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FERTILITY AND MINERALOGY OF SOILS SUBJECT TO VINASSE APPLICATION ON A FARM SITUATED IN SANTA CRUZ DAS PALMEIRAS-SP, BRAZIL

FERTILIDADE E MINERALOGIA DO SOLO SUJEITO À DISPOSIÇÃO DE VINHAÇA DE UMA FAZENDA EM SANTA CRUZ DAS PALMEIRAS-SP, BRASIL

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ABSTRACT - With the ethanol production expansion in Brazil, there was an increase in the production of vinasse, an effluent from sugar and alcohol industry, which under certain conditions can be used for fertirrigation of sugarcane crops. The research aimed to evaluate the effect of vinasse applied in the period from 2012 to 2017, considering the chemical and mineralogical properties of the soil of a farm located in Santa Cruz das Palmeiras-SP, Brazil. From the results obtained, it was possible to observe that in 2014, which was the year of highest calculation of vinasse dosage, low fertility conditions occurred with high concentrations of H+Al and total CEC. The temporal analysis of one of the farm fields exposed that, in controlled doses, the application of vinasse can improve soil fertility and promote the reduction of acidity and aluminum toxicity. The granulometric and mineralogical tests characterize the studied soil as a sandy soil with high concentrations of silicates and oxides of iron and aluminum. The research evaluated the possibility of leaching of base cations and concentration of H+Al in rainy periods, while moisture and macronutrients would be replaced with fertirrigation in dry periods.
Keywords: Agricultural Engineering. Environment. Pedology.

RESUMO - Com a crescente produção de etanol no Brasil, houve o aumento da geração da vinhaça, efluente da indústria sucroalcooleira, que sob certas condições pode ser utilizada para a fertirrigação de lavouras de cana-de-açúcar. O objetivo do estudo foi avaliar o efeito da vinhaça aplicada no período de 2012 a 2017, considerando as propriedades químicas e mineralógicas do solo de uma fazenda situada em Santa Cruz das Palmeiras. A partir dos resultados obtidos, foi possível observar que no ano de 2014 de maior cálculo de dosagem de vinhaça ocorreram baixas condições de fertilidade com altas concentrações de H+Al e de CTC total. A análise temporal de uma das glebas da fazenda demonstrou que, em doses controladas, a aplicação de vinhaça pode melhorar a fertilidade do solo e promover a redução da acidez e da toxicidade por alumínio. Os ensaios granulométricos e mineralógicos caracterizaram o solo estudado como um solo arenoso com altas concentrações de silicatos e óxidos de ferro e alumínio. O estudo avaliou a possibilidade de lixiviação dos cátions de base e concentração de H+Al em períodos chuvosos, ao passo que haveria reposição de umidade e macronutrientes com a fertirrigação em períodos secos.
Palavras-chave: Engenharia Agrícola. Meio Ambiente. Pedologia.

INTRODUCTION

Since the implementation of the National Alcohol Program (or PROALCOOL program), Brazil has developed a great expansion of sugarcane cultivation for the sugar and alcohol industry, which has consequently resulted in increased vinasse production as the main residue of the ethanol manufacturing process (Mortatti, 2010; Casarini, 1989).

The vinasse is used mainly in the fertirrigation of sugarcane crops, since this effluent has high concentrations of organic matter and macro and macronutrients, through expressive concentrations of Potassium (K^+), Calcium (Ca^{2+}) and Magnesium (Mg^{2+}) with lower amounts of Nitrogen (N) and Phosphorous (P), which can increase soil fertility over time. On the other hand, depending on the way vinasse is poured or dosed, it may present a risk of contamination of soil, groundwater and surface water, with possible risk of eutrophication of hydraulic resources, salinization and acidity increase (Fuess, 2013; Oliveira, 2015).

Considering the possible environmental risk deriving from the application of vinasse on soils, norms and laws that aim at the appropriate management of this waste have been established, for example the Technical Standard P4.231 – *Vinasse: Criteria and procedures for application to agricultural soil* (CETESB, 2015), which defines criteria and procedures about storage, transport and application of vinasse, besides of annual elaboration of "Vinasse Application Plans" (VAP) for monitoring and inspection purposes through soil vinasse sampling.

