# Nutrient content and accumulation in sugarcane under mineral fertilization and high doses of vinasse

### Teor e acúmulo de nutrientes em cana-de-açúcar sob adubação mineral e doses elevadas de vinhaça

Monalisa Soares Costa<sup>1</sup>\*; Mário Monteiro Rolim<sup>2</sup>; Gerônimo Ferreira da Silva<sup>2</sup>; Djalma Euzébio Simões Neto<sup>3</sup>; Elvira Maria Regis Pedrosa<sup>2</sup>

#### Highlights \_

Effect of vinasse doses up to 1200 m<sup>3</sup> ha<sup>-1</sup> on nutrient absorption by sugarcane. Interaction between nutrients at high doses of vinasse in the nutrition of sugarcane. Tolerance of sugarcane to vinasse. Nutritional supply of sugarcane with application of vinasse.

#### Abstract

Vinasse, a waste from ethanol production, has been widely used as a means of fertigation in sugarcane. However, high dose limits application at and its effects on nutrient absorption and accumulation by the crop are not known. The aim of this study was to evaluate and compare, during the initial development of sugarcane, the effect of mineral fertilization and high doses of vinasse on the macronutrient and sodium contents of the top visible dewlap (TVD) leaf and their respective accumulations in the aerial part of the crop. The experiment was carried out in 100-dm<sup>3</sup> pots filled with Argisol. The treatments consisted of five doses of vinasse (0, 150, 300, 600 and 1200 m<sup>3</sup> ha<sup>-1</sup>) and an additional treatment with no vinasse and mineral fertilizer (500 kg ha<sup>-1</sup> of the NPK 14-24-18 formulation). The levels of nitrogen, phosphorus, potassium, calcium, magnesium and sodium were evaluated in the TVD leaf at 120 days after planting and their accumulation in the aerial part of the plant at 210 days. Data were subjected to analysis of variance and compared using regression analysis and orthogonal contrasts. The vinasse doses applied did not adequately nourish the crop in macronutrients. Mineral fertilization provided higher N and P levels. The highest doses of vinasse provided higher levels of K, Ca and Mg and larger accumulations of N, P, K and Na, but were detrimental to the accumulation of Ca. The descending order of accumulation of macronutrients and sodium with mineral fertilization was P> Ca> N> K> Mg> Na. For the application of vinasse, the descending order was K> P> Ca> N> Na> Mg. In the absence of mineral fertilization and vinasse, the descending order was P> Ca> K> N> Mg> Na.

Key words: Saccharum officinarum. Mineral nutrition. Residue use.

\* Author for correspondence

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<sup>&</sup>lt;sup>1</sup> PhD in Agricultural Engineering, Rural Federal University of Pernambuco, UFRPE, Recife, PE, Brazil. E-mail: monalisa\_ sc@hotmail.com

<sup>&</sup>lt;sup>2</sup> Profs. PhD, Agricultural Engineering Department, UFRPE, Recife, PE, Brazil. E-mail: mariorolim10@uol.com.br; geronimo. silva@ufrpe.br; elvira.pedrosa@ufrpe.br

<sup>&</sup>lt;sup>3</sup> PhD, Coordinator of the Carpina Sugarcane Experimental Station, UFRPE, Carpina, PE, Brazil. E-mail: djalmasneto@ hotmail.com

#### Resumo \_

A vinhaca, resíduo da produção do etanol, vem sendo amplamente utilizada como meio de fertirrigação da cana-de-açúcar, entretanto, não se conhece os limites da aplicação em altas doses e seus efeitos na absorcão e acúmulo de nutrientes pela cultura. Objetivou-se avaliar e comparar, durante o desenvolvimento inicial da cana-de-acúcar, o efeito da adubação mineral e doses elevadas de vinhaca sobre o teor de macronutrientes e de sódio na folha diagnóstico e seus respectivos acúmulos na parte aérea da cultura. O experimento foi conduzido em vasos de 100 dm<sup>3</sup> preenchidos com um Argissolo. Os tratamentos consistiram de cinco doses de vinhaça (0, 150, 300, 600 e 1200 m<sup>3</sup> ha<sup>-1</sup>) e um tratamento adicional com ausência de vinhaça e adubação mineral (500 kg ha<sup>-1</sup> da formulação NPK 14-24-18). Os teores de nitrogênio, fósforo, potássio, cálcio, magnésio e sódio foram avaliados na folha diagnóstico aos 120 dias após o plantio e seus acúmulos na parte aérea da planta aos 210 dias. Os dados foram submetidos à análise de variância e comparados mediante análise de regressão e contrastes ortogonais. As doses de vinhaça aplicadas não nutriram adequadamente a cultura em macronutrientes. A adubação mineral propiciou a obtenção de maiores teores de N e P. As doses mais elevadas de vinhaca proporcionaram maiores teores de K, Ca e Mg e, maiores acúmulos de N, P, K e Na, porém, desfavoreceram o acúmulo de Ca. A ordem decrescente de acúmulo dos macronutrientes e do sódio com a adubação mineral foi: P > Ca > N > K > Mg > Na. Para a aplicação de vinhaça a ordem decrescente foi: K > P > Ca > N > Na > Mg. Já para as ausências de adubação mineral e de vinhaça a ordem decrescente foi: P > Ca > K > N > Mg > Na.

Palavras-chave: Saccharum officinarum. Nutrição mineral. Aproveitamento de resíduo.

