Decomposition of the organic matter of natural and concentrated vinasse in sandy and clayey soils

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ABSTRACT

Vinasse has been used as fertilizer by sugarcane growers, due to its potential to completely replace mineral fertilizers. However, if the application is not adequate, this practice may cause environmental contamination. This study used a respirometry test to evaluate the organic matter (OM) decomposition present in natural vinasse and concentrated vinasse (CV), with or without urea addition. The experiment involved two soil types and two types of vinasse at different application rates. The vinasse chemical characterization showed high levels of pseudo-total potassium (K) in both vinasses, which are not considered in the application rates. Decomposition rates above 90% and between 70 and 80% were obtained for sandy and clayey soils, respectively, over a brief 41-day period, indicating rapid OM decomposition. Positive priming effect was observed for CV and CV + urea treatments in sandy soil. An important implication of these findings revealed that K not available in vinasse was released in the soil solution by the OM mineralization, indicating the possibility of overestimation in the vinasse application rates. Therefore, K pseudo-total values should be considered in the calculation of the vinasse application rates. However, studies involving K mobility into soil are needed to validate this hypothesis.

Key words | organomineral fertilizer, respirometric test, sugarcane

INTRODUCTION

The degradation of different wastes depends on local and regional conditions (climate, soil type, decomposing microorganisms, etc.). Soil properties, such as clay, pH, organic matter (OM) and aeration act as environmental factors of the decomposition process. The waste degradability is related to its relative amount of different substrates or chemical components, such as organic acids, which are readily assimilable, amides that are moderately assimilable and lignin with slow assimilation (Moreira & Siqueira 2006). The fraction composed of readily decomposable substrates rapidly turns into carbon dioxide (CO₂) and biomass.

Nitrogen is essential for the growth of soil microorganisms, which can use a wide range of nitrogen (N) compounds. According to Geisseler et al. (2009) when organic N sources are degraded, microorganisms can either decompose simple organic molecules directly (direct route), or organic N may be mineralized first and taken up in the form of mineral N (mineralization–immobilization–turnover route). In soil ecosystems, results from Gibbs & Barraclough (1998), indicate that the direct route may be important. Geisseler et al. (2009) affirmed that little information is known about the factors that determine the relative importance of the two routes. The C/N may have an effect, as microorganisms may take up N containing organic molecules not only because of the need for nitrogen (N), but also to meet their carbon (C) and energy requirements.

The contribution of organic materials to the soil under equilibrium conditions stimulates the microbial population as a function of the amount of oxidizable C and increases the nutrient consumption by the decomposing microbiota. According to Moreira & Siqueira (2006), when the C/N of the waste is between 20 and 30, a balance is reached between immobilization and mineralization.

Vinasse is a waste product obtained from the fractional distillation of sugarcane juice to obtain ethanol, and 7 to 20 liters of vinasse can be generated per liter of ethanol, depending on the alcohol concentration (Fernandes 2003). This waste presents high levels of labile organic C and potassium (K), as well as sulfur (S), calcium (Ca), nitrogen (N), magnesium (Mg) and phosphorus (P) (Otto et al. 2009).
As vinasse presents a medium to high C/N ratio and in order to avoid N immobilization in the soil, it is a common practice to supplement vinasse with mineral N (Silva et al. 1999).

Vinasse has been used as fertilizer by sugarcane growers, because there is the potential to completely replace mineral fertilizers for sugarcane. In addition to representing growers, because there is the potential to completely replace practice to supplement vinasse with mineral N (Silva et al. 1999)). As vinasse presents a medium to high C/N ratio and in order to avoid N immobilization in the soil, it is a common strategy for Brazil. From January to April 2016, the sales of K₂O fertilizer represented approximately 53% compared with nitrogenous and phosphate fertilizers (ANDA 2016). According to Barbosa et al. (2012), the savings generated by the use of vinasse as a K source is close to US$898.00 per hectare.

