reaching a plateau. The energy to complete dispersion was assessed based on the SDCC plateau.

The aggregate-disruption characteristic curve (ADCC) can be represented by the equation:

$$ADCC = y_0 + a . exp(-c . x) \qquad (2)$$

where ADCC represents the 53 to 2,000 µm aggregate size or sand fraction in a given ultrasonic energy level (x); y_0, a and b are fitting parameters; c is referenced as a disaggregation constant (k_1) (g J^{-1}).

The ADCC represents the amount of 53 to 2,000 µm aggregate size or sand fraction remaining after each applied ultrasonic energy level. As the ultrasonic energy level increases this fraction decreases reaching a plateau, limited by the sand fraction content.

Considering that in an applied ultrasonic energy level, the sum of masses of < 2, 2 to 53 and 53 to 2,000 µm fractions is equal to 1, the releasing of aggregate and dispersion curve (RADC) can be described as:

$$RADC = 1 - [y_0 + a . exp(-c . x) + a . (1 - exp(-b . x)] \qquad (3)$$

where RADC is the 2 to 53 µm aggregate size or silt-size fraction in a given ultrasonic energy level (x); y_0, a and b are fitting parameters; c and b are, respectively, the disaggregation constant (k_1) and dispersion constant (k_2).

The RADC contains two parts; the first part is characterized by the increasing of the 2 to 53 µm aggregate

size as consequence of aggregate breakdown; the second part represents the dispersion of the 2 to 53 µm aggregate size. From RADC a stepwise breakdown of 2 to 53 µm aggregate size is obtained. So, the energy required to complete the total liberation of 2 to 53 µm aggregates size (E_{crit}) can be calculated by the equation:

$$E_{crit} = \frac{\ln\left(k_2 / k_1\right)}{k_2 - k_1} \qquad (4)$$

The data of the models were fitted using the Sigma Plot software version 10.0. The significance of the models was evaluated by the F-Test ($p > 0.05$) and means were compared considering the standard deviation.

Pearson's correlation between E_{crit}, k_1 and k_2 constants and some soil attributes related to soil aggregation were performed.

Results and Discussion

Effect of vinasse on chemical attributes

The main effect of vinasse on chemical attributes of soil samples was the increase in the potassium content (Table 2). For the Acrudox sample, CEC saturation by potassium was 14 % and 19 %, respectively for 150 and 300 m³ ha⁻¹ doses. For the Hapludox, these values were 12 % and 16 %, and for the Hapludult sample they were 13 % and 19 %. These results are above the ideal saturation of potassium on CEC for agricultural purposes (up to 5 %). In these soils, having low Ca and very low Mg contents (Table 1) under natural conditions, the maximum amount of vinasse to be applied should not be over 22, 36 and 38 m³ ha⁻¹ respectively for Acrudox, Hapludox and Hapludult. If these soils receive liming and/or fertilization, these values should be recalculated.

The Technology and Environmental Sanitary Company of the São Paulo State (CETESB, 2006), Brazil, takes into account these criteria for establishing the rates of vinasse to be applied on soils. High saturation of K in the CEC may lead to soil dispersion (Igwe and Okebalama, 2006) and, consequently, to soil erosion and land degradation (Auerswald et al., 1996). In soils amended with beet vinasse a degradation of soil structure and increase on erosion were observed due to the enrichment of the

CEC by monovalent cations, such as K (Tejada and Gonzalez, 2006; Tejada et al., 2007).

Previous results obtained for the soils used in this work showed that the vinasse increased pH (Ribeiro et al., 2012). This may contribute to increase microbial activity and, consequently, soil aggregation. The ΔpH (pH in 1M KCl – pH in water) and the zeta potential of the samples amended with vinasse were near zero (data not shown), which favor clay fraction flocculation (Lima et al., 2000).

Aggregate breakdown and dispersion

Figure 1 shows the SDCC (A), represented by clay size fraction (< 2 µm), the ADCC (B), represented by sand size fraction (53 to 2,000 µm), and the RADC(C), represented by silt size fraction (2 to 53 µm) for samples of the three soils, adjusted to models proposed by Field and Minasny (1999) and Field et al. (2006).