In relation to the effects of the application of residues such as vinasse or sludge from Sewage Treatment Stations (STS) on soil fertility of sugarcane crops, researches such as the ones performed by Pereira (2015), Lopes (1984), Nascimento et al. (2004) and Barros et al. (2010) analyzed the increase of the organic matter (OM) concentration, pH reduction at the moment of the effluent application with posterior original values return after determined time, increase of the Cation Exchange Capacity (CEC), expressive addition of cations such as K^+, Ca^{2+} e Mg^{2+} and the consequently increase of the sum of bases (SB) and the base saturation (V%), besides of the reduction of the changeable aluminum (Al^{3+}) .

As some soil minerals compose part of the colloidal fraction and have the ability to adsorb cations, for example the clay minerals and aluminum and iron oxides and hydroxides, some effects caused by vinasse application such as pH changes and the increase of organic carbon, CEC and of microbiological activity in addition to elevated disposal of base cations can possibly influence changes in soil mineralogical properties (Ribeiro et al., 2011), and consequently could cause impacts on sugarcane crops productivity.

The current research aims to analyze physical chemical and mineralogical properties variations of a soil of a sugarcane crop because of the vinasse application during the period from 2012 to 2017.

MATERIALS AND METHODS

Research area characterization

The research area is located in *Santa Cruz das Palmeiras* city, which is situated in São Paulo state at the coordinates 21º49'36" S and 47º15'03"W and it belongs to the Mesoregion of Campinas and to the Microregion of *Pirassununga* (IBGE, 2008; *Santa Cruz das Palmeiras*, 2018).

According to technical relatories made at the research area (Arcadis Tetraplan, 2011; Vinasse Application Plan, 2017; CBH-MOGI et al, 1999), this city is inserted in the context of the Hydrographic Basin of the *Mogi Guaçu* River and in the Geomorphologic Unit of the *Paulista* Peripheral Depression, where there are geologic units that are locally characterized by basic intrusive rocks and rocky outcrops of the *Corumbataí* Formation. The region weather of the research area is characterized by a tropical wet weather, with average temperatures between 15ºC to 18ºC at a month period and it is situated at approximately 600 meters above sea level (Arcadis Tetraplan, 2011).

The monthly normal hydric balance of the region of *Pirassununga* city (Figure 1) exposes that most of the rainy period (or surplus water period) occurred from October to the final of March and in May (Vinasse Application Plan, 2017). On the other hand, the period from June to September manifests low precipitation levels that are lower than the Potential Evapotranspiration (PET) measures, and consequently represents the driest period of the year (or the period of water deficit).

The predominant soil type in *Santa Cruz das Palmeiras* is characterized by red latosoils, which is classified as highly intemperized and deep soils with elevated concentrations of clay minerals and Fe_2O_3, which results in an intense red color aspect, and can be found in locals with undulating to smooth undulating relief with low slope.

Vinasse and soil properties

The vinasse analytical results and the physical-chemical properties of soil were obtained by VAP reports that correspond to the period from 2012 to 2017, according to the Technical Standard P4.231 (CETESB, 2015). This Technical Standard also defines how to calculate the maximum vinasse volume to be applied, which is determined from the K_2O content on soil (Equation 1):

$$\frac{m^3 of\ Vinasse}{ha} = \frac{[(0{,}05\ x\ CEC - ks) x\ 3744 + 185]}{kvi} \quad (1)$$

Where CEC is the cation exchange capacity ($cmol_c\ dm^{-3}$); ks is the potassium content on soil ($cmol_c\ dm^{-3}$); and kvi is the vinasse potassium concentration (kg of $K_2O\ m^{-3}$).

The vinasse dosage calculations data were compared to the physical chemical properties of soil, in addition to the analysis of physical-chemical properties of the effluent. On the other hand, the mineralogy characterization was performed by collecting six samples of soil at 30 cm and 100 cm deep on the 27, 29 and 56 farm fields, besides of granulometric, chemical and mineralogical analysis obtained by sifting and weighing the samples and by X-Ray Fluorescence Spectrometer (XRF) and X-Ray Diffraction (XRD), respectively.