The increase in vinasse generation and, consequently, intensification of its application can cause soil, surface and groundwater contamination. The Environmental Company of Sanitation Technology (CETESB) of São Paulo state, Brazil, approved the Technical Standard P4.231, which describes the criteria and procedures for the application of vinasse in agricultural soils in São Paulo state (CETESB 2015). The standard requires that the concentration of vinasse applied to agricultural soils must take into account the K concentration in the soil and in the waste. In order to conduct a less complex analytical method, the literature recommends the K determination by emission flame photometry, using as arguments its simplicity and speed, making it accessible to most laboratories (Glória & Rodella 1972). However, this determination could overestimate the application rate of vinasse in soils, which could result in contamination of the soil-water system.

In Brazil, one alternative to reduce logistical costs, environmental impacts and increase the fertilization at distances greater than 30 km away from sugarcane growers is concentrating the vinasse and supplementing it with N fertilizers. In this process, which uses specific evaporators that operate with viscous solutions, the vinasse volume can be reduced by up to 80% (Carvalho & Silva 2010). On the other hand, this process also increases the concentration of non-volatile substances, either in solution or in a suspension. However, the concentrated vinasse (CV) retains its fertilizing properties, and thus can be applied in smaller quantities compared to natural vinasse (NV).

Despite all the benefits associated with organic fertilization and the use of vinasse in agricultural soils, the characteristics of this waste product combined with inadequate management can result in imbalances in soil, particularly losses in nitrogen or carbon. According to Hamer et al. (2009), soil microorganisms may be activated by the supplied easily available organic substrates, thereby, enhancing the mineralization of native soil OM (priming effect), especially in combination with N fertilization. This represents an unwanted condition for soils in the tropics which are poor in OM, and this has not been considered in the Brazilian environmental legislation. CETESB defined a minimum of 30% of C-organic degradation of wastes within 80 days in order to avoid the accumulation of organic recalcitrant material in soil (CETESB 1999).

The respirometry test is used to evaluate the process of biodegradation of organic wastes incorporated into soil through the action of microbiota present in the soil. Microbial respiration can be assessed by monitoring the amount of CO₂ produced during the process of biodegradation. Therefore, this is a biochemical parameter which can be used to indicate microbial activity in soil. This technique is based on the addition of biodegradable OM to a particular soil. The presence of fresh OM stimulates the growth of the native soil microbial, which results in substrate decomposition, subsequently increasing the oxygen (O₂) consumption and generation of CO₂. The respirometry test has been shown to be quite versatile in studies that have assessed biodegradable compounds, as well as recalcitrant ones, including oils, volatile organic compounds of high and low solubility and phenolic compounds (Haines et al. 1996).

This study used a respirometry test to evaluate the decomposition rate of OM present in NV and CV, with or without urea addition, in clayey and sandy soils, in order to determine the impact of their application in soils cultivated with sugarcane. The tested hypothesis was that pseudo-total K in vinasse is released into the soil solution.

**METHODS**

**Respirometry test**

The respirometry test, modified by Bartha (CETESB 1990), were performed in sealed glass flasks containing 500 g of a medium-sandy textured soil with 12% of total sand (Dystrofertic Red Yellow Argisol – Hapludult), or with a clayey-textured soil containing 63% of clay (Eutrophic Red Latosol – Eutorthox). Soil samples were collected from the 5 to 20 cm layer of different fields previously cultivated with sugarcane located in Piracicaba City, São Paulo, Brazil. The soils were placed in 0–0.20 cm layers, and then moistened with deionized water to achieve 70% of the field
capacity. The weight of the flasks was noted to allow for the water content to be kept constant.

The respirometry test was conducted using a completely randomized design, consisting of two soil types (sandy and clayey), two types of waste (NV and CV) and two vinasse application rates (20,000 and 6,500 L ha\(^{-1}\) for NV and CV, respectively), with three replicates in each treatment, totaling 24 plots. An additional treatment of CV + urea (75 kg ha\(^{-1}\) of N-urea) was performed to study the change in waste decomposition in the presence of an appropriate N concentration to the sugarcane cycle. Considering that the CV was three times more concentrated than the NV, the NV application corresponded to this concentration rate, which is not used in agricultural practices. In addition, this reduced rate was defined in order to avoid changes in the values for vinasse organic load degradation, which could potentially mask the results.