#### Introduction \_\_\_\_

Sugarcane is an important crop for the economy and development of Brazil, as, among other factors, it is a raw material for the processing of ethanol, a biofuel that has been widely used in the country and worldwide. However, the ethanol industry generates vinasse, a waste made up of about 93% water and 7% total solids. The solid fraction is known to consist of 75% organic matter and 25% minerals, among which K predominates (Pazuch et al., 2017).

Vinasse is commonly used as a means of meeting part of the nutritional requirement of sugarcane. Conversely, the compound has an acid pH, high electrical conductivity (Christofoletti, Escher, Correia, Marinho, & Fontanetti, 2013) and disproportional distribution of mineral nutrients in relation to the nutritional need of sugarcane, directly affecting the ability of the plant to properly nourish. Thus, situations of deficiency or toxicity by a certain element may ensue due to nutrient imbalance.

Inadequate management of the amount of vinasse applied causes unfavorable chemical changes in the soil and compromises the development of sugarcane due to the high concentrations of K, Ca and Na present in the compound (Christofoletti et al., 2013; Ortegón, Arboleda, Candela, Tamoh, & Valdes-Abellan, 2016; Garcia, Souza, Souza, Christofoletti, & Fontanetti, 2017). This nutritional imbalance causes saturation of the soil with these nutrients and, consequently, antagonistic relationships, especially those of K with Ca and Na (Aquino, Medina, Brito, & Fonseca, 2015; Rhodes, Miles, & Hughes, 2018), ultimately compromising the nutritional balance of the crop.

According to E. C. A. Oliveira et al. (2010), sugarcane demands K predominantly over Ca, N, Mg and P. In this respect, a recommended fertilization strategy for the crop with vinasse doses of up to 300 m<sup>3</sup> ha<sup>-1</sup> can meet its nutritional requirements (especially of potassium), promote stalk production and reduce the amount of mineral fertilizer to be applied (Barbosa, Arruda, Pires, Silva, & Sakai, 2012). Nonetheless, no consolidated information exists in the literature regarding the application of vinasse doses higher than 300 m<sup>3</sup> ha<sup>-1</sup> on nutrient absorption by sugarcane. Moreover, studies comparing the application of mineral fertilizer versus vinasse doses - high doses, mainly - on the nutritional behavior of the crop are still incipient.

Therefore, research investigating the management of the vinasse application to the soil for the mineral nutrition in sugarcane is warranted. Likewise, studies comparing mineral fertilization with use of vinasse for fertilizing the crop are of great relevance, as they can provide important information regarding the possibility of using vinasse in the partial or total replacement of mineral fertilization.

Among the methods used to evaluate and compare the effects of application of mineral fertilizer and vinasse doses on the initial development of sugarcane is the nutritional status of the crop, which is determined evaluating the top visible dewlap (TVD) leaf and the amount of nutrients accumulated in the aerial part of the plant. According to Grangeiro et al. (2011), these data are essential, as they can allow for increased efficiency of fertilization programs and the adequate supply of nutrients in a readily assimilable form. In this way, the crop can achieve the maximum productive capacity, especially when this is associated with nutritional analysis of the plants in a given moment of their cycle.

This study proposes to examine and compare the effect of mineral fertilization and high doses of vinasse on the macronutrient and sodium contents of the TVD leaf and their respective accumulations in the aerial part of the crop during sugarcane initial development.

#### Materials and Methods \_\_\_\_\_

The experiment was carried out in pots with volume capacity of 100 dm<sup>3</sup>, in a protected environment located at 8°01'05" S and 34°56'48" W, at altitude of 6.49 m. The soil used for filling the pots was an abrupt Dystrophic-Cohesive Yellow Argisol with a sandy loam texture, collected at 7°51'13" S and 35°14'10" W, in the municipality of Carpina, PE, Brazil, in a region of coastal plateau soils. Chemical characterization of the soil followed before the implementation of the experiment is described in Table 1. Vinasse was collected in the rural area of Lagoa de Itaenga, in the "Mata Norte" region of the state of Pernambuco, Brazil, in the pipeline from the distillery to the distribution pond. The vinasse chemical characterization, performed immediately after collection, is shown in Table 2.

### Table 1Chemical attributes of the Argisol before application of vinasse

Parameter	Unit	Result 0-20 cm	Classification*	Result 20-40 cm	Classification*
рН		5.80	Good	5.60	Good
Phosphorus	mg dm⁻³	8.00	Very low	5.00	Very low
Potassium	cmolc dm <sup>-3</sup>	0.09	Low	0.06	Low
Calcium	cmolc dm <sup>-3</sup>	1.75	Medium	1.25	Medium
Magnesium	cmolc dm <sup>-3</sup>	0.75	Medium	0.75	Medium
Sodium	cmolc dm <sup>-3</sup>	0.08	Medium	0.07	Medium
CEC	cmolc dm <sup>-3</sup>	5.80	Medium	5.60	Medium
Effective CEC	cmolc dm <sup>-3</sup>	2.75	Medium	2.25	Medium
Potential acidity	cmolc dm <sup>-3</sup>	3.13	Medium	3.46	Medium
SB	cmolc dm <sup>-3</sup>	2.70	Medium	2.10	Medium
Base saturation	%	46.00	Medium	38.00	Medium
Organic matter	%	2.45	Medium	2.05	Medium
ESP	%	1.37	Non-sodic	1.25	Non-sodic
Sand	%	75.50		73.85	
Silt	%	19.80	Sandy-loam	21.84	Sandy-loam
Clay	%	4.70		4.31	

\*Classification based on Ribeiro, Guimarães and Alvarez (1999); pH in water (1:2.5); Phosphorus extracted by Mehlich<sup>-1</sup> (double acid extractor: H2SO4 0.0125 mol L<sup>-1</sup> + HCl 0.05 mol L<sup>-1</sup>); Potassium and sodium extracted with HCl 0.05 mol L<sup>-1</sup>; Calcium and magnesium extracted with KCl 1 mol L<sup>-1</sup>; Potential acidity (H + Al) extracted with 0.5 M calcium acetate and pH 7.0; CEC = cation-exchange capacity; SB = sum of exchangeable bases; ESP = exchangeable sodium percentage.