As the < 2 µm fraction increased (Figure 1A), the 53 to 2,000 µm fraction decreased (Figure 1B), reaching respectively the clay and sand contents of the soils. Based on SDCC, the Hapludult needed less ultrasonic energy to complete dispersion (1,732 J g⁻¹), followed by Acrudox (4,357 J g⁻¹) and Hapludox (13,440 J g⁻¹). These differences are closely related to the factors that affect the soil aggregation, such as soil texture, organic matter and oxide content (Six et al., 2004). The Hapludult has less clay content and organic matter (Table 1). These observations do not allow distinguishing the higher resistance of Hapludox aggregates compared to Acrudox aggregates. It appears related to the intensive use of Acrudox area (experimental area – plowing and harrowing over time), which may contribute to higher aggregate disruption, while in the Hapludox area the aggregates were collected from a well-established pasture (*Brachiaria* sp.).

The shape of the RADC curve (Figure 1C) indicates soil-aggregate hierarchy. A stepwise breakdown of the 2 to 53 µm aggregates was observed as a consequence of bonding between and within the silt-sized aggregates (Field and Minasny, 1999; Field et al., 2006). First, the 2 to 53 µm fraction increased reaching a maximum value, indicating that the releasing of aggregates is

Table 2 – Potassium contents of the Acrudox, Hapludox and Hapludult, influenced by vinasse rates (values in parentheses refer to the % K saturation on CEC).

Acrudox			Hapludox			Hapludult		
				Vinasse rates				
V0	V1	V2	V0	V1	V2	V0	V1	V2
				1 day incubation time				
				Potassium (mg dm⁻³)				
73 (2.8)	387 (13.1)	499 (15.4)	28 (0.9)	331 (10.1)	487 (14.2)	33 (1.5)	293 (12.3)	487 (18.6)
				30 days incubation time				
73 (2.8)	374 (13.9)	512 (20.5)	28 (0.9)	318 (12.2)	474 (17.4)	33 (1.5)	312 (12.7)	456 (19.8)
				60 days incubation time				
73 (2.8)	368 (14.1)	505 (19.9)	28 (0.9)	318 (12.5)	443 (17.8)	33 (1.5)	275 (12.8)	456 (19.8)

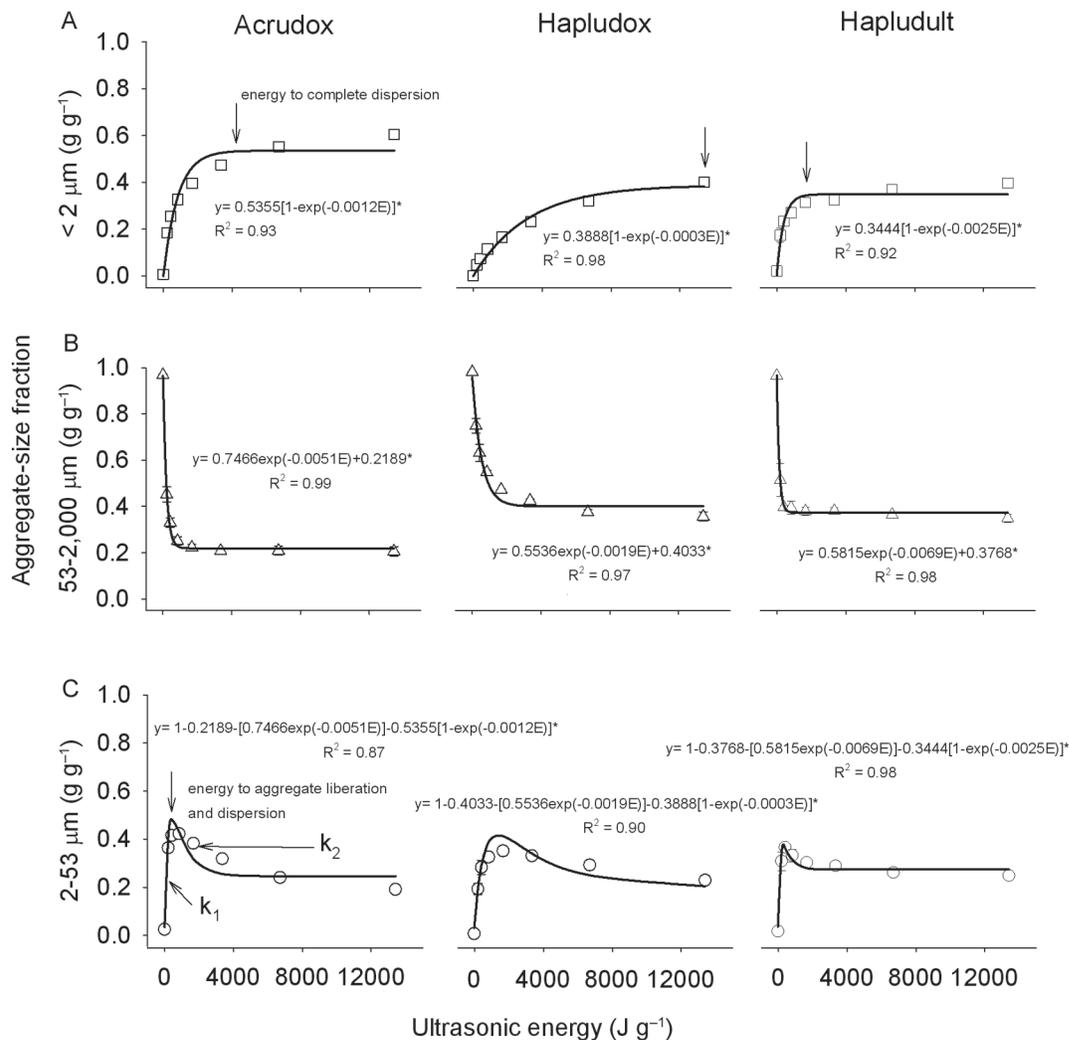V0 – control; V1 = 150 m³ ha⁻¹; V2 = 300 m³ ha⁻¹.

