

## Short-term changes in soil properties due to sanitary wastewater irrigation used as a potassium source

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### Abstract

Irrigation with wastewater is an agricultural practice used to supply plants with nutrients that can reduce the nutrient load impact on fresh water sources and save conventional water sources. However, the physical and chemical properties of soil can be altered according to specific wastewater characteristics. This study aimed to investigate short-term changes in soil physical and chemical properties after irrigation of cotton with preliminary treated wastewater (PTW) and tertiary treated wastewater (TTW) as potassium sources in the semi-arid region of Minas Gerais State, Brazil. A randomized complete block design (RCBD) with four replications and five treatments was used: a combination of two wastewaters (PTW and TTW) as potassium topdressing sources and two equivalent doses to 40 and 60 kg K<sub>2</sub>O ha<sup>-1</sup>, as well as a control treatment with a conventional mineral fertilizer. After the first soil sampling, the liming, NPK fertilizer, urea and potassium chloride treatments increased the base saturation and electrical conductivity up to 0.4 m soil depth. Wastewater potassium sources did not promote chemical excesses in the four soil depths evaluated, although the sodium content increased up to 0.6 m depth with 60 kg K<sub>2</sub>O ha<sup>-1</sup> via TTW. The same dose (60 kg K<sub>2</sub>O ha<sup>-1</sup>) via PTW decreased the soil pH in the top 0.2 m and the water-dispersible clay (WDC) level up to 0.6 m depth due to the better chemical balance of this wastewater. In general, it is recommended to use PTW when providing 60 kg K<sub>2</sub>O ha<sup>-1</sup> in topdressing.

**Keywords:** raw sewage, treated sewage, Oxisol, clay dispersion.

**Abbreviations:** WDC\_water-dispersible clay, PTW\_preliminary treated wastewater, TTW\_tertiary treated wastewater, EC<sub>e</sub>\_soil electrical conductivity of the saturated paste extract, DAE\_days after emergence, RCBD\_randomized complete block design, ESP\_exchangeable sodium percentage.

### Introduction

Disposal in the soil is one of the oldest practices for the treatment, final disposal, and/or recycling of sanitary sewage. In the United Kingdom, at the beginning of the nineteenth century, the first experiences with sewage application were known as “sewage farms”. In Melbourne, Australia, the Western Treatment Plant, in operation since 1897, occupies 11,000 ha and receives 485 million litres of sewage per day that is used to irrigate pastures for 15,000 cattle and 40,000 sheep (Melbourne Water, 2014). Wastewater use schemes, if properly planned and managed, can have a positive environmental impact and be applied in agricultural production. Environmental improvement may be related to the avoidance of surface water pollution; conservation or more rational use of fresh water resources, especially in arid and semi-arid areas, via the use of fresh water for urban demand and wastewater for irrigation purposes; and reduced requirements for artificial fertilizers, with a concomitant reduction in energy expenditure and industrial pollution elsewhere (WHO, 2006). Irrigation with treated wastewater can improve the water retention capacity of soil by an average of 12%, increase the soil organic matter level by an average of 30%, and enhance the soil microflora count by 80% (Mekki et al., 2014). However, the final disposal of urban wastewater in receiving soil, be it preliminary treated

wastewater (PTW) or tertiary treated wastewater (TTW), frequently and at high doses can promote physical and/or chemical changes in soils because of the chemicals and organic matter present (Leal et al., 2009; Sandri et al., 2009; Souza et al., 2010; Ferreira et al., 2011; Medeiros et al., 2011). Disposal of secondary treated wastewater by surface drip irrigation can alter the transient Na content under the emitter in the soil fractions (Cary et al., 2015), increasing the soil’s electrical conductivity (EC) due to high Na values in wastewater and decreasing aggregate stability due to wetting and drying cycles associated with irrigation (Morugán-Coronado et al., 2011). Amongst these soil changes, the risks of salinization, permeability reduction, clay dispersion and nutrient imbalance are important to consider (Medeiros et al., 2005a; Kiziloglu et al., 2008; Almeida Neto et al., 2009; Silva et al., 2010; Souza et al., 2010; Xu et al., 2010; Varallo et al., 2012). Thus, application criteria should be established to maintain a frequency, dose, and crop nutrient requirement that are compatible with the environment by avoiding the accumulation of chemical contaminants in the soil, with adequate monitoring (Medeiros et al., 2005b; Fonseca et al., 2007; Varallo et al., 2010; Matos et al., 2013).