The flasks were incubated in a biochemical oxygen demand incubator at 25 ± 2 \(^{\circ}\)C, and the amount of CO\(_2\) released from vinasse, as well as from the soil native OM material, was quantified by measuring the electrical conductivity (EC) of a NaOH solution (0.5 mol L\(^{-1}\)) contained within the flask. The measurements were conducted until EC values in the NaOH solution had stabilized over three consecutive measurements as proposed by Rodella & Saboya (1999). After the test, the soils were removed from the respirometers and samples from replicates of the same treatment were homogenized to perform the analysis of fertility parameters.

Soil and vinasse analysis

The vinasse physical-chemical analysis included the determination of pH, EC, water content (Brazil, 2007), organic C (OC) (Walkley & Black 1934), organic N (Kjeldahl method), N-NO\(_3\) + NH\(_4\)\(^+\) (AOAC 1984) and available and pseudo-total concentrations of K and P (Mattiazzo-Prezotto & Glória 1990). It is known that the OM present in vinasse easily decomposes in soil, and most Brazilian analytical laboratories are able to determine the available K concentration of vinasse. Therefore, the pseudo-total concentrations of vinasse were also determined to assess the suitability of these rates of application, in order to maximize the efficiency of the nutrients. It is more common to find the pseudo-total denomination for metals when using a milder digestion. However, the term can also be adapted for nutrient determinations.

For K and P pseudo-total determinations, only nitric acid was used in the digestions. Abreu et al. (1996) demonstrated that, when compared to conventional methods employing nitric-perchloric extractions, this type of digestion exhibited similar results and extracted 80 to 87\% of nutrients bound to organic material.

The fertility parameters were analyzed according to the fertility analysis methods developed by the Agronomic Institute of Campinas, São Paulo, Brazil (van Raij et al. 2001).

Statistical analysis

Results for the released C-CO\(_2\) were adjusted depending on the incubation period using a first-order chemical kinetics equation (Latham 1974) for obtaining the rate of OM degradation, the potential amount of degraded C and the half-life of the organic waste (Equation (1)). The adjusted results were statistically analyzed using the Tukey’s test at a 5\% significance level, using the statistical analysis program R (version 2.15.1).

\[
C_{\text{degraded}} = C_0 \cdot \left(1 - e^{-kt}\right) \quad (1)
\]

\(C_{\text{degraded}}\) is the amount of carbon in the form of CO\(_2\) released during the test period (mg 500 g\(^{-1}\) soil\(^{-1}\)).

\(C_0\) is the potentially mineralizable carbon during the test period (mg 500 g\(^{-1}\) soil\(^{-1}\)).

\(k\) is the reaction rate constant for the degradation of organic carbon (day\(^{-1}\)).

\(T\) is the experimental time period (days).

Equation (1) estimates the amount of C released as CO\(_2\) (C-degraded) derived from the degradation activity of the OM present in the soil of each treatment. Additionally, the potential degraded C values as CO\(_2\) (\(C_0\)) were estimated, which represented the potential amount of C-CO\(_2\) released from OM degradation, as well as its speed of degradation for each treatment (\(k\)). From these determined parameters, the half-life (\(T_{1/2}\)) was estimated, which represented the period (days) required for 50\% of the \(C_0\) value to be released from OM degradation for each treatment.

RESULTS

Vinasse and soil physical-chemical characterization

Table 1 shows the analytical results for vinasses used in the experiment. The concentration process resulted in a 16\% reduction in the water content and did not promote pH alteration. In contrast, there was an increase greater than 50\% in EC values. The chemical parameters were concentrated, on average, four times. K\(_2\)O pseudo-total
The concentration was significantly higher than the available one in both vinasses (138% and 52%, respectively).

**Table 2** shows the analytical results for the investigated soils. In tropical conditions, the formation of acid soils is common due to the high weathering process. The clayey soil, although presented a lower pH, for the other fertility parameters, higher values were observed in relation to the sandy soil.

**OM decomposition**

Figure 1(a) and 1(b) illustrate the amount of C-CO$_2$ evolved from sandy and clayey soils treated with NV or CV (with or without urea), respectively. In the initial phase of the incubations, there was a greater C-CO$_2$ release and after 30 days, a stabilization of the C-CO$_2$ evolved was observed; however, the experiment was conducted for up to 41 days to confirm the results. The same trends were observed among the vinasses curves only differing from the amount of C-CO$_2$ evolved. As CV originally was 1.3 times more concentrated in organic C, the losses were also approximately 1.3 times higher compared to NV.

