

## Application of organomineral fertilizers sourced from filter cake and sewage sludge can affect nutrients and heavy metals in soil during early development of maize

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### Abstract

This study determined the changes in soil nutrients, organic carbon, organic matter and heavy metals during the early development stages of maize fertilized with different sources and doses. Also, this study confirmed that the used biosolid in the formulation of organomineral fertilizer makes greater efficacy than the effects promoted by mineral or filter cake organomineral fertilizers already used in agricultural. The experiment was done in a greenhouse at the Federal University of de Uberlândia - Campus Umuarama. The experimental design was randomized block design, as a 2 x 5 + 2 factorial, containing two sources of organomineral fertilizer consisting of organic residues (biosolid and filter cake), five doses of organomineral fertilizer (60, 80, 100, 120 and 140% of P<sub>2</sub>O<sub>5</sub> recommendation for maize crop), a positive control with mineral fertilization (100%) and a negative one with no fertilization with four replications. The soil pH and contents of phosphorus, potassium, calcium, magnesium base saturation, base sum, micronutrients, organic carbon, organic matter and heavy metals were determined subsequently after plant removal, 65 days after sowing. The organomineral fertilizers based on biosolid and filter cake yielded increases in phosphorus, boron and manganese in the soil, especially the biosolid source. Regardless of the fertilizer organic matter source, soil phosphorus increased linearly with increasing doses of the sources. Doses equal or below the recommendation for maize (100% P<sub>2</sub>O<sub>5</sub>) were less effective in supplying potassium. The organomineral sources did not add heavy metals to the soil. Fertilizers based on biosolid can supply nutrients, such as phosphorus, boron and manganese at high doses, replacing mineral fertilization, while maintaining and improving soil fertility with no contamination by heavy metals.

**Keywords:** waste, recycling, *Zea mays* L.

**Abbreviations:** DAS\_ days after sowing, TOC\_ Total organic carbon.

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### Introduction

Corn cultivation (*Zea mays* L.) has become essential in a scenario that demands more and more high productivity, while growing the crop must respect the sustainability of the production chain.

Regarding plant nutrition, possible changes can be made through fertilization, maize crop is highly responsive to it. However, the demands for improvements and adjustments in nutrient supply by fertilizers, especially nitrogen, phosphorus and potassium, are still evident. These are the nutrients most extracted by the plant and are also the most lost by processes of leaching, volatilization and adsorption (Coelho; França, 2015). Faced with this reality, organomineral fertilizers represent an alternative to soften the problem of nutrient supply to plants. These fertilizers are formulated with organic sources enriched with mineral sources.

The organic matter present in organomineral fertilizers can exert soil conditioning in the long term, as it influences the

physical and chemical properties of the same through water retention, formation of aggregates, and increase in the cation exchange capacity and carbon stocks. In addition, it can increase nutrient levels as it enables the reduction of nitrogen and potassium leaching effects and fixation of phosphorus by iron and aluminum oxides (Benites et al., 2010; Sousa et al., 2012).

There is also the possibility of associating micronutrients with organic matter in the formulation of these technologies. This is another advantage of organomineral fertilizers, since the organic fraction of the soil rises and promotes a chelating effect on micronutrients, such as iron, manganese, copper, zinc and boron.

In the organomineral fertilizer composition, the organic fraction can be obtained from different sources, such as the filter cake that, commercially, is already used for the manufacture of this type of fertilizer. This residue has high

levels of organic matter, phosphorus, calcium and potassium, but its composition varies according to the type of soil, variety and maturation of the cane and the process of clarification of the broth (Fravet et al., 2010). In general, the phosphorus predominant in the filter cake is organic, which together with the nitrogen, by processes of mineralization and action of the microorganisms of the soil, are released slowly leading to high utilization by the plants (Almeida Júnior et al., 2011; Vazquez et al., 2015).

The technology of organomineral fertilizers still presents benefits related to socio-environmental aspects, since it allows the use of commonly used waste for this purpose, such as sewage sludge. With the incentive of the National Solid Waste Policy (Brazil, 2010), this type of fertilizer allows the adaptation of the sewage sludge destination, being an alternative that meets the demands for more sustainable productive processes (Benites et al., 2014).

The use of sewage sludge in agriculture is a practice frequently undertaken by countries such as Germany and France (Silva et al., 2006). In Brazil, this technology is still questioned due to the possibility of inadequate use causing risks to the environment and human health. Nevertheless, the proper use of urban waste as fertilizer represents an important alternative, both for the socio-environmental problem and for agriculture in the context of alternative fertilizer technologies (Nogueira et al., 2007; Resolution nº 375, 2006).

After the sanitation processes for later use in agriculture, the sewage sludge is renamed "Biosolids" (Bettio; Camargo, 2006; Melo; Marques, 2000; Nunes Júnior, 2008). Several studies with the biosolids carried out with the purpose of verifying their efficiency, verified their potential to recover degraded soils, improving their fertility (Bonini et al., 2015; Rocha et al., 2004).