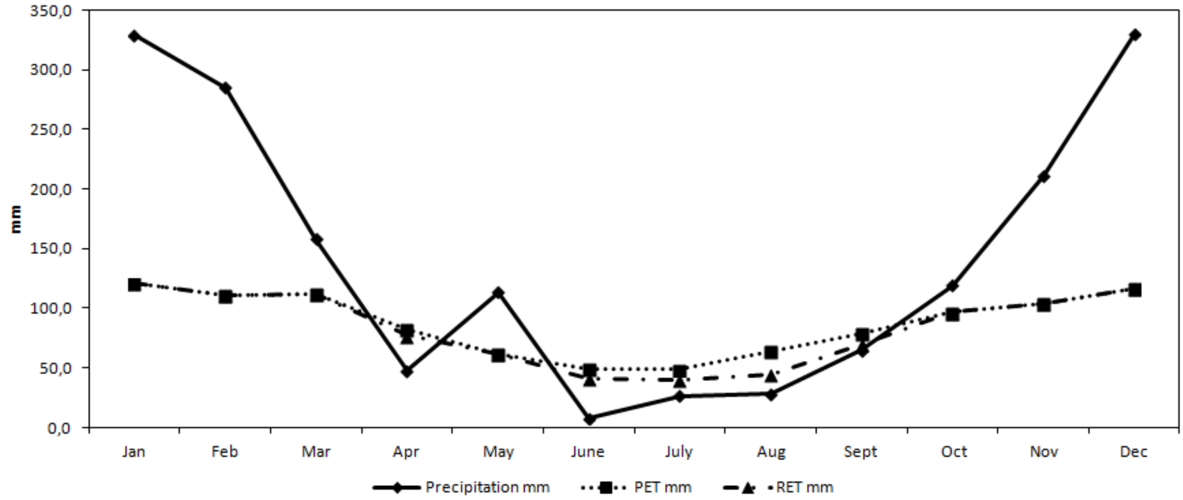

Figure 1 - The monthly normal hydric balance graph of the region of *Pirassununga* city with Precipitation, Potential Evapotranspiration (PET) and Real Evapotranspiration (RET) rates. **Source:** Vinasse Application Plan (2017).

RESULTS AND DISCUSSION

Physical chemical properties of vinasse

The main physical chemical parameters of vinasse (Table 1) that were used on the analysis of soil fertility of the research area were the pH, base cations (K^+, Ca^{2+} e Mg^{2+}), Na^+ (salinity) and COD and BOD, which were compared to the reference values obtained by Fuess (2013) in 2010 at the same sugar and alcohol plant located on the research area.

The variation of pH results was between 3.8 to 6.07 during the period from 2012 to 2017, which most of the analyzed vinasse samples were characterized as acidic and were similar to the reference value of 4.59 obtained by Fuess (2013).

Regarding the concentration of base cations, the vinasse samples presented high K^+ accumulation with an average of 1857.55 ± 1718.83 mg L^{-1} higher than the value presented by Fuess (2013) of 1330.40 ± 0.10 mg L^{-1}, thus being an indicator of the elevated potencial of vinasse as a supplier of this macronutrient to the soil. The average values of Ca^{2+} and Mg^{2+} during the period from 2012 and 2017 were about 451.84 and 219.26 mg L^{-1}, respectively, that were comparable to average results calculated by Fuess (2013) of 458.40 mg L^{-1} of Ca^{2+} and 235.39 mg L^{-1} of Mg^{2+}, which probably indicate a pattern concentration of these cations in vinasse samples.

In relation to the Na^+ quantity, the obtained average was 27.24 ± 19.62 mg L^{-1}. Although this value was approximately three times higher than the reference average of 10.64 mg L^{-1} analyzed by Fuess (2013), the observed Na^+ content was significantly lower than the average Ca^{2+} and Mg^{2+} concentrations, resulting in low risk of salinity.

The average of COD (16.05 ± 9.71 gO_2 L^{-1}) was higher than the BOD average (5.47 ± 3.55 $gDBO_5$ L^{-1}) in a ratio of $R_{COD/BOD}$ correspondent to 2.93, which demonstrates that there were a moderate amount of biodegradable substances in most of the analyzed samples, probably provided by the Vinasse OM. On the other hand, the COD and BOD obtained by Fuess (2013) were respectively 24.63 ± 0.55 gO_2 L^{-1} and 14.40 ± 0.53 $gBOD_5$ L^{-1} with $R_{COD/BOD}$ of 1.71, which presented larger amount of biodegradable fraction additionally to more intense oxygen consumption by biological processes.

Vinasse dosages and physical chemical properties of soil

The highest dosages of vinasse occurred in 2014 in the field 75 with 1557.98 m^3 ha^{-1} and in the field 38 with 1019.63 m^3 ha^{-1}. In 2013, 2015 and 2016, there was the application of vinasse in the field 15, allowing the temporal analysis in this plot. Additionally, the lowest vinasse dosage calculation during the period from 2012 to 2017 was obtained in the field 15 in 2013, corresponding to 21.48 m^3 ha^{-1}.