To fill each pot, a layer of gravel #0 was added to the bottom and geotextile fabric was laid on top of it. Each pot was filled with 77 dm<sup>3</sup> of soil in a 55-cm deep layer arranged so that the lower half of the pot contained the portion collected at the depth of 20-40 cm and the upper half contained the soil collected at the depth 0-20 cm, with a porosity of 46.15%, aiming to maintain an apparent density close to  $1.4 \text{ g cm}^{-3}$ .

#### Table 2

#### Chemical attributes of the Argisol before application of vinasse

Parameter	Supply water	Vinasse
рН	6.3	4.2
Electrical conductivity (dS m <sup>-1</sup> )	0.128	15.06
Calcium (mg L <sup>-1</sup> )	0.9	888.3
Magnesium (mg L <sup>-1</sup> )	0.6	395.3
Sodium (mg L <sup>-1</sup> )	5.1	729.1
Potassium (mg L <sup>-1</sup> )	2.5	1053
Total nitrogen (mg L <sup>-1</sup> )	-	51.8
Total phosphate (mg L <sup>-1</sup> )	-	6.78
Carbon (mg L <sup>-1</sup> )	-	1,032.5
Total solids (mg L <sup>-1</sup> )	-	22,368
Biochemical oxygen demand (BOD <sub>5.20</sub> ) (mg L <sup>-1</sup> )	-	12,300
Chemical oxygen demand (mg L <sup>-1</sup> )	-	27,250

The experiment was laid out in a completely randomized design with six treatments and six replicates. The treatments were represented by five vinasse doses, namely, 0 (D0), 150 (D150), 300 (D300), 600 (D600) and 1200 (D1200) m<sup>3</sup> ha<sup>-1</sup>, and an additional treatment corresponding to mineral fertilization (MF). These doses were chosen based on the dose of 300 m<sup>3</sup> ha<sup>-1</sup>, which is

recommended as the adequate dose for the potassium requirement of a sugarcane cycle.

The amounts of macronutrients and sodium present in each vinasse dose were estimated for the equivalent in kg ha<sup>-1</sup> and are described in Table 3. Vinasse was applied manually, with the quantities being calculated in proportion to the surface area of the pots, at 20 days before sugarcane was planted.

#### Table 3

Amounts of nitrogen (N), phosphorus ( $P_2O_5$ ), potassium ( $K_2O$ ), calcium (Ca), magnesium (Mg) and sodium (Na) determined for each of the vinasse doses applied

Dose	Ν	P <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	Ca	Mg	Na
m³ ha⁻¹			kg ha	a <sup>-1</sup>		
150	7.77	1.01	189.00	133.24	59.29	109.36
300	15.54	2.03	378.00	266.49	118.59	218.73
600	31.08	4.07	756.00	532.98	237.18	437.46
1200	62.16	8.14	1512.00	1065.96	474.36	874.92

Mineral fertilization consisted of a 14-24-18 NPK dose corresponding to 500 kg ha<sup>-1</sup> considering the upper area of the pot for proportional dosing. Ammonium sulfate was used as the N source; single superphosphate as the  $P_2O_5$  source; and potassium chloride as the  $K_2O$  source. These were applied based on soil analysis carried out before the start of the experiment and on the fertilization recommendation for the state of Pernambuco by Cavalcanti et al. (2008). All mineral fertilization was applied before the planting of sugarcane, with no need for fertilization after the beginning of the crop cycle.

The sugarcane cultivar evaluated was RB867515, with four billets planted in each pot, each containing one bud. After germination, thinning was performed to leave only three billets. The crop was irrigated manually and in an equal amount for all plots, following a 2-day irrigation shift, according to the need of the crop, and maintaining soil moisture at field capacity. Throughout the experimental period (210 days), about 160 dm<sup>3</sup> of water were applied to each pot, that is, 851 mm.

The levels of N, P, K, Ca, Mg and Na were analyzed on the top visible dewlap (TVD) leaf (+1) at 120 days after planting. The +1 leaf was considered to be the first leaf with a visible ligule. In removing this leaf for analysis, the extremities were discarded and only the middle third part was sent for laboratory analysis. The central nerve was also discarded (Martinez, Carvalho, & Souza, 1999). At 210 days, macronutrient (N, P, K, Ca and Mg) and Na accumulation in the aerial part of the plant (stem, tip and leaves) was quantified. Accumulation was determined by multiplying each of the levels of nutrients found by the amount of dry biomass accumulated in the respective aerial part of the plants.