Studies report the versatility of this residue to be used as a substrate constituent for seedlings of fruit and forest species (Trigueiro and Guerrini, 2003). Oliveira (2016) verified that organomineral fertilizers based on organic sewage sludge were efficient for sorghum cultivation. However, studies that verify the potential of biosolids as part of the organic matter of organomineral fertilizers are still incipient.

The possibility of using urban waste, such as biosolid in the constitution of the organic matrix of pelleted organomineral fertilizers, can meet the nutritional needs of crops in a more efficient, sustainable and economically viable way.

Thus, the objective of this work was to determine the macronutrient, micronutrient and heavy metal contents in soil cultivated with maize under fertilization with pelleted organomineral fertilizer based on sewage sludge and filter cake.

## Results and discussion

### *Macronutrients in soil after application of pelleted organomineral fertilizers*

The level of phosphorus (P) in the soil was increased with the application of organomineral fertilizers with biosolids and filter cake, compared to the additional treatments (Table 1). The organomineral based on biosolids at the doses of 80, 100, 120

and 140% of the recommended dose of  $P_2O_5$  resulted in greater availability of the nutrient, while for the filter cake similar results were found from the 100% dose, compared to the absence of mineral (Table 1).

The organomineral fertilizer with biosolids at doses 100, 120 and 140% and filter cake in the dose of 100 and 120% provided greater availability of P than that observed with the mineral source (Table 1).

Biosolids fertilizer applied at 80% of the recommended dose provided about 36% more phosphorus compared to no fertilization. While in relation to the 100% dose this same source raised the level of phosphorus by 39% more than that found when fertilized with exclusively mineral sources (Table 1).

The fact that the biosolids fertilizer provides more phosphorus than the fertilizer with filter cake in relation to the mineral fertilizer and absence, is justified by the biosolids having a higher concentration of  $P_2O_5$  (2.23%) and higher C/N ratio (28/1) than the filter cake (0.95%  $P_2O_5$  and 13.72/1 C/N ratio) (Supplementary table 3 and 4).

Some authors (Araújo and Machado, 2006; Novais and Smyth, 1999) affirm that dynamic application of organic residues, such as corral manure, constantly replenish the organic acids that are responsible for restricting the adsorption of P, maintaining the blockage of nutrient adsorption sites continuously. This effect is also influenced by the concentration of P of the residue, where it is observed that in concentrations below 0.2% of total P, the immobilization of the solution P becomes greater than the organic P mineralization.

Silva et al. (2010) stated that organomineral fertilizers provide phosphorus slowly and the solubilization is gradual during the development of the crop, because organic matter rich in humic substances has the property of increasing the availability of negative charges in the phosphate release region of organomineral fertilizers. In an experiment conducted by the same authors it was also possible to observe higher phosphorus release for those fertilizers with higher concentrations of  $P_2O_5$ .

Soil phosphorus levels at 65 DAS- days after sowing- varied as a function of increasing doses of organomineral fertilizers, regardless of the type of organic matter used in their manufacture. It was observed that the amount of P increased linearly as there was increase of the organomineral doses, in which every 1 kg of organomineral fertilizer with biosolid and filter cake applied, there was an increase of 0.0252 and 0.0203  $mg\ dm^{-3}$  of P in the soil (Figures 1 and 2).

Phosphorus results confirm the possibility of using filter cake in agriculture, specifically for maize cultivation, through the availability of phosphorus, mainly at high dosages. González et al. (2014) found that the filter cake enriched with natural phosphate and biofertilizers promoted an increase in the population of fungi and phosphate solubilizing microorganisms and that, in the short term, the addition of the filter cake enriched with natural phosphate contributed to the soluble phosphorus in the soil.

The levels of potassium (K) in the soil were also not influenced by organic sources and doses of organomineral fertilizers, however the nutrient contents were lower in relation to the mineral source when a dose equal to that recommended (120

kg ha<sup>-1</sup> of P<sub>2</sub>O<sub>5</sub>) with the biosolid source was used (Table 2). It was also observed that the lower than recommended dose (80% P<sub>2</sub>O<sub>5</sub>) for the two organic sources did not provide amounts of potassium as fertilization with mineral sources (Table 2).

Organic matter is able to retain nutrients such as potassium so that it is not lost by washing the soil profile by rainwater or irrigation. In this work, doses above the recommendation (100%) maintained potassium levels similar to those found by mineral fertilization, whereas the lower amounts of organic matter present in the lower doses of the two sources were not sufficient to maintain K levels in the soil (Table 2).

The low values of this nutrient in the doses of 80 and 100% of the recommended dose, for both sources, in comparison to the mineral fertilization, may have occurred due to the mineralization that possibly did not occur in the totality, or it can still be considered that the available potassium has been more absorbed and used by plants.

The fact that potassium is a monovalent cation makes it more easily substituted in soil retention processes than bivalent cations such as calcium and magnesium. Thus, the K<sup>+</sup> ion, often adsorbed as an outer-sphere complex in clay minerals and organic matter in the soil, is lost more in the environment under conditions of less organic matter (Meurer, 2006; Mosaic, 2016).

It is further emphasized that the similar behavior between the two organic sources, for both phosphorus and potassium, demonstrates that biosolids can be used in agriculture as well as filter cake.