Figure 1 – Soil dispersion characteristic curve – SDCC (A), aggregate disruption characteristic curve – ADCC (B) and releasing of aggregate and dispersion curve – RADC (C) of 1-2 mm aggregates from the Acrudox, Hapludox and Hapludult. Error bars indicate the standard deviation (n = 3). *$p > 0.05$.

greater than dispersion. This is represented by the disaggregation constant (k_1). Lower k_1 values indicate more resistance of aggregates. Taking into account the hierarchy of soil aggregation (Perfect and Kay, 1991), the 2 to 53 µm aggregates were released from larger aggregates as the 1 to 2 mm aggregates used in this work. The k_1 is a measure of bonding among the 2 to 53 µm aggregates. The Hapludult showed the highest k_1 value, followed by Hapludox and Acrudox (Figure 2).

With continuing energy input, the 2 to 53 µm aggregates decreased indicating that the dispersion is greater than releasing of aggregates (Figure 1C). Now, this is represented by the dispersion constant (k_2). Lower k_2 values indicate that the 2 to 53 µm aggregates are more resistant to dispersion and represent the bonding of discrete particles (silt and clay) within the aggregates.

Again, the aggregates from Hapludox were more resistant than Acrudox and Hapludult (Figure 1). Additionally, the energy required to complete aggregate releasing and dispersion of 2-53 µm aggregates released ($E_{crit.}$) decreased as follows: Hapludox > Acrudox > Hapludult (Figure 2).