To determine both the content (120 days) and accumulation (210 days) of macronutrients and Na by the crop, all the plant material was taken to the laboratory and washed with deionized water. Subsequently, the material was packed in paper bags duly identified with the respective treatments and dried in a forced-air oven (65 °C) until a constant dry biomass was achieved. Next, the dry material was processed in a Wiley mill with a 2-mm sieve and placed in closed containers for later quantification of macronutrients and Na.

Macronutrients (P, K, Ca and Mg) and Na were extracted from plant tissue in a closed system using a microwave oven as a source of heat and concentrated nitric acid ( $HNO_3$ ) to digest the dry matter. For N, sulfuric digestion was performed in a digester block according to the methodology proposed by F. C. Silva (2009). Total N was quantifiedby the Kjeldahl steam distillation method; K and Na by flame photometry; P by the colorimetric molybdovanadate method; and Ca and Mg were determined by atomic absorption spectrophotometry.

The obtained data were subjected to analysis of variance by the F test. When significant effects were detected, regression analysis (vinasse doses) was performed at 5% probability. An analysis was also carried out using orthogonal contrasts to compare mineral fertilization versus vinasse doses (MF × vinasse (D150 to D1200)); mineral fertilization versus non-application of vinasse (MF × D0); mineral fertilization versus vinasse dose of 300 m<sup>3</sup> ha<sup>-1</sup> (MF × D300); mineral fertilization versus vinasse dose of 1200 m<sup>3</sup> ha<sup>-1</sup> (MF × D1200); and non-application of vinasse versus all vinasse doses (D0 × vinasse (D150 to D1200)).

#### Results and Discussion \_\_\_\_\_

#### Nutritional status

The levels of the macronutrients evaluated in the TVD (+1) leaf were significantly influenced by the treatments. However, the same was not true for Na (Table 4). Of the evaluated contrasts (Table 5), for N, a difference was only detected for MF versus the vinasse dose corresponding to  $300 \text{ m}^3 \text{ ha}^{-1}$  (D300), with average leaf N contents of 15.9 g kg<sup>-1</sup> obtained

with the application of MF and 11.8 g  $kg^{-1}$  with the application of D300 (Table 4).

At 120 days after planting, the vinasse doses applied were not sufficient to provide an N content considered appropriate in the TVD leaf (Figure 1a). The highest vinasse dose applied (1200 m<sup>3</sup> ha<sup>-1</sup>) provided a leaf N content of 12.9 g kg<sup>-1</sup>, nearing the lower limit considered adequate for the crop (12 to 25 g kg<sup>-1</sup>) by F. C. Silva (2009).

#### Table 4

Effect of mineral fertilizer (MF) and vinasse doses (D0 to D1200) on the levels of nitrogen (N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg) and sodium (Na) in the top visible dewlap leaf 120 days after planting

Nutrient		Treatment							Reference
(g kg⁻¹)	MF	D0	D150	D300	D600	D1200	F	P-value <sup>1</sup>	values <sup>3</sup>
Ν	15.90±1.14 <sup>2</sup>	14.88±1.14	10.64±0.81	11.84±0.81	12.45±0.81	12.75±0.81	3.83	0.0108	18-25
Р	1.47±0.09	1.36±0.09	1.28±0.07	0.96±0.07	1.05±0.07	1.08±0.07	6.20	0.0008	1.5-3.0
К	12.85±1.15	12.25±1.15	12.60±0.81	14.09±0.81	14.74±0.81	18.15±0.81	8.57	<0.0001	10-16
Са	0.84±0.21	1.26±0.21	1.06±0.15	0.59±0.15	1.35±0.25	2.00±0.15	10.00	<0.0001	2.0-8.0
Mg	0.73±0.05	0.84±0.05	0.75±0.03	0.65±0.05	0.86±0.05	1.0±0.05	11.78	<0.0001	1.0-3.0
Na	1.70±0.42	1.67±0.42	2.63±0.30	1.75±0.30	1.53±0.30	1.52±0.30	1.94	12.47	-

<sup>1</sup>Significant results at 5% (p-value <0.05). <sup>2</sup>Mean ± standard error. F: F value calculated from analysis of variance. <sup>3</sup>Values considered adequate in the top visible dewlap leaf, in g kg<sup>-1</sup>, according to F. C. Silva (2009).

#### Table 5

Orthogonal contrasts comparing the effect of mineral fertilizer (MF) and vinasse doses (D0 to D1200) on the levels of nitrogen (N), phosphorus (P), potassium (K), calcium (Ca) and magnesium (Mg) in the top visible dewlap leaf of the sugarcane crop

Contract			F-value		
Contrast	N	Р	K	Ca	Mg
MF × Vinasse (D150 to D1200)	3.11 <sup>NS</sup>	9.00*	3.15 <sup>NS</sup>	3.44 <sup>NS</sup>	3.27 <sup>NS</sup>
MF × D0	2.30 <sup>NS</sup>	11.42*	48.91*	6.29*	6.76*
MF × D300	4.70*	18.85*	4.72*	8.68*	10.17*
MF × D1200	0.40 <sup>NS</sup>	0.60 <sup>NS</sup>	0.37 <sup>NS</sup>	3.03 <sup>NS</sup>	2.63 NS
D0 × Vinasse (D150 to D1200)	0.00 <sup>NS</sup>	1.27 <sup>NS</sup>	67.52*	39.08*	40.08*