#### ***Hydrogen potential (pH), calcium, magnesium, base saturation (V) and sum of bases (SB) in soil after application of pelletized organomineral fertilizers***

For the pH variable, it was observed that the soil used for the work was within the level considered adequate (Alvarez et al., 1999) (Table 3). In this way, it was not possible to observe changes in this characteristic in detriment of the sources and their doses.

The pH is highly correlated to the availability of other elements, such as calcium and magnesium, which also did not change according to the sources and doses applied. Although hydrated lime was used for sanitation, the dose of organomineral fertilizer is considered small to promote significant changes in the soil pH. As these nutrients did not differ with the sources of raw material remaining unchanged, the base saturation (V) and the sum of bases (SB) also remained constant and without significant changes (Table 3).

#### ***Micronutrients in soil after application of pelletized organomineral fertilizers***

Similar to that analyzed with the macronutrients presented, the levels of the micronutrients Copper (Cu), Iron (Fe), Zinc (Zn), Boron (Bo) and Manganese (Mn) in the soil were not influenced by organic matter sources and increasing doses. Not even when it was compared alone with the absence of fertilization and the mineral source (Table 4), except for boron and manganese (Tables 5 and 6, respectively).

For the micronutrients that did not obtain alterations, it is believed that the increments can be better visualized in the long term of application of the organomineral. Trannin et al. (2005) also emphasize the hypothesis that the effects of the use of organic wastes on agriculture may have clearer results in the long run. In the work conducted by these authors, an increase in P and other nutrients, such as Zn, Cu, Mn, Fe and Ni, was observed, especially after the second application, indicating the residual effect.

In general, boron levels in the soil were increased in relation to the control for both sources of organic residues, where from the 80% (96 kg ha<sup>-1</sup> P<sub>2</sub>O<sub>5</sub>) dose there was greater availability of boron (Table 5). These results are justified because organomineral sources have 0.1% boron in their composition, causing the high dosages to reflect differences in this nutrient in the soil.

The soil used in the work presented the boron content classified by Alvarez et al. (1999) as "very low" (less than 0.15 mg dm<sup>-3</sup>) and this condition allowed for micronutrient increases in soil solution, especially for higher doses of fertilizers, confirming the potential of organic biosolids residue in the organomineral to provide this micronutrient efficiently for maize.

The use of organomineral fertilizers gave results similar to conventional fertilization when applying the entire recommendation. Doses above that increased soil boron concentrations when compared to mineral fertilization, with emphasis on the biosolid source that starts to present this advantage from the dose of 120% of P<sub>2</sub>O<sub>5</sub> (59% more boron compared to mineral fertilization), while for the filter cake this occurred only at the highest dose (140% P<sub>2</sub>O<sub>5</sub>) (Table 5).

Regarding manganese, it was observed a greater availability in relation to the control and mineral fertilization, for 80 and 120% of the recommended dose for both sources; being that the greater dose for the source with filter cake also provided an increase for this nutrient (Table 6).

The initial levels of manganese in the soil were found to be "medium" by Alvarez et al. (1999), and with the application of organomineral fertilizers, there was an increase of this micronutrient to a lower dose at the source with biosolids (80 and 120% P<sub>2</sub>O<sub>5</sub>), while with the filter cake a higher dose was required (80 and 140% P<sub>2</sub>O<sub>5</sub>) for the elevation of manganese levels in the soil (in relation to the mineral source).

This shows that the biosolids had higher manganese contents than the filter cake, which required less amount of the source with biosolids to promote greater availability of the nutrient in the soil. Although the fertilizers do not present in their composition manganese complementation, as with boron, it is known that organic compounds, such as sewage sludge and filter cake, have varying levels of micronutrients in their composition.

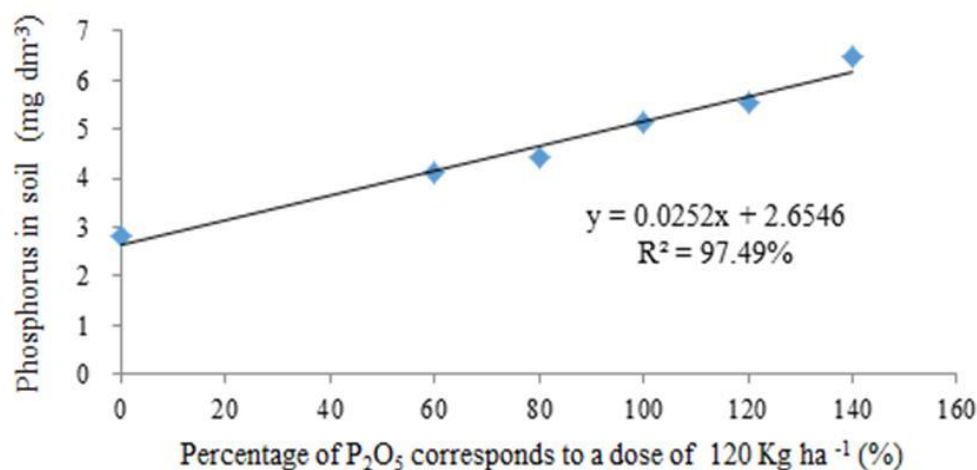
This soil response to manganese may also be related to the pH remaining at levels appropriate to the micronutrient (Dechen and Nachtigal, 2006) and also by the action of potassium sources, such as potassium chloride, present in the formulation that may promote solubility, mobility and availability in manganese through the anions associated with these potassium sources (Santos, 2013).