Cotton farming in northern Minas Gerais State, Brazil has increased in recent years in association with incentives given

by the state government. The cotton crops are mainly grown by small-scale farmers. Because the main commercial product is inedible, applying wastewater to the crop is a feasible alternative for both providing nutrients and rationing the water supply (Medeiros et al., 2011) as well as reducing the cost of crop production for farmers with limited resources. Furthermore, close to crop areas, there are rural communities without wastewater-treatment systems that could use preliminary treated wastewater for crop production. Assessing changes in the soil physical and chemical properties is essential for sustainable production.

This study aimed to investigate the short-term changes in soil physical and chemical properties after cotton was irrigated with different sources and doses of wastewater as potassium sources in the semi-arid region of Minas Gerais State, Brazil.

## Results and Discussion

### *Initial soil conditions, irrigation water, wastewater constituents and soil inputs*

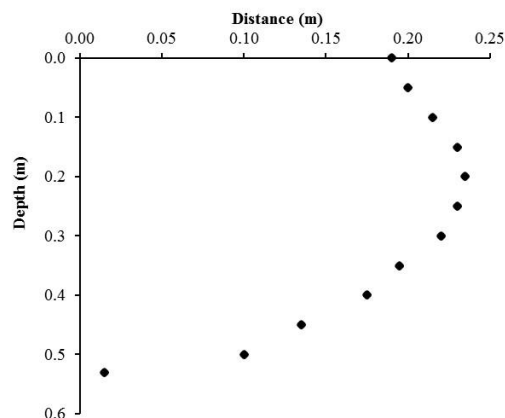
Five days before applying the treatments, samples were collected from 0-0.2 m and 0.2-0.4 m soil depths in the experimental plots. At this time, none of the variables differed among the treatments (Table 1), showing uniformity of liming and starter fertilization at sowing across all the experimental plots. Compared to the results of the first soil sampling (Table 2), the application of dolomite lime, fertilizer (NPK 4-30-10), urea, and potassium chloride increased the base saturation and the soil electrical conductivity ( $EC_e$ ) up to a depth of 0.4 m depth (Table 1). The  $EC_e$  mainly increased in association with the potassium mineral source (Yamasaki and Kishita, 1972; Andrade Neto et al., 2012). The average composition of the PTW was 92.3 mg L<sup>-1</sup> total nitrogen, 13.4 mg L<sup>-1</sup> phosphorus, 30.3 mg L<sup>-1</sup> potassium, 47.3 mg L<sup>-1</sup> sodium, 3.2 mg L<sup>-1</sup> calcium, 73.0 mg L<sup>-1</sup> oils and greases, and 3.0 mg L<sup>-1</sup> iron, with 642.7 mg L<sup>-1</sup> biochemical oxygen demand (BOD), pH of 7.2, and  $EC_e$  of 1.4 dS m<sup>-1</sup>. The TTW contained 32.7 mg L<sup>-1</sup> total nitrogen, 9.0 mg L<sup>-1</sup> phosphorus, 29.5 mg L<sup>-1</sup> potassium, 60.9 mg L<sup>-1</sup> sodium, 5.1 mg L<sup>-1</sup> calcium, 10.8 mg L<sup>-1</sup> oils and greases, and 1.1 mg L<sup>-1</sup> iron, with 187.3 mg L<sup>-1</sup> BOD, pH of 7.4, and  $EC_e$  of 1.2 dS m<sup>-1</sup>. The PTW contained higher levels of organic matter and total nitrogen but lower sodium levels. Aiming to meet the potassium demands in the treatments in which the wastewater was applied, irrigation ranged between 110.6 mm (T3) and 177.4 mm (T2) (Table 3). A higher TTW irrigation was necessary because of its lower potassium level, which provided greater amounts of nitrogen, phosphorus (in the form of P<sub>2</sub>O<sub>5</sub>), oils and greases, iron, and BOD (organic matter) inputs to the treatments subjected to PTW (Table 4). The opposite pattern was observed for potassium (K<sub>2</sub>O), calcium, and sodium; the treatments with TTW irrigation received higher inputs of these nutrients than the treatments that received PTW. The highest sodium input occurred with the TTW equivalent dose to 60 kg K<sub>2</sub>O ha<sup>-1</sup> (T2); the mean sodium content was 29% higher in the TTW compared to the PTW. Additionally, there was also a higher TTW demand to reach the equivalent potassium dose in this treatment.