Compared with the results of physical chemical parameters of soil obtained in 2014 (Figure 2), the vinasse dosage calculations presented a negative correlation with the V% percentages. These percentages exposed a minimum value of 23.42% in the field 75, followed by 35.55% in the field 38, that characterizes these soils as infertile with V% values lower than 50% (Brito, 2013; Ronquim, 2010).

Table 1 - Average Physical Chemical parameters of Vinasse at the period from 2012 to 2017, which were compared to results obtained by Fuess (2013).

Parameter	Unity	2012 to 2017	Fuess (2013)
pH	---	3.8-6.07	4.59
TSS	---	3288.10 ± 2836.68	2757.5 ± 150.64
EC	dS m^{-1}	5.70 ± 4.92	8.703
NO^{3-}	mg L^{-1}	7.96 ± 7.36	55.00 ± 5.00
NO^{2-}	mg L^{-1}	1.66 ± 2.48	2.25 ± 0.05
NH_3	mg L^{-1}	8.50 ± 7.14	87.50 ± 7.50
$N_{Kjeldahl}$	mg L^{-1}	141.06 ± 103.14	1130.50
Na^+	mg L^{-1}	27.24 ± 19.62	10.64
Ca^{2+}	mg L^{-1}	451.84 ± 308.29	458.4
K^+	mg L^{-1}	1857.6 ± 1718.83	1330.40 ± 0.10
Mg^{2+}	mg L^{-1}	219.26 ± 120.93	235.39
SO_4^{2-}	mg L^{-1}	1250.49 ± 783.79	3701 ± 1154.70
P_{Total}	mg L^{-1}	30.04 ± 49.06	5.58 ±0.07
BOD	$gBOD_5 L^{-1}$	5.47 ± 3.55	14.40 ± 0.53
COD	$gO_2 L^{-1}$	16.05 ± 9.71	24.63 ± 0.55
Total Alkalinity	mg L^{-1}	2172.66 ± 1193.70	137

Regarding the results of the field 15 (Figure 3), the percentage of V% was 63.93% in the year 2013 of lowest dosage. From 2015 to 2016, the results did not present a significant variation from one year to the next (from 76.26% to 76.26%), although the dosage calculated in 2015 (168.31 m^3 ha^{-1}) was higher compared to the volume of vinasse calculated in 2016 (88.19 m^3 ha^{-1}). Since V% is the percentage ratio between SB and Total CEC (Ronquim, 2010), its variation is linked to the changes of these two parameters and the cations that compose them.

Regarding the SB parameter, corresponding to the sum of Ca^{2+}, Mg^{2+} e K^+ (Lopes, 1984; Pereira, 2015; Ronquim, 2010), in 2014 the lowest SB value was equivalent to 67.59 $mmol_c.dm^{-3}$ in the field 75 (Figure 2), being negatively correlated with the high dosage calculation obtained in this field. Thus, the soil of this field had low concentrations of base cations (which serve as macronutrients for sugarcane crops) associated with low fertility rates, regardless of the high volumes of vinasse calculated.

Concerning to the SB variations in the field 15 (Figure 3), base cation concentrations increased slightly from 67.39 $mmol_c$ dm^{-3} in 2013 to 80.45 $mmol_c$ dm^{-3} in 2016 (increase of 13.06 $mmol_c$ dm^{-3} over the reporting period), which contrasts with the reduction in dosage calculations from 2015 to 2016.

By understanding base cation variations, it is possible to analyze CEC changes in conjunction with modifications of the Al^{3+} and H^+ concentrations in order to understand soil fertility changes in the researched area. Compared to the 2014 dosage calculations (Figure 2), the highest obtained CEC values were 288.59 $mmol_c$ dm^{-3} in the field 75 and 277.72 $mmol_c$ dm^{-3} in the field 38, which were positively correlated with the calculated vinasse volumes and with a pattern opposite to that observed in the V% results. Since total CEC corresponds to the sum of Ca^{2+}, Mg^{2+}, K^+, Al^{3+} cations and H^+ ions (Ronquim, 2010), probably there was a greater collaboration of Al^{3+} and H^+ in the increase of CEC in the field 75 than the base cations, with consequent reduction of soil fertility indices.