**Table 1.** Phosphorus in soil ( $\text{mg dm}^{-3}$ )<sup>1</sup> at 65 days after sowing of maize submitted to different doses of organomineral fertilizer composed of biosolids and filter cake in relation to mineral fertilization and absence of fertilization.

Percentage of Phosphorus (%)	Organomineral Fertilizer	
	Biosolids	Filter Cake
60	4.10	4.18
80	4.43 *	4.33
100	5.15 **	4.53 *
120	5.53 **	5.13 **
140	6.48 **	5.88 **
Average	5.13	4.81
Mineral fertilization	3.15 °	
Absence of fertilization	2.82 *	

CV% = 9.04; DMS<sub>Dunnett</sub> = 0.3958; DMS<sub>source</sub> = 0.1242; <sup>2</sup> W = 0.890; F levene = 2.396; F additivity = 0.523

<sup>1</sup> Phosphorus available in the soil solution (extractor Mehlich<sup>1</sup>), ° and \*: different by the Dunnett test at 0.05; Averages followed by distinct letters on the line differ from each other by the Tukey test at 0.05. <sup>2</sup> W, F levene, F additivity: statistics of Shapiro-Wilk, Levene and Tukey tests for additivity, respectively; values in bold indicate residues with normal distribution, homogeneous variances and additivity, all at 0.01 of significance.



**Fig 1.** Phosphorus in soil ( $\text{mg dm}^{-3}$ ) at 65 days after sowing with doses of organomineral fertilizer based on biosolids.

**Table 2.** Potassium in soil ( $\text{mg dm}^{-3}$ )<sup>1</sup> at 65 days after sowing of maize submitted to different doses of organomineral fertilizer composed of biosolids and filter cake in relation to mineral fertilization and absence of fertilization.

Percentage of Phosphorus (%)	Organomineral Fertilizer	
	Biosolids	Filter Cake
60	0.14	0.14
80	0.12 °	0.12 °
100	0.13 °	0.14
120	0.14	0.14
140	0.16	0.16
Average	0.13	0.14
Mineral fertilization	0.19 °	
Absence of fertilization	0.14 *	

CV% = 1.27; DMS<sub>Dunnett</sub> = 0.0277; DMS<sub>source</sub> = 0.0087; <sup>2</sup> W = 0.934; F levene = 1.639; F additivity = 1.809

<sup>1</sup> ° and \*: different by the Dunnett test at 0.05; Averages followed by distinct letters on the line differ from each other by the Tukey test at 0.05. <sup>2</sup> W, F levene, F adit: statistics of Shapiro-Wilk, Levene and Tukey tests for additivity, respectively; values in bold indicate residues with normal distribution, homogeneous variances and additivity, all at 0.01 of significance.

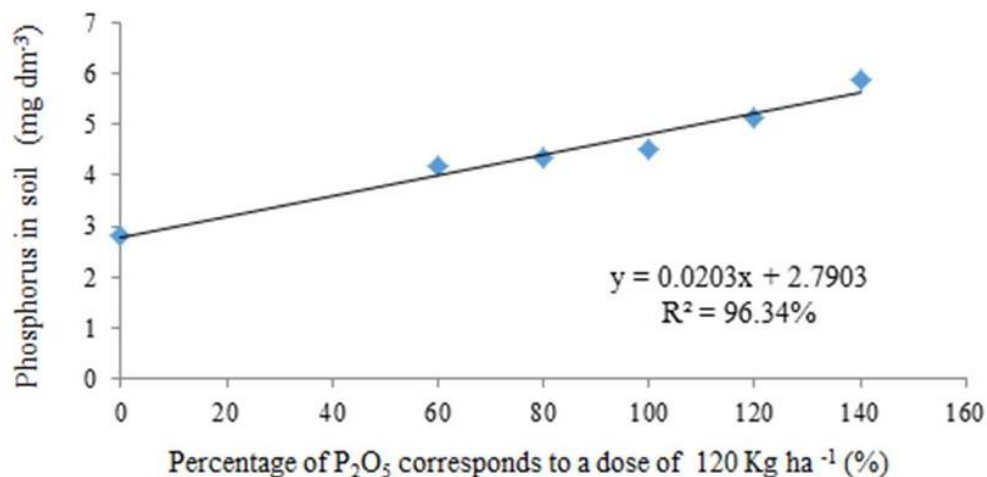


Fig 2. Phosphorus in soil (mg dm<sup>-3</sup>) at 65 days after sowing with doses of organomineral fertilizer based on filter cake.

**Table 3.** pH, Calcium (cmol<sub>c</sub> dm<sup>-3</sup>), Magnesium (cmol<sub>c</sub> dm<sup>-3</sup>), Base saturation-V (%) and sum of bases -SB (cmol<sub>c</sub> dm<sup>-3</sup>) in soil at 65 days after sowing of maize submitted to different doses of organomineral fertilizer composed of biosolids and filter cake in relation to mineral fertilization and absence of fertilization.