**Table 3** shows some statistical parameters of the experiment, as well as the amount of C evolved of each treatment with its correspondent percentage of C-waste degraded, which was calculated from the relation between the amounts of C evolved and added from the vinasse. The first-order mathematical model showed data reliability, as the variation coefficient values ranged from 1.0 to 6.0% for various parameters and correlation coefficients were close to 1.0 (Table 3).

The half-life was significantly higher in sandy soil when compared with clayey soil for all treatments (Table 3). In the sandy soil, the longest decomposition period was observed for the control soil, followed by the CV + urea treatment, CV and then, NV (Table 3). No significant difference was found for the half-lives among NV, CV and CV + urea treatments considering the same soil (Table 3).

The highest decomposition rates were observed for the sandy soil for all treatments and additional OM decomposition rates were observed for the CV + urea and CV treatments, respectively (Table 3). In sandy soil, decomposition rates greater than 100% reflected complete decomposition of CV organic load and additional soil OM decomposition compared with the control soil.

**Physical-chemical characteristics of the investigated soils: data on fertility parameters**

**Table 4** shows the analytical results for some fertility parameters of the control and treated soils after the respirometry test to determine any changes after the incubation of the soils with equal NV or CV concentrations. In relation to K$_2$O, the amount provided to the soils were 52 and 124 kg of K$_2$O ha$^{-1}$ for available and pseudo-total concentrations for NV treatment, respectively, and 102.7 and 156.0 kg of K$_2$O ha$^{-1}$ for the CV treatment, respectively. For NV and CV, there was an average incorporation of 17.5 kg of N ha$^{-1}$.

In relation to the P concentration, the available concentration provided by NV added 29.5 and 144.1 kg ha$^{-1}$ of P$_2$O$_5$ for the natural reserves of the sandy and clayey soils, respectively (Table 4). For the soils treated with 6.5 m$^3$ of
CV, 30.8 and 145.4 kg ha\(^{-1}\) of P\(_2\)O\(_5\) was supplied for the sandy and clayey soils, respectively (Table 4).

There were increases of 0.5 and 0.1 in pH values of sandy soil with NV and CV addition, respectively. The addition of urea with CV did not alter the pH of the soil (Table 4). There was also no change in pH in the clayey soil after the 41-day test period, demonstrating the higher buffering ability of this soil.

The EC was naturally higher in the clayey soil control, reflecting a greater amount of ions in solution and, thus, higher fertility compared with sandy soil. However, after the respirometry test, the sandy soil had greater increases in EC compared with clayey soil. This indicates higher OM decomposition rates and nutrient release in the sandy soil, thus confirming the respirometry test results (Table 4). In sandy soil, the increased EC differed between the treatments (65.4, 113.9 and 232.6% for NV, CV and CV + urea, respectively), which was reduced in the clayey soil (53.0, 63.7 and 54.5%, respectively). This suggests that the highest decomposition rate of OM occurred in sandy soil, which supports the results obtained in the respirometry test (Table 3).

The OM concentrations observed after the incubation period in both soils (Table 4) accurately reflects the process of waste decomposition described in Table 3. The inputs of sulfate (S-SO\(_4\)\(^2-\)) from NV and CV application rates were 30 and 45 kg ha\(^{-1}\), respectively (Table 4). For the clayey soil, S-SO\(_4\)\(^2-\) concentrations were the same for both the NV and CV treatments. The values V% and CEC do not accurately reflect the use of vinasse in soils, because the sandy soil was collected in a sugarcane planting area in which liming had been performed, while the clayey soil was collected in a ratoon sugarcane area in the reform phase.

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**Figure 1** | Amount of C-CO\(_2\) evolved (mg 500 g\(^{-1}\) soil\(^{-1}\)) from sandy (a) and clayey soils (b) treated with the NV or CV (with or without urea).
DISCUSSION

Physical-chemical characterization of vinasse

There are some published papers in the literature on CV physical-chemical characterization (Ueno et al. 2014; Dalri et al. 2014). Otto et al. (2017) performed the same characterization in CV and their results differed up to 66% compared to the present study. This difference may be related to the vinasse composition, which is influenced by the aspects of sugarcane culture, as well as its processing (Rossetto et al. 2008).