The k_1 (aggregate releasing) was much greater than k_2 (aggregate dispersion) for all soils (Figure 2). In highly weathered tropical soils (oxide-rich), very stable microaggregates are quite common. The oxides (mainly Al oxides) have been considered the dominant binding agent in weathered soils (oxide-rich), mainly at the microaggregate hierarchy level (Six et al., 2004). The role of Al oxides as binding agent of discrete soil particles was shown by Reichert and Norton (1994) and Itami and Kyuma (1995). The mechanisms responsible for high mi-
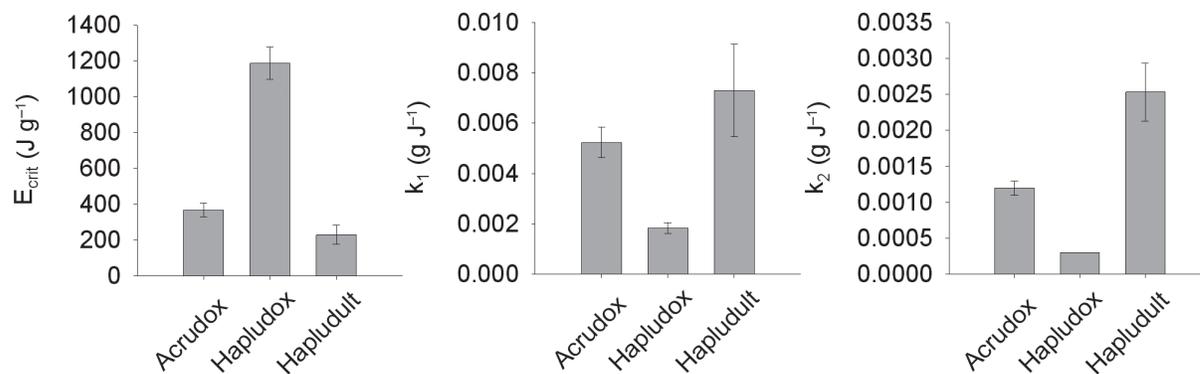
Figure 2 – Energy for aggregate release and dispersion ($E_{crit.}$), disaggregation constant (k_1) and dispersion constant (k_2) of 2 to 53 µm size aggregates released from 1 to 2 mm aggregates of the Acrudox, Hapludox and Hapludult. Error bars indicate the standard deviation (n = 3).

croaggregate stability are related to (El-Swaify and Emerson, 1975): i) organic compounds adsorbed on oxides surfaces; ii) interaction between the positively charged oxides and negatively charged clay minerals and organic matter.

This study showed soil aggregation hierarchy for all three soils. However, this cannot be taken as true for all the soils since, in some cases, macro-aggregates can breakdown into discrete particles, as observed for some Oxisols (Oades and Waters, 1991; Zhu et al., 2010). The hierarchical system of aggregation seems to occur where the organic matter is the main stabilizing agent between soil aggregates (Oades and Waters, 1991; Zhu et al., 2010). When the oxides are the main stabilizing agent between soil aggregates a non-hierarchical aggregation may occur (Zhu et al., 2010).

Table 3 shows the correlation between $E_{crit.}$, k_1 and k_2 constants and some soil attributes related to aggregation. Positive correlations for $E_{crit.}$, and negative for k_1 and k_2 with these attributes indicate more stable aggregates. This was observed for Ca and organic matter contents. Calcium plays an important role in clay flocculation (Kjellander et al., 1988; Six et al., 2004) – the first step for soil aggregation. Calcium is considered an important element for the stabilization of soil organic matter and aggregation due to formation of clay-polyvalent cation-organic matter complexes (Clough and Skjemstad, 2000). Soil organic matter has been the main attribute related to aggregate resistance, when submitted to ultrasonic irradiation.

A high correlation was observed between $E_{crit.}$, k_1, k_2 and SiO_2 content. In this case, the correlation values indicate a weak aggregation. Researchers have demonstrated the relationships between increasing amounts of silicates (mainly kaolinite) and poorly aggregated soils (Barthès et al., 2008); on the other hand, the Al- and Fe-oxides contribute to increasing soil aggregation and stability. However, in this work, the Al_2O_3 content and the Al_2O_3/Fe_2O_3 ratio had a negative correlation with $E_{crit.}$, and a positive correlation with k_1 and k_2. Therefore, the

Table 3 – Pearson's correlation between $E_{crit.}$, k_1 and k_2 constants and some soil attributes related to soil aggregation.

	$E_{crit.}$	k_1	k_2
SiO_2	-0.99***	0.91***	0.89***
Al_2O_3	-0.86**	0.67*	0.51 ns
Fe_2O_3	-0.27 ns	0.02 ns	-0.22 ns
Al_2O_3/Fe_2O_3	-0.67*	0.78**	0.93***
Ki	0.90**	-0.72*	-0.58 ns
Kr	0.68*	-0.45 ns	-0.25 ns
Ca^{2+}	0.77**	-0.69*	-0.62*
Soil organic matter	0.90***	-0.84**	-0.87**

*($p > 0.05$), ** ($p > 0.01$) and ***($p > 0.001$). ns: not significant.

indexes of the total oxide contents themselves seem do not explain soil aggregation, because they do not represent the mineralogy of the soil (Resende et al., 2011).