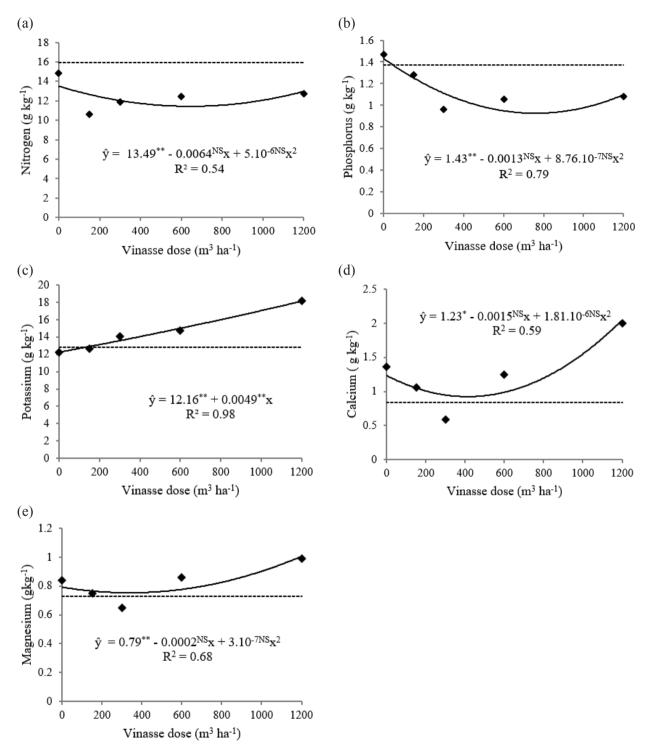
\*Significant (p<0.05); <sup>№</sup>Not significant (p>0.05).

Given the insignificance of the contrast between MF and D1200 and the proximity of results of the N levels in the leaf obtained with the application of the doses of D600 (12.8g kg<sup>-1</sup>) and D1200 (12.9 g kg<sup>-1</sup>) (Figure 1a), it can be inferred that the dose of 600 m<sup>3</sup> ha<sup>-1</sup> complemented with mineral fertilizer can be used as a strategy for producers who have this waste available on their farms, which will bring economic and environmental benefits.

The inadequate supply of N for sugarcane, demonstrated by N levels in TVD leaf lower than the adequate for the crop, could be an after reflect of the low concentration of N provided by the vinasse. According to Meyer (2013), along with K, N is the nutrient most absorbed by sugarcane. Moreover, N is responsible for 40% of the plant dry biomass by in the initial growth phase (Franco et al., 2011).

Another factor that may have contributed to the N levels being below the ideal in the leaf was the low concentration of P in vinasse, which leads to a low concentration of the element in the soil and, consequently, inadequate supply of P to the plants (Figure 1b). The P concentration found in vinasse was lower than that of N, and because P and N act synergistically (Malavolta, 2006), a higher concentration of P in vinasse and, consequently, in the soil, possibly contributed to a better use of the available N in vinasse and a higher N content in the TVD leaf of the crop. The test of contrasts performed for the P content in the TVD leaf (Table 5) revealed significant differences for the contrasts of MF × vinasse doses (D150 to D1200), MF × D0 and MF × D300.





**Figure 1.** Concentrations of nitrogen (a), phosphorus (b), potassium (c), calcium (d) and magnesium (e) in the top visible dewlap leaf (+1) of sugarcane according to the application of vinasse doses (D0 to D1200) and mineral fertilizer at 120 days after planting.

\*\*Significant at 1% probability; \*Significant at 5% probability; <sup>NS</sup>Not significant at 5% probability.

The obtained results suggest that the vinasse doses applied were not sufficient to provide P levels comparable to those obtained with mineral fertilization, which generally supplied higher levels of the nutrient to the leaf (Figure 1b). These findings are possibly due to the low concentration of phosphate in vinasse (6.8 mg L<sup>-1</sup>) (Table 2), which was reflected in the low supply of the nutrient to the soil and, consequently, in the lower leaf levels of the nutrient, as compared with mineral fertilization.

The vinasse doses applied were not sufficient to provide P content in the TVD leaf deemed adequate for the crop. According to F. C. Silva (2009), the range of adequate P content is between 1.5 and 3.0 g kg<sup>-1</sup>. The N and P levels in the TVD leaf of sugarcane obtained in the present study corroborate those obtained by J. Silva, Cazetta and Togoro (2015), who examined the effects of the vinasse application to the soil on the mineral nutrition of sugarcane and did not find significant increases in the N or P concentrations in the TVD leaf. Likewise, Carvalho, Andreotti, Buzetti and Carvalho (2013) evaluated the yield of sugarcane as a function of the use of plaster and vinasse and observed that the application of up to 200 m3 ha<sup>-1</sup> of vinasse did not cause a significant change in N or P concentrations in the leaf.

At 120 days, significant interaction effects of MF × D0, MF × D300 and D0 × vinasse dose (D150 to D1200) were found for K content (Table 5). The application of MF resulted in higher K content in the TVD leaf (12.9 g kg<sup>-1</sup>) as compared with no vinasse application (12.3 g kg<sup>-1</sup>) (Table 4). The application of the D300 dose provided higher content of the nutrient in the leaf (14.1 g kg<sup>-1</sup>) when compared with MF (12.9 g kg<sup>-1</sup>). When comparing only the application of vinasse doses, we observe that any level would provide higher K content in leaf than that achieved with non-application of the waste (Figure 1c). As also shown in Figure 1c, starting at the vinasse dose of 144.8 m<sup>3</sup> ha<sup>-1</sup>, the K content in leaf was already higher than that obtained with mineral fertilization.