Percentage of Phosphorus (%)	pH water		Calcium		Magnesium		V		SB	
	Organomineral Fertilizer									
	Bio. <sup>1</sup>	F. C. <sup>2</sup>	Bio.	F. C.	Bio.	F. C.	Bio.	F. C.	Bio.	F. C.
60	5.80	5.70	3.03	3.15	0.88	0.90	56.28	57.58	4.04	4.19
80	5.63	5.58	3.15	3.10	0.88	0.88	54.75	57.18	4.14	4.10
100	5.70	5.63	3.13	3.10	0.90	0.88	56.48	57.35	4.15	4.12
120	5.60	5.75	3.10	3.15	0.80	0.88	55.20	56.15	4.04	4.17
140	5.80	5.68	3.10	3.03	0.85	0.88	57.10	56.98	4.11	4.06
Average	5.71	5.67	3.10	3.11	3.10	3.11	55.96	57.05	4.09	4.13
Mineral	5.75 °		3.05 °		0.87 °		55.93 °		4.12 °	
Absence	5.73 *		3.15 *		0.88 *		58.23 *		4.16 *	
CV%	2.60		5.51		7.09		5.32		5.59	
DMS Dunnett	0.3034		0.3509		0.1267		6.1824		0.4721	
DMS Source	0.0952		0.1101		0.0397		1.9397		0.2781	
<sup>2</sup> W	0.965		0.936		0.894		0.993		0.937	
F levene	1.234		2.778		1.654		1.476		2.330	
F additivity	0.140		0.051		0.354		0.020		0.089	

<sup>1</sup> Biosolids; <sup>2</sup> Filter cake; ° and \*: different by the Dunnett test at 0.05; Averages followed by distinct letters on the line differ from each other by the Tukey test a 0.05. <sup>2</sup> W, F levene, F additivity: statistics of Shapiro-Wilk, Levene and Tukey tests for additivity, respectively; values in bold indicate residues with normal distribution, homogeneous variances and additivity, all at 0.01 of significance.

**Table 4.** Copper, Iron e Zinc in soil ( $\text{mg dm}^{-3}$ ) at 65 days after sowing of maize submitted to different doses of organomineral fertilizer composed of biosolids and filter cake in relation to mineral fertilization and absence of fertilization.

Percentage of Phosphorus (%)	Cu		Fe		Zn	
	Organomineral Fertilizer					
	Bio. <sup>1</sup>	F. C. <sup>2</sup>	Bio.	F. C.	Bio.	F. C.
60	8.57	8.45	25.00	24.00	4.78	4.73
80	8.65	8.55	25.50	24.75	4.85	4.75
100	8.38	8.20	25.75	25.75	4.73	4.53
120	8.55	8.45	25.00	23.00	4.83	4.95
140	8.05	8.30	24.75	26.00	4.53	5.03
Average	8.44	8.39	25.20	24.70	4.74	4.80
Mineral fertilization	7.95 °		23.25 °		4.48 °	
Absence of fertilization	8.00 *		24.00 *		4.40 *	
CV%	4.11		6.32		6.56	
DMS Dunnett	0.7042		3.2085		0.6345	
DMS Source	0.2209		1.0067		0.1990	
<sup>2</sup> W	0.980		0.984		0.98	
F levene	0.659		0.453		1.154	
F additivity	8.127		0.254		0.003	

<sup>1</sup>Biosolids; <sup>2</sup> Filter cake. ° and \*: different by the Dunnett test at 0.05; Averages followed by distinct letters on the line differ from each other by the Tukey test a 0.05. <sup>2</sup> W, F levene, F additivity: statistics of Shapiro-Wilk, Levene and Tukey tests for additivity, respectively; values in bold indicate residues with normal distribution, homogeneous variances and additivity, all at 0.01 of significance.

**Table 5.** Boron in soil ( $\text{mg dm}^{-3}$ )<sup>1</sup> at 65 days after sowing of maize submitted to different doses of organomineral fertilizer composed of biosolids and filter cake in relation to mineral fertilization and absence of fertilization.

Percentage of Phosphorus (%)	Organomineral Fertilizer	
	Biosolids	Filter Cake
60	0.20	0.18
80	0.24 *	0.22 *
100	0.19	0.22 *
120	0.29 °*	0.24 *
140	0.34 °*	0.32 °*
Average	0.25	0.24
Mineral fertilization	0.14 °	
Absence of fertilization	0.09 *	

CV% = 2.25; DMS<sub>Dunnett</sub> = 0.0509; DMS<sub>source</sub> = 0.0159; <sup>2</sup> W = 0.972; F levene = 1.874; F additivity = 7.453

<sup>1</sup> ° and \*: different by the Dunnett test at 0.05; Averages followed by distinct letters on the line differ from each other by the Tukey test a 0.05. <sup>2</sup> W, F levene, F additivity: statistics of Shapiro-Wilk, Levene and Tukey tests for additivity, respectively; values in bold indicate residues with normal distribution, homogeneous variances and additivity, all at 0.01 of significance.