### *Impacts on soil chemical properties*

At the end of the experiment, there were no significant differences in organic matter and phosphorus levels among the treatments for any of the depths analysed (Table 5). For soil pH, potassium, calcium, and base saturation, differences only occurred within topsoil up to 0.2 m depth. Iron content

differed between the treatments and control at 0.2-0.4 m soil depth, and sodium differed at up to 0.6 m soil depth. There were no differences in any of the variables among the treatments at 0.4-0.6 m soil depth. The amount of organic matter added by the wastewater did not modify the soil organic matter level of the treatments (Supplementary Table 1). Conceptually, BOD can be used to estimate the organic matter content of wastewater (Matos et al., 2013). In this sense, for the sake of comparison, assuming that all wastewater BOD input was in organic matter form, there was an increase of 1,036.7 kg ha<sup>-1</sup> of organic matter in T4 (Table 4). This input is relatively low, considering that changes in organic matter content from soil surface to a depth of 0.15 m can be detected starting with an addition of 10,000 kg ha<sup>-1</sup> solid waste in wheat post-harvest (Latare et al., 2014). Thus, no change was expected. Additionally, during the 62 days between the last wastewater application and soil sampling, most of the organic matter added to soil by the wastewater may have been mineralized as a result of the carbon/nitrogen ratio, which is typically lower than 30/1 (Blum et al., 2013). Notably, during the soil sampling time, cotton plants had not been removed, generating a slight increase in soil organic matter. The available soil phosphorus content was very low throughout the profile (Ribeiro et al., 1999) despite the addition of up to 55 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup> by wastewater and fertilization at sowing (Supplementary Table 1). The phosphorus added via wastewater, although quantified as total phosphorus, was predominantly in the organic and orthophosphate ion form (Von Sperling, 2005). A portion of orthophosphate can be adsorbed into the colloidal fraction of the Latosol (Oxisol), which is composed of iron and aluminium oxy-hydroxides, and because of the medium's high pH, a portion can react with the available calcium (Supplementary Table 1), becoming insoluble (Novais et al., 2007). Although the extractant (Mehlich-1) solubilizes the fraction of phosphorus bound to calcium, Novais et al. (2007) have stated that this extraction method can underestimate the phosphorus content because of a soil buffering effect on the acid extractant and because of the rapid entry of SO<sub>4</sub><sup>2-</sup> ions into sites not occupied by phosphate. A pattern similar to that previously described for organic matter may have occurred for the organic phosphorus. Sandri et al. (2009) reported similar results for the first 0.2 m soil depth after TTW application in two lettuce crop cycles. In the present study, although phosphorus was not quantified in the soil profile before the treatment application to 0.8 m depth (Table 1), it can be inferred that any phosphorus input that was retained would remain in the topsoil because of its low mobility and that the phosphorus fraction not taken up by the crop may have reacted with cations (Ca<sup>2+</sup>) and precipitated as unavailable Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> (Novais et al., 2007). Additionally, the availability of phosphorus in the soil remained unchanged except for an increasing trend observed in the PTW treatments. Similar results were reported by Medeiros et al. (2011), who applied various dilutions of treated swine wastewater (SWW) to a Red-Yellow Latosol (Oxisol) cultivated with "BRS Camaçari" cotton, supplying an estimated input of 746 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup> during two crop cycles of a treatment that received 100% effluent to reach the crop water requirement. There was no difference between the treatments and control regarding soil pH up to 0.2 m depth (Supplementary Table 1). However, the soil pH of T4 was 6.2, lower than that of T1 and T2 but equal to that of T0 and T3. For the other depths, soil pH tended to decrease, without differing among the treatments, a pattern similar to that observed by Medeiros et al. (2005a). Xu et al. (2010) reported that 20 years of TTW application decreased the soil