The vinasse doses applied provided K levels in the TVD leaf considered adequate (10 and 16 g kg<sup>-1</sup>) by F. C. Silva (2009), except for the D1200, which caused a concentration of the nutrient (18.0 g kg<sup>-1</sup>) above the maximum recommended (Figure 1c). This result can be attributed to the high concentration of the nutrient (1053 mg L<sup>-1</sup>) in the composition of vinasse (Table 2).

Borges et al. (2015) evaluated the application of vinasse on the nutritional status and sugarcane yield in an organic cultivation system and found that K from vinasse applied at the dose of 600 m<sup>3</sup> ha<sup>-1</sup> increased the nutrient content in the TVD leaf of the crop by 44.5% in relation to mineral fertilization. According to Silveira, Vitusso and Medina (2015), the sugarcane plant accumulates most of the K present in the tissues during the first months of development. Coupling this with the fact that vinasse adds large amounts of K to the soil, the plant is able to absorb most of this available nutrient even if it is not needed, and from that period on it may exhibit physiological and morphological disorders caused by the excess of the nutrient itself.

For both the Ca and Mg contents in the TVD leaf, the test of contrasts (Table 5) indicated significant differences for MF × D0, MF × D300 and D0 × vinasse doses (D150 to D1200). The application of MF resulted in higher Ca (0.8 g kg<sup>-1</sup>) and Mg (0.7 g kg<sup>-1</sup>) contents in TVD leaf as compared with D300, which provided Ca and Mg contents of 0.6 and 0.7 g kg<sup>-1</sup>, respectively.

In the analysis of the isolated effect of vinasse doses on the leaf levels of Ca (Figure 1d) and Mg (Figure 1e), the doses applied were found to provide higher values of these nutrients in the plants than mineral fertilization.

The application of vinasse doses provided Ca and Mg contents in the TVD leaf below those indicated as adequate (2 to 8 g kg<sup>-1</sup> for Ca and 1 to 3 g kg<sup>-1</sup> for Mg) by F. C. Silva (2009), except for the D1200 dose, which resulted in a leaf Ca content (2.03 g kg<sup>-1</sup>) considered adequate for the crop (Figure 1d). These results can be attributed to the low concentrations of Ca (888.3 mg L<sup>-1</sup>) and Mg (395.3 mg L<sup>-1</sup>) (Table 2) in the composition of the applied vinasse, which, overall, resulted in leaf nutrient contents below the recommended for the crop.

Carvalho et al. (2013) examined the productive of sugarcane in response to the use of plaster and vinasse and observed a low concentration of Mg in the TVD leaf of the crop (0.78 g kg<sup>-1</sup>) following the application of 200 m<sup>3</sup> ha<sup>-1</sup> of vinasse. For Ca, the authors found content considered to be adequate for the crop (2.6 g kg<sup>-1</sup>). Borges et al. (2015) evaluated the application of vinasse, in an organic cultivation system, on the nutritional status and yield of sugarcane and found that the application of the 600 m<sup>3</sup> ha<sup>-1</sup> dose did not contribute to the adequate nutrition of the crop and that the Ca and Mg levels in the TVD leaf remained below recommended.

V. S. G. Silva et al. (2017) investigated the nutritional status of sugarcane varieties and found higher average K levels in the TVD leaf of the crop than Ca and Mg. According to Nascimento, Souza, Moreira and Moraes (2017), vinasse doses applied to the soil increase the K content of plants, but can reduce their Ca and Mg levels. The higher K contents in relation to Ca and Mg found in this study may be related to the low CEC of the roots shown by sugarcane, considering that monocotyledonous roots have low root CEC, preferring the absorption of monovalent cations such as K (Büll, Mello, Soares, & Boareto 1993). In view of this, adequately supplying the soil with Ca and Mg is of paramount importance to allow these nutrients to compete for binding sites in the root system of the crop, given the greater preference for the absorption of K, which has a competitive inhibition on the absorption of Ca and Mg.

## Macronutrient (N, P, K, Ca and Mg) and sodium (Na) accumulation in the aerial part

The test of orthogonal contrasts (Table 6) revealed a significant difference for MF × D0 on the accumulations of N, K, Mg and Na, with higher values obtained with the application of MF (Table 7). As can also be seen in Table 6, when MF is contrasted with D300, larger Ca accumulation (11.9 g kg<sup>-1</sup>) is obtained with the use of the D300 dose (Table 7). The contrast between D0 and vinasse doses (D150 to D1200) (Table 6) showed that the application of vinasse provides larger accumulations of N, P, K and Na (Table 7). As regards the accumulations of Ca and Mg, as observed for the levels of these nutrients in the TVD leaf of the crop (Table 4), the application of vinasse doses reduced their accumulations in comparison with the levels obtained with non-application of the waste (D0) (Table 7).