According to Table 1, increases of 140% and 52% were observed in K2O pseudo-total values compared with the available ones for NV and CV, respectively. These results indicate the possibility of interaction of some organic acids present in vinasse (Silva et al. 2014) in the kinetics of K release, according to Meurer & Castilhos (2001). However, further studies are needed to prove this hypothesis.

OM decomposition

Half-lives

The longest half-lives were observed for both control soils, which was probably related to the presence of increased recalcitrant organic compounds derived from the soil native OM. The adsorption of organic compounds to clay and silt particles is an important determinant of the OM stability in soils (Hassink 1997). According to Baldock & Skjemstad (2000), the OM protection from the presence of mineral particles results in recalcitrant materials during decomposition, because the labile components are preferentially utilized, resulting in concentration of recalcitrant structures. The non-significant relation for half-lives among NV, CV and urea additions indicated a reduced effect of

| Table 3 | Parameters used in the first-order kinetics equation, adjusted for the C-CO2 evolved from the waste as observed in the respirometry test and fraction degradation |
|-------------------|-------------------|-------------------|-------------------|-------------------|
| Treatment         | R²                | t½ (day)          | C0 (mg 500 g⁻¹ soil⁻¹) | C-evolvedb       | C-waste degraded (%) |
| Sandy             |                   |                   |                   |                   |                     |
| Control soil      | 0.99              | 19.7Aa            | 206.0Ca            | 151.0Ba           | 21.6Ca               |
| NV                | 0.97              | 8.4Da             | 265.2Ba            | 109.0Ca           | 74.8Ba               |
| CV                | 0.97              | 9.9Ca             | 376.1Aa            | 212.0Aa           | 112.2Aa              |
| CV + urea         | 1.00              | 12.8Ba            | 397.5Aa            | 193.9Aa           | 102.6Aa              |
| Clayey            |                   |                   |                   |                   |                     |
| Control soil      | 0.98              | 12.3Ab            | 109.9Cb            | 96.1Bb            | 2.2Cb                |
| NV                | 0.97              | 6.8Bb             | 186.8Bb            | 89.9Bb            | 61.7Bb               |
| CV                | 0.98              | 5.6Bb             | 245.4Ab            | 152.9Ab           | 80.9Ab               |
| CV + urea         | 0.98              | 5.3Cb             | 261.4Ab            | 169.7Ab           | 89.8Ab               |
| Variation         | 5.8               | 4.4               | 1.1               | 6.5               |                     |

Each data point is an average of the three replicates. Means followed by the same uppercase letter do not differ among treatments and means followed by the same lowercase letter do not differ between soil types.

\( \text{C}_0 \) = potentially mineralizable C.

\( \text{t}_{1/2} \) = half-life.

| Table 4 | Physical-chemical of the investigated soils treated with NV and CV after the respirometry test |
|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|
| Treatments        | pH                | CaCl₂ (EC) µS cm⁻¹ | OM%              | kg ha⁻¹            | mmol dm⁻³ |
| Sandy soil        |                   |                   |                   |                   |         |
| Control soil      | 5.4               | 89                | 1.6              | 27.5              | 15       |
| NV                | 5.9               | 117               | 1.8              | 32.1a             | 16       |
| CV                | 6.1               | 203               | 1.5              | 36.7a             | 13       |
| CV + urea         | 6.2               | 415               | 1.4              | 32.1a             | 15       |
| Clayey soil       |                   |                   |                   |                   |         |
| Control soil      | 4.4               | 295               | 1.8              | 142.1             | 42       |
| NV                | 4.5               | 314               | 2.3              | 32.1a             | 52       |
| CV                | 4.5               | 376               | 2.1              | 41.2a             | 52       |
| CV + urea         | 4.3               | 327               | 2.1              | 45.8a             | 52       |

\( a \) = Liquid element input in soils.

– Analysis not performed.
vinasse OM and urea fertilization on the speed of decomposition (Table 3).

Prezotto et al. (2008) evaluated the degradation of organic C present in NV and CV from three successive applications in sandy and clayey soils at rates corresponding to 200 kg ha$^{-1}$ of K$_2$O in each application. For NV, the half-life ranged from 2 to 8 days and for CV, it was between 5 and 10 days. The successive applications of vinasse did not alter the half-lives in relation to the first application.