The CEC indices of the field 15 (Figure 3) had similar values in 2013 and 2016 (of 105.36 $mmol_c.dm^{-3}$ and 105.45 $mmol_c$ dm^{-3}). On the other hand, the concentration of this parameter was 96.89 $mmol_c$ dm^{-3} in 2015 (which was the year with the highest dosage of vinasse in this area), being the lowest recorded CEC in the field 15.

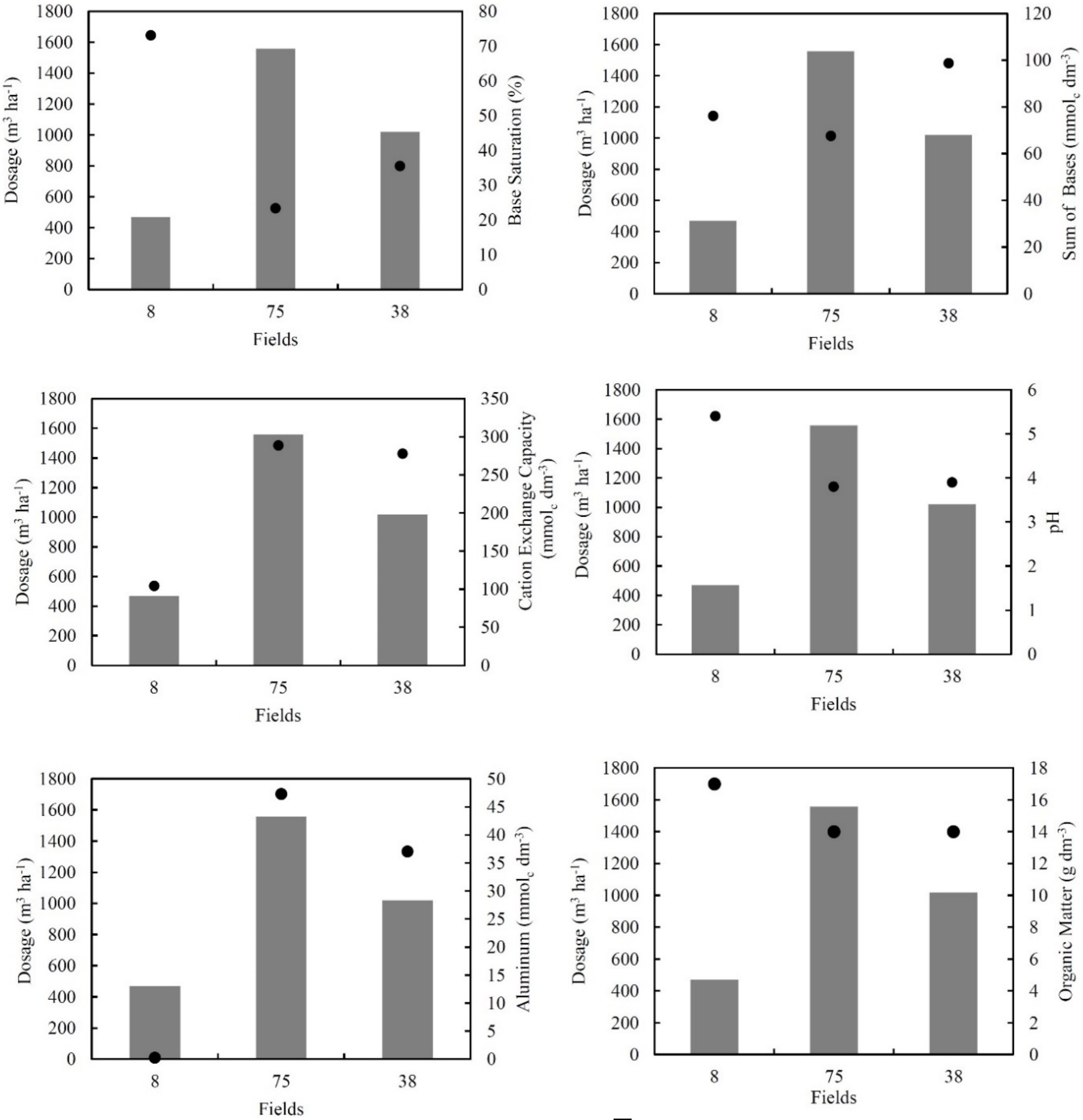

Figure 2 - Comparison of the vinasse dosage calculation in 2014 (█) with the soil fertility parameters (●) for the same year: V%; SB; CEC; pH; Al^{3+}; soil's OM. **Source:** Vinasse Application Plans (2012 to 2017).