#### Table 6

Orthogonal contrasts comparing the effect of mineral fertilizer (MF) and vinasse doses (D0 to D1200) on the accumulation of nitrogen (N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg) and sodium (Na) in the aerial part of the sugarcane crop at 210 days after planting

Contract			F-value		
Contrast	N	K	Ca	Mg	Na
MF × Vinasse (D150 to D1200)	1.87 <sup>NS</sup>	0.02 <sup>NS</sup>	0.12 <sup>NS</sup>	0.41 <sup>NS</sup>	0.00 <sup>NS</sup>
MF × D0	17.11*	32.38*	3.35 <sup>NS</sup>	4.43*	19.37*
MF × D300	0.50 <sup>NS</sup>	0.38 <sup>NS</sup>	9.21*	0.86 <sup>NS</sup>	0.04 <sup>NS</sup>
MF × D1200	2.77 <sup>NS</sup>	0.61 <sup>NS</sup>	0.29 <sup>NS</sup>	0.07 <sup>NS</sup>	0.00 <sup>NS</sup>
D0 × Vinasse (D150 to D1200)	19.70*	71.17*	10.39*	16.29*	43.16*

\*Significant (p<0.05); <sup>NS</sup>Not significant (p>0.05).

#### Table 7

Effect of mineral fertilizer (MF) and vinasse doses (D0 to D1200) on the accumulations of nitrogen (N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg) and sodium (Na) in the aerial part of the sugarcane crop at 210 days after planting

Nutrient	nt Treatment							
(g kg⁻¹)	MF	D0	D150	D300	D600	D1200	F	P-value <sup>1</sup>
Ν	$7.87 \pm 1.10^{2}$	5.27±1.10	6.23±0.78	6.23±0.78	7.25±0.78	10.87±0.78	5.62	0.0014
Р	14.43±2.59	14.16±2.59	14.90±1.83	14.26±1.83	15.41±1.83	15.45±1.83	0.08	0.9947
K	7.03±2.54	9.84±2.54	11.08±1.80	11.76±1.80	10.87±1.80	27.58±1.80	14.86	<0.0001
Са	9.62±4.15	12.81±4.15	11.88±2.94	18.45±2.94	7.64±2.94	3.50±2.94	8.28	0.0001
Mg	0.83±0.15	0.78±0.15	0.59±0.10	0.61±0.10	0.67±0.10	1.16±0.10	4.06	0.0082
Na	0.60±0.14	0.61±0.14	0.64±0.10	0.58±0.10	0.66±0.10	1.40±0.10	9.13	<0.0001

<sup>1</sup>Results significant at 5% (p-value <0.05). <sup>2</sup>Mean ± standard error. F: F value calculated in analysis of variance.

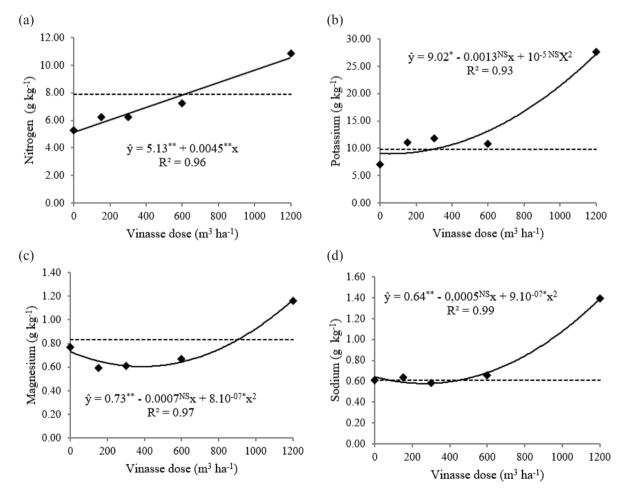
Nitrogen accumulation increased linearly, by 0.0045 g kg<sup>-1</sup>, with each additional unit of vinasse (Figure 2a). The highest accumulation observed for this macronutrient was 10.54 g kg<sup>-1</sup>, which was estimated with the application of the vinasse dose corresponding to 1200 m<sup>3</sup> ha<sup>-1</sup>, representing a 51.2% percentage increase in relation to D0.

These results highlight the need for nitrogen fertilization as a complement to the application of vinasse, regardless of the dose applied. In this respect, Schultz, Lima, Pereira and Zonta (2010) recommended N dose of up to 80 kg ha<sup>-1</sup> to optimize the yield of sugarcane when fertigated with vinasse.

Phosphorus accumulation by the crop was not significantly influenced by any of the evaluated treatments (Table 7). These results show that even though vinasse has a low P concentration, the amount supplied to the plants by the waste was sufficient to provide an accumulation analogous to that obtained with the application of mineral fertilizer. In addition, the low requirement of the macronutrient by the crop may have contributed to these results, since, as described by E. C. A. Oliveira et al. (2010), P is required in the lowest amount among the macronutrients essential to sugarcane.

Considering the accumulation of K as a function of the vinasse doses applied (Figure 2b), any of the evaluated doses was found to provide a larger accumulation of the nutrient than MF. Also according to Figure 2b, there was an increasing accumulation of the nutrient with the application of doses from 235.1 m<sup>3</sup>

ha<sup>-1</sup>. Given these results, it is assumed that the presence of high concentrations of vinasse in the soil may have caused the sugarcane to absorb more K than its actual requirement for maximum yield (luxury uptake). In this respect, Oliveira, Medeiros, Freire, Simões and Oliveira (2016) described that when present in higher concentrations in the soil solution, the high concentrations of K in vinasse associated with the preference of the crop to absorb the nutrient can contribute to the achievement of more marked accumulations of K in the plant.



**Figure 2.** Accumulation of nitrogen (a), potassium (b), magnesium (c) and sodium (d) in the aerial part of the sugarcane crop according to the application of mineral fertilizer and vinasse doses (D0 to D1200) at 210 days after planting.