C-CO$_2$ evolved and C/N ratio

Prezotto et al. (2008) observed similar C-CO$_2$ values to those reported by this study (600 and 1,300 mg 500 mg$^{-1}$ soil$^{-1}$ for NV and CV, respectively); however, these authors did not observe differences between the same soil textures. Minhoni & Cerri (1987) evaluated the vinasse decomposition under different soil moisture levels (40, 60 and 80% of the soil water capacity retention). These authors concluded that, in spite of the initial differences in the average daily rates of C release, at the end of the incubation period, the total amount of C released was similar.

The CV + urea treatment in clayey soil showed higher rates of C-CO$_2$ evolved (Figure 1(b)) when compared with sandy soil (Figure 1(a)). However, there were no significant differences between the CV and CV + urea treatments over time (Table 3). The N addition to a C-rich clayey soil may have provided a better balance in the system C/N, resulting in increased C-CO$_2$ release. Ermani et al. (2002) observed that the application of 100 kg ha$^{-1}$ of N in oats straw promoted a 69% increase in its decomposition in relation to the treatment without N. The N application decreased the C/N of the waste and activated the microbial decomposition process, as observed in the present study.

The C/N of NV and CV were 20/1 and 26/1, respectively. The ratio for NV was closest to the ratio of the soil native OM, therefore, is more easily adaptable to decomposition by soil microflora. This resulted in a shorter C half-life for both types of soils with NV addition.

The C/N of CV required adaptation for the soil microflora, with the potential temporary immobilization of N from soil, which slowed down the decomposition process, thus resulting in a longer half-life of the potentially mineralizable C (Table 3). Furthermore, during the NV concentration process, which can reach temperatures above 80°C, it is possible that alcohols are released and may have formed complex aromatic molecules (insoluble organic acids and phenols), thus, changing the OM structure present in vinasse (Parnaudeau et al. 2008). Such compounds could result in higher and, consequently, higher resistance to decomposition, particularly for applications in clayey soil, in which there is increased protection against the rapid OM decay (Hassink 1997).

Decomposition rates

The OM decomposition in the sandy soil was more pronounced than in the clayey soil, even when the soil native OM was considered, as the decomposition rate was 10% over the same period and under the same humidity and temperature. The increased aeration of sandy soils favors aerobic processes, and therefore, reduces changes in the concentration of minerals such as kaolinite, iron oxides and gibbsite. According to Six et al. (2002), C dynamics in soil are strongly related with the soil structure and its ability stabilize soil OM. These minerals are abundant in clayey soils in the tropics, which protect against the rapid OM decay (Zech et al. 1997; Bayer et al. 2002), as shown in Table 3.

In clayey soil, the majority of the degraded C came from the wastes addition, which resulted in greater preservation of its soil native OM (only 2.2%), due to its higher recalcitrance. In these soils, the preservation of the native OM and incorporation of new organic material compounds resulted in an increase in the OM pool, which is important for the maintenance of soil fertility.

CV and CV + urea promoted higher decomposition rates which did not differ considering the same soil texture (Table 3). Most of the OM in the investigated soils was provided by the vinasse, and the CV presented a greater amount of potentially mineralizable C and higher decomposition rates (Table 3). However, in sandy soil, both NV and CV addition promoted higher decomposition rates of mineralizable C, enhancing the mineralization of native OM (priming effect).

The priming effect is characterized by the extra release of soil-derived C or N. According to Kuzyakova et al. (2000), the most important mechanisms for this process are the acceleration or retardation of soil OM turnover due to increased activity or amount of microbial biomass. For easily available organic substances, the possible causes for priming effect are the increase in microorganism’s activity and acceleration of soil OM mineralization (Kuzyakova et al. 2000). Vinasse presents a high proportion of its organic constituents in the soluble form, such as glycerol and organic acids, which have immediate degradation in the soil (Caldeira 1997).

There are few extended studies concerned with priming effect in vinasse decomposition. Grigatti et al. (2010)
observed for different application rates of vinasse a mineralization up to 100%. Prezotto et al. (2008) evaluated the organic C degradation of two types of vinasse applied to different soil types, and determined that the average C degradation rate was 61.4% in NV and 56.8% for CV. These authors did not verify the priming effect for CV decomposition in sandy soil. It is important to mention in relation to priming effect, results are strongly dependent on the experimental conditions, especially considering which kind of substances were added to the soil (Kuzyakova et al. 2000).