In order to understand the total CEC variations in that farm, it was necessary to evaluate the concentration of Al^{3+} and H^+, as well as the base cations results. From 2012 to 2017, the lowest pH values acquired were 3.8 in the field 75 and 3.9 in the field 38, both measured in 2014 (Figure 2). Considering that acidic pH soils have the highest amount of H^+ ions and that the field 75 presented the lowest percentage of V% in 2014, probably the high amount of H^+ ions present in the soil could be related to the reduction of fertility in that period. In turn, the pH variation in the field 15 (Figure 3) was 5.2 to 5.1 from 2013 to 2016, with a minimum temporal variation of this parameter of only 0.1, regardless of variations in the vinasse dosage calculations in the same period.

Regarding the Al^{3+} results, the minimum value obtained during the period from 2012 to 2017 was 0.14 $mmol_c.dm^{-3}$ at the field 15 in 2015, whereas the maximum observed concentrations were 47.33 $mmol_c$ dm^{-3} at the field 75 and 37,05 $mmol_c$ dm^{-3} at the field 38 in 2014. Compared with the vinasse dosages calculated in 2014 (Figure 2), the aluminum content stated in the field 8 was 0.27 $mmol_c.dm^{-3}$, which approaches to zero on the graph. However, the high amounts of Al^{3+} in the fields 75 and 38 were positively correlated to the vinasse dosage calculations, similar to the CEC results and inversely to V%. The variations of this cation in the field 15 (Figure 3) represented the lowest aluminum content during the analyzed period (0.14 $mmol_c$ dm^{-3}), which probably influenced the reduction of Total CEC in 2015 for the same area.