----- Mineral fertilizer; Means of vinasse doses

\*\*Significant at 1% probability; \*Significant at 5% probability; <sup>NS</sup>Not significant at 5% probability.

According to Zörb, Senbayram and Peiter (2014), luxury uptake associated with high availability of K in the soil occurs when there are high concentrations of K in the tissues. Besides the physiological need of the plant, K absorption can occur through the efficient mechanisms of absorption and distribution throughout the cells and the plant, with the cytoplasm and vacuole being the compartments used in the plant to store the absorbed nutrient (Ahmad & Maathuis, 2014). In addition to this, the cultivar evaluated in this study, RB867515, also has high affinity for the nutrient (R. I. Oliveira et al., 2016).

For Ca accumulation, there was no difference in the evaluated contrasts between MF and vinasse doses (D150 to D1200) (Table 6). However, among the applied doses of the waste, D600 and D1200 provided less accumulation of the nutrient by the crop than the other tested doses (D0, D150 and D300) (Table 7).

An antagonistic interaction was observed between K and Ca. These cations are known to possibly compete for the same absorption sites; however, sugarcane has preference for K. Malavolta (2006) stated the antagonism between K and Ca, as well the advantage of K because it is monovalent and has lower degree of hydration, in addition to being more mobile in the soil. Thus, under high amounts of K, the plant absorbs less Ca, as was observed in D600 and D1200.

Rhodes et al. (2018) evaluated the interaction of K with Ca in sugarcane and reported that the Ca content of the TVD leaf was suppressed by the presence of K in proportion to its availability in the soil. Also according to the authors, this is due to the relationship that K ions have with H ions, as

they share the same transmembrane transport protein, which facilitates their absorption by the plant to the detriment of Ca. In the present study, among the evaluated treatments, from D300, the supply of K was sufficient to allow the plant to absorb less Ca.

There was no difference for Mg accumulation when contrasting MF × vinasse doses (D150 to D1200) (Table 6). From the dose corresponding to 437.5 m<sup>3</sup> ha<sup>-1</sup>, there was an increase in the accumulation of the nutrient by the crop (Figure 2c). The contrast of MF × vinasse doses (D150 to D1200) did not reveal a significant effect on Na accumulation by the plants (Table 6). However, comparing the vinasse doses in relation to the accumulation of the element by the plants (Figure 2d), the application of vinasse doses as low as to or greater than 490 m<sup>3</sup> ha<sup>-1</sup> provided a larger accumulation of sodium in the plants than that obtained with the application of MF.

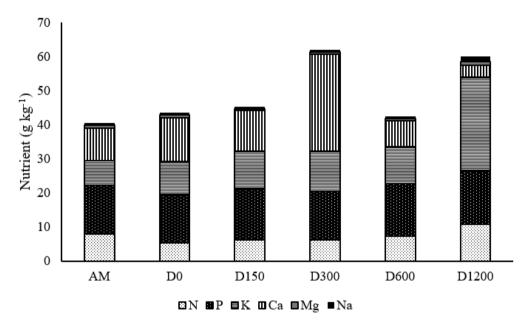
The accumulation of K and Na occurring at vinasse doses greater than 277 m<sup>3</sup> ha<sup>-1</sup> (Figures 2b and 2d, respectively) is explained by the fact that these ions are monovalent and, as such, compete with each other at the moment of absorption. In the present study, the K concentration in vinasse (1053 mg L<sup>-1</sup>) was 44.4% higher than its Na concentration (729.1 mg L<sup>-1</sup>), which may have contributed to the predominance of K over Na and, consequently, to the preference for the absorption of the nutrient over Na. In this regard, Benito, Haro, Amtmann, Cuin and Dreyer (2014) stated that the plant has preference for K absorption over Na and that this preference will depend on the concentration of Na in the medium in relation to the concentration of K.

Figure 3 shows the order of accumulation of nutrients according to each



treatment. As illustrated, P was the nutrient accumulated in largest quantity, despite the low concentration found in vinasse. Sugarcane started to accumulate more K than Ca from D600, whereas N, Mg and Na were the least

absorbed nutrients. Oliveira, Freire, Oliveira, Oliveira and Freire (2011) stated that maximum P and K accumulation in sugarcane occurs in the early stages of development.



**Figure 2.** Order of accumulation of macronutrients and sodium in sugarcane according to vinasse doses (D0, D150, D300, D600 and D1200) and mineral fertilizer (MF) at 210 days after planting.

#### Conclusions \_

The vinasse doses applied did not increase the leaf contents of macronutrients sufficiently to be considered enough to adequate cultivation of sugarcane;

The highest leaf contents of N and P in the crop, at 120 days, were obtained with mineral fertilization. For K, Ca and Mg, the highest levels were achieved with the application of the highest doses of vinasse (600 and 1200 m<sup>3</sup> ha<sup>-1</sup>);

Up to 210 days after planting, the application of the highest doses of vinasse (600 and 1200 m<sup>3</sup> ha<sup>-1</sup>) provided larger accumulations of N, P, K and Na. However, they did not favor the accumulation of Ca, whose maximum value was reached with the application of  $300 \text{ m}^3 \text{ ha}^{-1}$  of vinasse;

The descending order of macronutrients and Na accumulation at 210 days after planting by sugarcane under mineral fertilizer was P> Ca> N> K> Mg> Na; under application of vinasse, K> P> Ca> N> Na> Mg; and in the absence of mineral fertilization and vinasse, P> Ca> K> Ng> Na.



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