Potrich et al. (2014) evaluated the effect of the application of different rates of N on the cultural residues of sugarcane and the priming effect was verified in all tested N rates. Hamer et al. (2009) track the effects of urea fertilization on soil OM of an active and abandoned pasture site and observed that the fertilization with urea induced positive priming effect and promoted an alteration of the microbial community structure in favor of fungi.

**Correlations between K pseudo-total concentrations and the vinasse application rates**

The results of vinasse characterization in relation to K, as well as the results of this waste decomposition on the investigated soils, revealed the presence of pseudo-total concentration of K in vinasse (Table 1) and that it can be released into the soil solution by the OM mineralization (Table 3). This confirmation may indicate the possibility of overestimation in the application rates of the vinasse in cultivated soils. However, studies involving K mobility into soil are needed to predict with greater certainty the possibility of the use of photometry flame for K determination causing possible environmental contaminations.

**Fertility analysis of the investigated soils**

**Potassium**

According to Table 4, both vinasse additions provided an additional amount of K2O for the control soils. However, for NV and CV, considering the pseudo-total concentrations (Table 1), only the clayey soil would supply 185 kg ha\(^{-1}\) of K2O required for sugarcane growth (CETESB 2015). The contribution of the natural K2O reserves in both soils did not reflect in increases in their solutions, because the same net contribution, of 59 kg ha\(^{-1}\), was observed. This value was 13% greater than the available concentration (2.6 kg m\(^{-3}\)), contributed by the application of vinasse and represented the excess concentration derived from the OM decomposition in both the sandy and clayey soils (Table 3).

The values for the sandy and clayey soils treated with CV presented 75% and 108%, respectively, of the amount contributed by the application rate (Table 4). These values did not follow the trend of OM decomposition, which was higher in sandy soil (112.2%) compared with clayey soil (80.9%), indicating that K in the CV was already present in an available form with no correlation with C degradation (Table 3).

In CV + urea treatment, almost 100% recovery of the available K2O concentration added from the waste was observed in both soil types (Table 4). This result demonstrates that, although the N addition had accelerated the microbial degradation, its recovery was similar in CV treatments with and without N, as K is not an element of structural nature.

There was an increase from 30 to 90% in available K concentration in the sandy and clay soils, respectively, when treated with CV compared with NV. The sandy soil treated with CV + urea presented 28.6% more K2O than the same soil treated with CV. When the NV and CV + urea treatments were compared, the K2O concentrations were 67% and 74% higher for the CV + urea treatment, for the sandy and clay soils, respectively.

Mehlich 1 is a mild extractor, and that the K extracted by other extractors, such as nitric acid, may be closer to or higher than the obtained K pseudo-total value in vinasse. This mild extractor was selected to determine the concentration of elements in soil due to the waste decomposition rate and the release of nutrients to the system. Furthermore, the analyses were performed on homogenized soil samples from each treatment after the incubation period, which may have diluted the mineralized elements. Under field conditions, it is possible that these processes are localized in just a few centimeters of soil.

**Phosphorus**

As observed in K2O concentrations for NV treatments, both soils presented 32.1 kg ha\(^{-1}\) of P2O5, which was approximately one and half times more than the total amount contributed to the sandy soil, indicating the OM decomposition in the soil and P release. Despite the higher K reserve in clayey soil, the rate of OM decomposition was higher in sandy soil (Table 3) resulting in an equal P intake. NV supplied 32.1 kg ha\(^{-1}\) of P2O5 to both soils, which would supply most of the requirements for the sugarcane (Orlando Filho 1993; van Raij et al. 1997). Unlike K net balance, P was derived from soil mineralization and not
from vinasse, since the $P_2O_5$ input of NV and CV was only 2.0 and 3.3 kg ha$^{-1}$, respectively (Table 1).