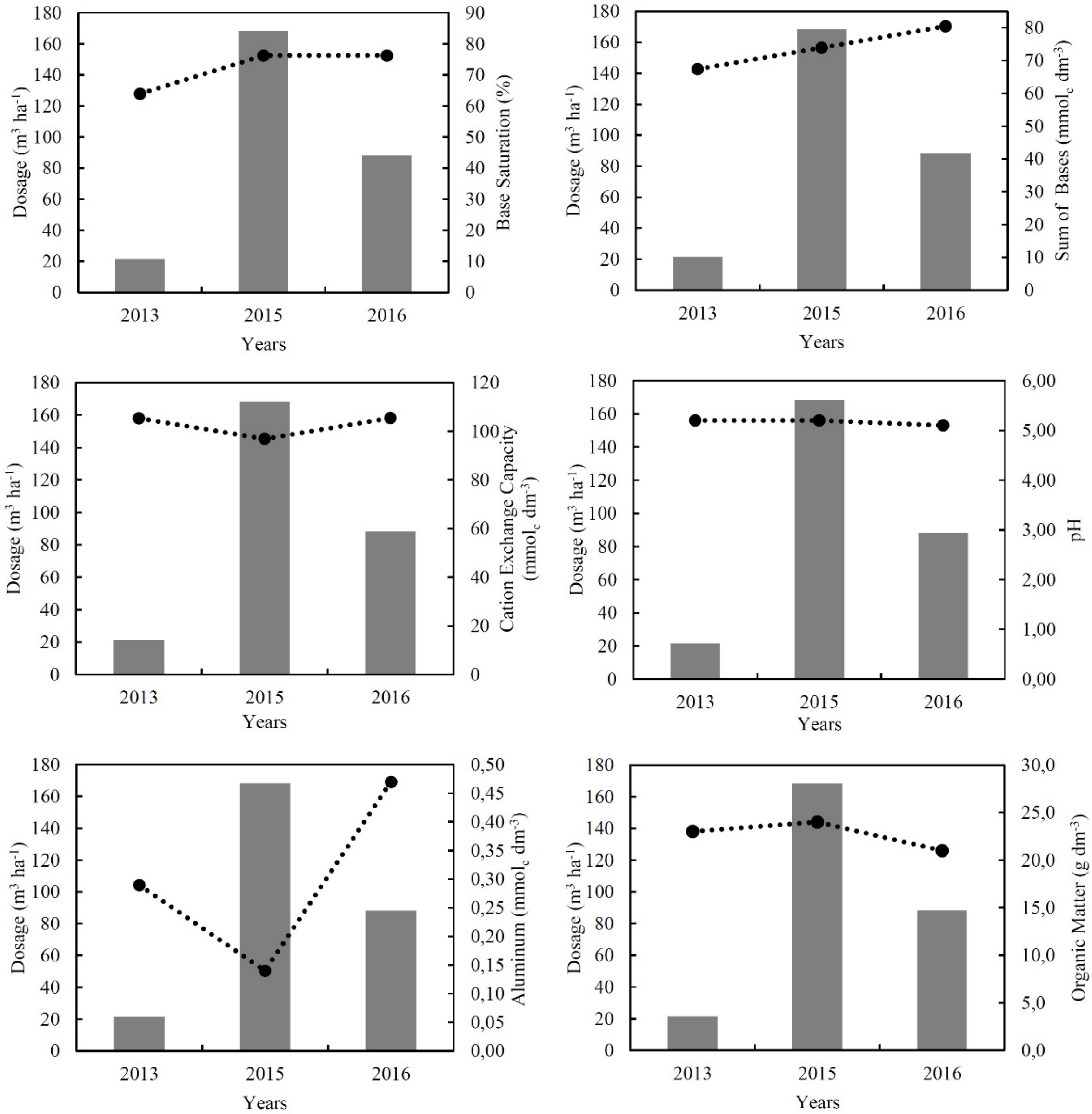

Figure 3 - Comparison of the vinasse dosage calculations in the field 15 (█) with the soil fertility parameters (●) for the same field: V%; SB; CEC; pH; Al^{3+}; soil's OM (Vinasse Application Plans, 2012 to 2017).

In most cases, the results indicated a reduction on the soil fertility with higher concentrations of H^+ and Al^{3+} than SB. In this case, there is a greater collaboration of H^+ and Al^{3+} in the Total CEC than the other exchangeable cations, which resulted in decreased fertility and increased soil acidity and toxicity. As Total CEC concentration is considered to be one of the variables of the vinasse dosage equation (Eq. 1), even soils with high acidity and Al^{3+} toxicity can result in high vinasse volume calculations, and consequently it requires care to not cause risks of reduction on the soil fertility with the application of vinasse along the years.

Besides to base cations and H+Al concentrations, the CEC parameter also depends on the amount of OM present in the soil, as it acts as colloidal particles that have the ability to exchange Ca^{2+}, Mg^{2+}, K^+, Al^{3+} and H^+ ions.

The comparison of soil OM quantities with the vinasse dosages calculated in 2014 (Figure 2) exposed the same amount of OM quantified by 14 g dm^{-3} in the fields 75 and 38, which was associated with different vinasse dosage calculations. As the CEC concentrations of these fields were also similar, this may be an indicative of the direct influence of the amount of soil OM on the increase of cation adsorption. On the other hand, the OM concentration of the field 15 (Figure 3) exposed a positive correlation with the vinasse dosage calculations, which the OM amount measured from 2013 to 2015 varied merely 1 g dm^{-3} (from 23 to 24 g.dm^{-3}), regardless of the difference in the calculated vinasse volumes, followed by a reduction in the amount of OM from 24 to 21 g dm^{-3} in the period from 2015 to 2016. However, there was a decrease in the CEC content in 2015 compared to the other years of application of vinasse in the field 15, which could be an evidence that the OM increment is not the only factor that influences on the CEC increase on soil, also having the influence of the presence of clay minerals and iron and aluminum oxides that compounds the colloidal fraction of the soil and also have the capacity to adsorb cations.

Mineralogical analysis

The granulometric analysis of the collected soil samples presented higher fraction of fine sand (0.25 to 0.125 mm) with percentages between 42.303% to 61.155%, followed by medium sand fraction (0.5 to 0.25 mm) with content between 12.478% to 29.587% and very fine sand (0.125 to 0.064 mm) with percent between 12.108% a 21.886%, which characterizes the soil of the research area as very fine to medium sandy. The XRF results indicate high percentages of silica (SiO_2) between 33.81 to 83.10%, and significant Fe_2O_3 content between 4.66 to 25.21% and aluminum (Al_2O_3) with percentages of 6.27 to 21.45%, in addition to other identified trace elements that were measured in ppm such as V, Zr, Ba and Cr.

In its turn, the mineralogical composition obtained by the XRD test (Table 2) indicated the presence of clay minerals of the group of kaolinite (kaolinite, halloysite, dickite and nacrite), gibbsite, hematite and quartz, additionally to the occurrence of minerals of Birnessite, Anatase, Siderite and Magnesite.