**Table 6.** Manganese in soil ( $\text{mg dm}^{-3}$ )<sup>1</sup> at 65 days after sowing of maize submitted to different doses of organomineral fertilizer composed of biosolids and filter cake in relation to mineral fertilization and absence of fertilization.

Percentage of Phosphorus (%)	Organomineral Fertilizer	
	Biosolids	Filter Cake
60	10.95	11.13
80	11.65 °*	11.68 °*
100	10.98	11.18
120	11.93 °*	11.43 *
140	10.68	11.53 °*
Average	11.24	11.39
Mineral fertilization	10.10 °	
Absence of fertilization	10.03 *	

CV% = 5.85; DMS<sub>Dunnett</sub> = 1.33360; DMS<sub>source</sub> = 0.41845; <sup>2</sup> W = 0.986; F levene = 0.746; F additivity = 0.027

<sup>1</sup> ° and \*: different by the Dunnett test at 0.05; Averages followed by distinct letters on the line differ from each other by the Tukey test a 0.05. <sup>2</sup> W, F levene, F additivity: statistics of Shapiro-Wilk, Levene and Tukey tests for additivity, respectively; values in bold indicate residues with normal distribution, homogeneous variances and additivity, all at 0.01 of significance.

**Table 7.** Organic carbon and organic matter in the soil at 65 days after sowing of maize submitted to different doses of organomineral fertilizer composed of biosolids and filter cake in relation to mineral fertilization and absence of fertilization.

Percentage of Phosphorus (%)	Organic Carbon		Organic Matter	
	Organomineral Fertilizer			
	Biosolids	Filter Cake	Biosolids	Filter Cake
60	1.99	2.00	<sup>1</sup> 3.43	3.45
80	1.99	2.00	3.43	3.45
100	2.06	2.04	3.55	3.52
120	2.10	2.10	3.63	3.62
140	2.06	2.09	3.55	3.60
Average	2.04	2.05	3.10	3.11
Mineral fertilization	2.02 °		3.48 °	
Absence of fertilization	1.97 *		3.40 *	
CV%	4.96		4.94	
DMS Dunnett	0.2071		0.3554	
DMS Source	0.0649		0.1115	
<sup>2</sup> W	0.979		0.979	
F levene	0.580		0.619	
F additivity	0.101		1.416	

° and \*: different by the Dunnett test at 0.05; Averages followed by distinct letters on the line differ from each other by the Tukey test a 0.05. <sup>2</sup> W, F levene, F additivity: statistics of Shapiro-Wilk, Levene and Tukey tests for additivity, respectively; values in bold indicate residues with normal distribution, homogeneous variances and additivity, all at 0.01 of significance.

**Table 8.** Chromium, Molybdenum and Lead (mg L<sup>-1</sup>) in soil at 65 days after sowing of maize submitted to different doses of organomineral fertilizer composed of biosolids and filter cake in relation to mineral fertilization and absence of fertilization.

Percentage of Phosphorus (%)	Organomineral Fertilizer					
	Cr		Mo		Pb	
	Bio. <sup>1</sup>	F. C. <sup>2</sup>	Bio.	F. C.	Bio.	F. C.
60	4.40	4.70	0.05	0.04	3.71	4.16
80	4.73	5.11	0.02	0.02	3.97	3.61
100	4.59	6.07	0.02	0.04	3.98	3.75
120	5.17	2.78	0.02	0.03	3.87	3.99
140	2.64	3.70	0.02	0.01	3.96	3.36
Average	4.31	4.47	0.03	0.03	3.90	3.77
Mineral fertilization	4.09 °		0.03 °		4.12 °	
Absence of fertilization	3.93 *		0.02 *		3.57 *	
CV%	50.38		102.61		15.98	
DMS Dunnett	4.4727		0.05373		1.2588	
DMS Source	1.4033		0.01686		0.3949	
<sup>2</sup> W	0.978		0.850		0.979	
F levene	0.921		3.683		1.495	
F additivity	2.964		3.433		0.024	

<sup>1</sup>Biosolids; <sup>2</sup> Filter cake. ° and \*: different by the Dunnett test at 0.05; Averages followed by distinct letters on the line differ from each other by the Tukey test a 0.05. <sup>2</sup> W, F levene, F additivity: statistics of Shapiro-Wilk, Levene and Tukey tests for additivity, respectively; values in bold indicate residues with normal distribution, homogeneous variances and additivity, all at 0.01 of significance

#### **Organic carbon and organic matter in soil after application of pelletized organomineral fertilizers**

Organic carbon and organic matter were not influenced by sources of organic waste (biosolids and filter cake) or increasing doses. No changes were observed in these characteristics in the soil when compared to the mineral fertilization and not even with the absence of fertilization (Table 7).

The analysis of soil characterization indicated that the levels of Organic Carbon and Organic Matter are considered "medium" (1.17 – 2.32 dag kg<sup>-1</sup>) (Alvarez et al., 1999), remaining in this

same classification after 65 days of the application of the organomineral fertilizers.

Despite the absence of increases for these characteristics in the soil, it is possible to associate that the presence of these elements in the fertilizers were able to act in favor of the other nutrients, and a single application over a period of 65 days may not have been sufficient to cause large changes capable of being quantified.