The vinasse affected the k_1 and k_2 constants and the $E_{crit.}$ (Figure 3). In the case of Acrudox and Hapludox, at 1-day incubation time, the dose of 300 m³ ha⁻¹ increased the $E_{crit.}$ and reduced the k_1 and k_2. At 30 and 60 days no differences between the 150 and 300 m³ ha⁻¹ doses were observed. In the Hapludult, the effect of vinasse was observed only at 60 days. From these results the Oxisols seem interact better with vinasse than the Ultisol, resulting in a higher resistance to aggregate disruption and dispersion by ultrasonic energy. Benke et al. (1999) found a higher interaction of dissolved organic compounds from vinasse with iron oxides.

The effect of vinasse on soil aggregation could be related to organic compounds that are incorporated into the samples, acting in the following ways: i) organic compounds protect the aggregates against the cavitation produced by ultrasonic irradiation in the soil-water suspension (Hinds and Lowe, 1980); ii) increasing the microbial activity, resulting in the production of polysaccharides, mucilage and fungal hyphae, which play an important role in soil aggregation (Oades, 1984).

The effects of vinasse may be temporary because the organic compounds of vinasse are highly decom-
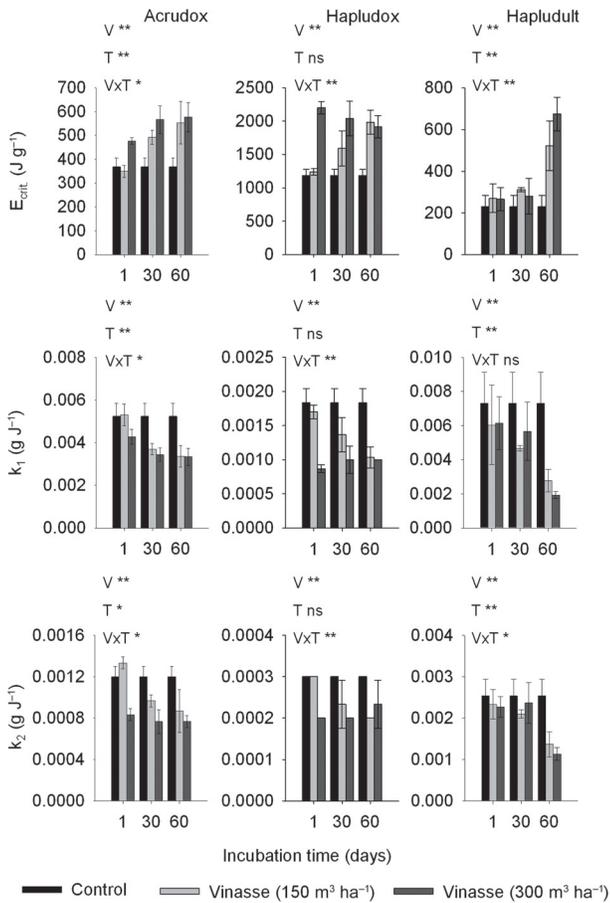
Figure 3 – Effect of sugarcane vinasse and incubation time on E_{crit}, k_1 and k_2 of 2-53 μm aggregates released from 1-2 mm aggregates of the Acrudox, Hapludox and Hapludult. Error bars indicate the standard deviation (n = 3). *, ** respectively $p > 0.05$ and 0.01 (F Test). V: vinasse; T: incubation time; VxT: interaction vinasse and incubation time.

pounds from vinasse cementing the microaggregates and also favoring the flocculation of clay fraction.

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posable (Doelsch et al., 2009). The long term effect of vinasse on aggregation would be related to periodical application on the soil, gradually improving the quantity and quality of organic matter, as observed by Canellas et al. (2003) for an Inceptisol amended with vinasse for a long time (35 years). On the other hand, for an Oxisol no effect of sugarcane vinasse was observed on soil aggregation after one year of application (Passarin et al., 2007). This may be interpreted in two ways: the organic compounds from vinasse (bonding agent) were totally decomposed or the methodology used was not sensitive to detect differences.

A possible initial dispersion caused by the enrichment of monovalent cations such as K from vinasse was not observed. On the other hand, the methodology used was sensitive to show the short term effect (up to 60 days) of vinasse as a bonding agent between and within aggregates. This effect may be related to organic com-

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