Table 2 - Mineralogical composition of the soil samples obtained on the research area and determined by XRD test with their respective chemical formulas.

Samples	Fields	Soil Mineralogy
P1- 30 cm	**Field 27**	Kaolinite - $Al_4(Si_4O_{10})(OH)_3$, Gibbsite - $Al(OH)_3$, Quartz – SiO_3, Siderite - $FeCO_3$, Magnesite – $MgCO_3$, Hematite - Fe_2O_3
P1-100 cm	**Field 27**	Kaolinite - $Al_4(Si_4O_{10})(OH)_3$, Halloysite $Al_4(Si_4O_{10})(OH)_8$, Birnessite (rica em Na) - $(Na_{0,7}Ca_{0,3})Mn_7O_{14}.2,8H_2O$, Gibbsite - $Al(OH)_3$, Quartz – SiO_3, Hematite - Fe_2O_3.
P2-30 cm	**Field 29**	Kaolinite - $Al_4(Si_4O_{10})(OH)_3$, Gibbsite - $Al(OH)_3$, Quartz – SiO_3, Hematite - Fe_2O_3.
P2-100 cm	**Field 29**	Dickite - $Al_4(Si_4O_{10})_3(OH)_{12}.3H_2O$, Gibbsite - $Al(OH)_3$, Quartz – SiO_3, Anatase – TiO_3, Siderite - $FeCO_3$, Magnesite – $MgCO_3$, Hematite - Fe_2O_3.
P3-30 cm	**Field 56**	Dickite - $Al_4(Si_4O_{10})_3(OH)_{12}.3H_2O$, Gibbsite - $Al(OH)_3$, Hematite - Fe_2O_3.
P3-100 cm	**Field 56**	Nacrite - $Al_4(Si_4O_{10})(OH)_8$, Gibbsite - $Al(OH)_3$.

The results of the analysis of the collected soil samples exposed a predominance of the main constituent minerals of dark red latosols that are usually typical in humid tropical climate regions, such as the clay minerals of kaolinite group, quartz and iron aluminum oxides (gibbsite and hematite). As clay minerals of the kaolinite group and iron and aluminum oxides have low CEC (Vieira, 1988), the addition of OM in the colloidal fraction of the soil through the application of vinasse concedes the increase of soil CEC. On the other hand, other less frequently occurring minerals are related to some of the trace elements observed, such as the Anatase, which Ti content can interfere with the V concentration results on the XRF test (Simabuco & Nascimento Filho, 1994), and the Birnessite, which may be associated with Ba, Zn, Ni, Cu, Sr and Co concentrations (Figueira et al., 2016).

The predominance of aluminum and iron minerals in a sandy soil possibly is an indicator of a high leaching of the Ca^{2+}, Mg^{2+} and K^+ cations (that are more soluble and easily transported) in the soil profile, with consequent accumulation of SiO_2, Fe_2O_3 and Al_2O_3 through weathering of primary minerals of basic intrusive rocks and sedimentary rocks of the *Corumbataí* Formation, which were originated by total or partial hydrolysis of kaolinites and gibbsites that release more Al^{3+} and H^+ to the soil (Vieira, 1988). The described process also has a considerable influence provided by the low amplitude and moderate slope reliefs with formation of deep soil horizons and by the regional water balance, considering that the leaching process is more prevalent in periods of water surplus. Therefore, the period of water surplus that corresponds to the farm's off-season period (Arcadis Tetraplan, 2011) can contribute to the base cations leaching and to the increase of the H+Al concentrations on the soil, whereas the application of vinasse in the harvest period, that occurs in the water deficit period, can increase the soil moisture and replenishes the amount of Ca^{2+}, Mg^{2+} and K^+, that were leached in the rainy season. However, some of the fertility results did not manifest higher H+Al concentration than SB, which can possibly be an evidence of the influence of the type of management used in the sugarcane crop, such as liming the crop in the rainy season.

CONCLUSIONS

In the water surplus season, the processes of the Ca^{2+}, Mg^{2+} and K^{+} leaching and the increase of H+Al in soil can be intensified as a result of the hydrolysis of kaolinites and gibbsites, which are influenced by soil granulometry and physical aspects such as geology and type of relief.

The vinasse application at the time of water deficit during the crop harvest provides an increase on the soil moisture and the macronutrients supply, resulting in good fertility rates.

However, the fertility variations do not depend exclusively on the vinasse application and may be caused by changes in water balance and by the use of some types of management, which should also be considered on the research.

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