One of the ways to improve the physical, chemical and microbiological characteristics of the soil is the application of organic matter. Ourives et al. (2010), verifying the use of an organic compound (Bokashi) and its effects on soil fertility destined for another grass (*Brachiaria brizantha* cv. Marandú),

found that the organic source was able to supply the soil and the plants at appropriate levels of phosphorus and other nutrients, maintaining dry mass production similar to that found in a conventional fertilization, and the elevation of phosphorus levels increased as the dose increased.

### **Heavy metals in soil after application of pelletized organomineral fertilizers**

As for the levels of heavy metals, absence of (Ni), Cadmium (Cd) and Cobalt (Co) were found for the two sources of all treatments. For the other investigated heavy metals (Chromium-Cr, Molybdenum-Mo and Lead-Pb) there were no changes in their levels in the soil as a function of the sources and doses, as well as in the comparison of these treatments with the additional ones (Table 8).

It is assumed that the levels of Ni, Cd and Co were well below the sensitivity of the methodology used, reflecting results with absence of these elements in the soil.

The greatest concern regarding heavy metals was in relation to sewage sludge, due to its original characteristics that tend to high levels of these substances. However, all heavy metals were below the limits established by the Resolution of the National Council for the Environment Conama 375/2006 (2006) (Ministry of the Environment National Environment Council, 2006), for accumulated theoretical loads allowed by the application of sewage sludge or byproducts in agricultural soils (Ni-74, Cd-4, Cr-154, Mo-13, Pb-41 Kg ha<sup>-1</sup>), reinforcing that the use of biosolids in organomineral fertilizer does not result in contamination of the soil by these heavy metals.

It is also worth noting that, when sewage sludge was treated, Alves Filho (2014) had already observed low levels of heavy metals for this same sanitary sewage sludge.

Trannin et al. (2005), studying the agronomic effects of the application of an industrial biosolid for maize crop, verified that Cd and Pd contents maintained acceptable limits of accumulated load even when in maximum dose treatments and with repeated applications. While Anjos and Mattiazzo (2000), analyzing parts of the corn plant, found that the metals Cd, Cr, Ni and Pb presented levels below the limits of determination of the analytical method employed by them, although metals were incorporated into biosolids treatments.

## **Materials and methods**

### **Study site**

The experiment was carried out in a greenhouse located at the Institute of Agrarian Sciences of the Federal University of Uberlândia in the Umuarama Campus, located at 18°91'86" south latitude and 48°27'72" west longitude of Greenwich, at an altitude average of 800 m, in the period between 21/01 to 27/03 of 2015.

### **Experimental design**

The experiment was conducted in a randomized complete block design with four replicates in a 2 x 5 + 2 factorial scheme, corresponding to two sources of organic waste (biosolids and

filter cake), five doses of organomineral fertilizer (60, 80, 100, 120 and 140%, based on the recommended dose of P<sub>2</sub>O<sub>5</sub> for corn), and two additional treatments, represented by mineral fertilizer (100% of the recommended dose) and the absence of fertilization, totaling 12 treatments (Supplementary table 1).

The doses of the organomineral fertilizers were defined with reference to the P<sub>2</sub>O<sub>5</sub> content of the soil and following the "Recommendation for use of correctives and fertilizers in Minas Gerais" (Alves et al., 1999), so that the 100% dose corresponded to 120 kg ha<sup>-1</sup> of fertilizer to supply the need to grow corn in P<sub>2</sub>O<sub>5</sub> and the other doses were in relation to this 100% dose.

The formulation of pelletized organomineral fertilizers based on biosolids and filter cake was 5-17-10 (0.1% Boron + 3% Silicon + 8% of organic carbon-TOC) and formulated by the company Geociclo, located in Uberlândia-MG, through the enrichment of organic fertilizers with mineral fertilizers. In the first place, a stable compound was obtained by means of composting the organic sources; after that, nutrients were balanced according to the requirements of the crop and what the soil can offer. REGULATION NR. 25, OF JULY 23, 2009, SECTION V, Art. 8, § 1 Ministry of Agriculture, Livestock and Food Supply (2009) states that organomineral fertilizers must comply with specifications and guarantees established by MAPA. The following parameters are established: minimum 8% organic carbon, maximum 30% humidity, minimum Cation Exchange Capacity of 80 mmole kg<sup>-1</sup> and at least 10% declared macronutrients for products with primary macronutrients.

Sewage sludge was obtained as a by-product of the urban sewage treatment processes at Municipal Water and Sewage Department - DMAE, located in the city of Uberlândia, MG. The filter cake was obtained from the process of treatment and clarification of the sugarcane juice done at the sugar and alcohol industry company Vale do Tijuco, located in the city of Uberaba, MG. The sewage sludge was sanitized with hydrated lime in the proportion of 30% in the dry base aiming at the elimination of the pathogens and the reduction of humidity was carried out through natural drying in the sun for 7 days, according to methodology developed by Alves Filho (2014). After this treatment the sewage sludge is renamed here as a biosolid.