The $P$ release in the system was 133% and 50% higher for the sandy and clay soils, respectively, which reflected the high OM mineralization rates in sandy soil (112.2%) and clayey soil (80.9%), as shown in Table 3. However, in this case, the available $P$ concentration would still be 13% higher in the clayey soil due to its reserve, indicating that the use of CV allowed greater $P$ availability, which is beneficial for soil fertility. The same trend was observed for the treatment with N addition. The amount of $P$ in clayey soil was 43% higher than in sandy soil, which was due to its natural reserve. In CV treatments with and without urea, the requirements of the sugarcane were met.

**pH**

Despite the vinasse acidity (pH 4.7), the increase in soil pH was temporary and resulted from $H^+$ complexation by the vinasse OM, as well as an increase in bases, such as $K^+$ (Reis & Rodella 2002). Camargo et al. (1983) found that the increase in pH observed in soil treated with vinasse was temporary, with a tendency to return to the original pH values. This occurrence was due to the release of $H^+$ ions, associated with carboxylic acids, phenolics, tertiary alcohols and sulfates, as a result of OM decomposition and protons released during the nitrification process.

**Electrical conductivity**

The CV addition increased the salt concentration in sandy soil, despite the fact that both concentrations of vinasse were equivalent in K. The addition of urea also substantially increased the EC values for sandy soil. This was not observed for clayey soil, reflecting its resistance to change, as this soil retains more ions in its mineralogy compared with sandy soil and, thus, fewer salts were present in solution.

Camargo et al. (1983) studying the physical-chemical characteristics of a soil that received vinasse for a long time, attributed the elevation of the EC values, in large part, to the increase in K concentration. However, the vinasse effects on the soils, salinity level are quite variable, depending on the type of vinasse, quantity and periodicity of application (Mazza 1985).

**Organic matter**

An increase in 0.2% in OM in sandy soil treated with NV resulted in 74.8% C-CO$\text{}_2$ release (Table 3). The remaining 15% of the C was used as a reserve for soil native OM. For the sandy soils treated with CV + urea and CV, there was a 0.1–0.2% reduction in OM concentration (Table 4), reflecting total waste and OM decomposition similar to control soil, with a small percentage of soil native OM, characterized by a priming effect.

As shown in Table 4, there was a 0.3% increase in OM concentration for clayey soil for the CV treatments, reflecting the same rates of decomposition (80–90%) and C-CO$\text{}_2$ losses, with the remaining OM (10–20%) associated with soil native OM. For NV treatment, the increase in inorganic matter concentration was 0.5%, reflecting the lower decomposition rate, which was still about 40% of the OM associated with the soil OM pool.

**Sulphate**

The inputs of S-SO$\text{}_4^{2-}$ were close to those found in the sandy soil treated with NV and CV, showing the low concentrations found in the control soil and dependency on addition of sulfate via vinasse or another chemical source in this type of soil. A 52% increase in soil S-SO$\text{}_4^{2-}$ concentration was only observed when urea was added to the CV (Table 4). The increase in the content of S-SO$\text{}_4^{2-}$ was mainly due to the mineralization of the vinasse OM and not of the soil (Abreu Junior et al. 2002).

For clayey soil, the same S-SO$\text{}_4^{2-}$ concentrations for both NV and CV treatments indicated that the waste decomposition did not affect the S-SO$\text{}_4^{2-}$ concentration. In the soil treated with CV + urea, there was a 17.5% increase in the S-SO$\text{}_4^{2-}$ concentration compared with CV treatment, also observed for the sandy soil. The addition of urea promoted different decomposition of the added OM, and the one present in this soil and/or the soil remaining material from this decomposition promoted differences in the adsorption of S-SO$\text{}_4^{2-}$. Hamer et al. (2009) observed a higher relative abundance of fungi after urea fertilization in pasture that induced positive priming effects.

**CONCLUSIONS**

The respirometry test confirmed the OM decomposition present in vinasses in both soils and positive priming effect was observed in CV + urea in the sandy soil. This maybe occurred as a result of microbiota stimulation with the fresh OM addition to the system. The period for total decomposition of vinasse OM was lower in clayey soil, suggesting the quicker release of nutrients to the sugarcane culture.
According to the results of vinasse characterization in relation to K and the decomposition rates of this waste, the tested hypothesis was confirmed about K pseudo-total being released into the soil solution. This confirmation may indicate the possibility of overestimation in the vinasse application rates in cultivated soils. However, studies involving K mobility into soil are needed to predict if the simplification in K determination is causing environmental contaminations.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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