In the treatment with exclusively mineral fertilization, the formulation 5-17-10 was prepared using urea (42%), simple superphosphate (18%) and potassium chloride (58%) to supply nitrogen, phosphorus and potassium, respectively. These sources were homogenized and applied to the soil to supply 100% of the P<sub>2</sub>O<sub>5</sub> recommendation for corn crop. In this way, the application of 1.76 g of the formulation 5-17-10 in 5 kg of soil was carried out (corresponding to 120 kg ha<sup>-1</sup> of P<sub>2</sub>O<sub>5</sub>).

### **Conduction of study**

The plots consisted of two pots with a capacity of 5 kg of soil, measuring 20 cm in height, 20 cm in upper diameter and 17 cm in lower diameter. The soil was collected at the Capim Branco experimental farm of the Federal University of Uberlândia, in the city of Uberlândia-MG and characterized as Eutrophic Red Latosol according to the Embrapa methodology (2013)



(Supplementary table 2). No acidity correction was required since it was within the ideal pH range for maize cultivation.

### **The organomineral fertilizer**

For the experiment, the sewage sludge used in the composition of the organomineral fertilizer came from the DMAE treatment plant located in the city of Uberlândia, MG, and the filter cake was supplied by Vale do Tijuco, Uberaba, MG. The sewage sludge was sanitized in the proportion of 30% hydrated lime in the dry base, aiming at the elimination of the pathogens and for the reduction of humidity, the natural drying in the sun was carried out for 7 days, according to methodology developed by Alves Filho (2014).

The chemical characteristics of the biosolid and the filter cake were analyzed in the Laboratory of Soil Analysis of the Federal University of Uberlândia (Tables 11 and 12).

Seeding was performed on January 21, 2015, at the depth of three centimeters, four seeds were placed per pot and after 15 days slabs were made leaving two plants per pot of the hybrid DKB 390. Cover fertilization was applied with 70 kg ha<sup>-1</sup> of ammonium sulfate (20% N) when the plants had eight fully developed leaves, according to "Recommendation for use of correctives and fertilizers in Minas Gerais" (Alves et al., 1999).

### **Assessments of plant and soil variables**

At 65 days after sowing, after the removal of the plants, soil samples were obtained through simple samples of the two vessels that composed the plot. The composite samples of each experimental plot were air dried and later sieved and conditioned in plastic bags for the determination of the macronutrient and micronutrient contents in the Laboratory of Soil Analysis of the Federal University of Uberlândia. The Mehlich-1 extractor was used to determine soil phosphorus levels, and for Ca, Mg, B, Cu, Mn and Zn contents of the soil, the methodology described by Raij et al (2001) was used.

Chromium (Cr), Nickel (Ni), Lead (Pb), Cobalt (Co) and Molybdenum (Mo) (mg L<sup>-1</sup>) were also analyzed in the soil by the same methodology as the micronutrients (DTPA 0.005 mol L<sup>-1</sup> + TEA 0.1 mol L<sup>-1</sup> + CaCl<sub>2</sub> 0.001 mol L<sup>-1</sup> at pH 7.3) by the Brazilian Laboratory of Agricultural Analyzes Ltda, located in the city of Monte Carmelo-MG.

### **Statistical analysis**

The data obtained were initially tested for assumptions of normality of residues (Shapiro-Wilk test), homogeneity of variances (Levene test) and block expansion (Tukey test for additivity), using SPSS software version 20.0. Subsequently, the means of the evaluated characteristics were submitted to the F test of the analysis of variance. The study of organomineral fertilizers formulated with sewage sludge and filter cake was done by the Tukey test to compare the means of the treatments. The study of the doses of organomineral fertilizers was performed by regression to obtain a statistical model.

For additional treatments, the Dunnett Test was applied with the assistance of the ASSISTAT statistical program version 7.6 beta (Silva, 2016). Sigma Plot software for Windows Version

11.0 Build 11.0.0.77 was used to search for regression models of the quantitative data, assuming as good models that were significant and with coefficient of determination (R<sup>2</sup>) above 70%.

The variables potassium (K), boron (B) and copper (Cu) were transformed by  $\sqrt{x + 1}$  and they began to meet the assumptions at 0.01 of significance, except for copper that even with data transformation was not significant for additivity of block. The phosphorus (P) was transformed by  $\sqrt{x}$ ; Magnesium (Mg) and Molybdenum (Mo) by  $\sqrt{x + 1}$ . The normality of residues was not met for P and Mg (being accepted transformation of data only for P); Mo only met the assumption of block additivity. All other variables considered the assumptions at 0.01 of significance without data transformation.

### **Conclusion**

Organomineral fertilizers formulated with biosolids have been shown to have the potential to be used for the supply of nutrients, mainly phosphorus, boron and manganese.

Doses equal to or below the fertilization recommendation for maize cultivation were less efficient in the availability of potassium.

The manganese in the soil was more influenced by the high doses of the two sources, in relation to the mineral fertilization, being that biosolids exerted this influence in a smaller dose (120% P<sub>2</sub>O<sub>5</sub>).

Increasing doses of organomineral fertilizers have not demonstrated soil contamination potential for the heavy metals Nickel, Cadmium, Cobalt, Chromium, Molybdenum and Lead.

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