pH from 6.5-7.0 to 5.4-6.3. Similarly, Kiziloglu et al. (2008) reported that soil acidity increased in the 0-0.3 m depth of soil irrigated with PTW. It is common for soil pH to decrease in areas treated with high and frequent doses of non-stabilized crude organic wastes because the acidifying initial phase of organic matter degradation produces carbon dioxide and organic acids (Matos et al., 2013). In the 0-0.2 m soil depth, potassium content after PTW applications was lower than that of T0 (Supplementary Table 1). The mean potassium contents were 161, 160, and 107 mg K dm<sup>-3</sup> in T0, T2, and T4, respectively, and these levels are considered very good, very good, and good (Ribeiro et al., 1999). Before wastewater application, the potassium content was at the very good level up to 0.4 m depth (Table 1). Because the nutrient inputs due to wastewater remained close to the established levels, i.e., T2 equal to 60 kg K<sub>2</sub>O ha<sup>-1</sup> and T4 equal to 57.1 kg K<sub>2</sub>O ha<sup>-1</sup> (Table 4), the potassium content in plants was not measured at the end of cotton growing cycle, and it is most likely that the higher amounts of nitrogen and phosphorous supplied in T4 increased the potassium uptake, which was exported via the cotton yield (Rosolem and Witacker, 2007). The same applies to T3. Similar to the results of this study, Medeiros et al. (2011) reported higher potassium content in soil under cotton irrigated with 100% swine wastewater (SWW) (input of 1,493 kg K<sub>2</sub>O ha<sup>-1</sup>), compared with a mineral control at 0-0.2 m depth, but no differences were observed among treatments for the other depths, even with potassium decreasing with depth. Due to this decrease, the authors stated that there was no evidence of potassium ion leaching. Due to the high iron input from PTW irrigation (Table 4), the highest soluble iron levels in soil (19.4%) occurred in the 0.2-0.4 depth compared to T0 (Supplementary Table 1). After applying topdressing fertilizer (Table 1), iron levels were 19.7% and 15.4% lower in the 0-0.2 and 0.2-0.4 m depths, respectively. For the PTW treatments, soluble iron in the 0.2-0.4 m depth was 8.6% greater because of the higher iron content in wastewater. PTW applications usually increase iron contents in soil (Kiziloglu et al., 2008), but sometimes, there is no change (Medeiros et al., 2011). An iron increase may or may not be good for crops because it is a micronutrient but may also promote phytotoxicity (Novais et al., 2007). The calcium and base saturation levels in the 0-0.2 m depth of T4 were lower than the levels of T0 (Supplementary Table 1). The calcium content in T4, however, remained similar to those of T1, T2, and T3. The base saturation in T4 differed from T1 and T2 (Supplementary Table 1). Calcium and base saturation levels were considered good in all the treatments at the end of the crop cycle (Ribeiro et al., 1999). Because the seed cotton yield in T4 (3,843.1 kg ha<sup>-1</sup>) was 50.8% greater than in T0 (2,548.6 kg ha<sup>-1</sup>) whereas the soil calcium content in T4 remained 22.5% lower than in T0, the T4 plants most likely exported a greater amount of calcium, and consequently, there was a lower soil calcium content at the end of the cycle. Another explanation may be associated with the extended crop cycle in T4, the result of supplying nitrogen and phosphorus, which led to greater calcium uptake. Regarding the soil sampling performed at 30 days after emergence (DAE) (Table 1), the calcium content in T4 was 20.1% lower at the end of the crop cycle compared to the beginning, whereas it was 3.1% higher in T0. In general, the calcium levels at the end of the cycle were considered adequate. Calcium from liming (Table 4) has low mobility in soil (Novais et al., 2007).



**Fig 1.** Emitter distance and depth reached by the wetting front of a drip irrigation system in a eutrophic Red Latosol (Oxisol) at 57 DAE of cotton fertigated with sanitary wastewater.

Thus, there were no differences in the other layers evaluated. The lower nutrient levels at the end of the cotton cycle are evidence that higher wastewater doses, especially PTW, may be used to reach the crop's nutritional requirements and replenish soil nutrients, promoting sustainable crop production. The higher sodium input from TTW explains the higher base saturation in T1 and T2, in addition to potassium and calcium levels remaining slightly greater than levels in the PTW treatments. Compared to 30 DAE (Table 1), base saturation decreased by 16.4% in T4 in the upper 0.2 m of soil depth. The higher sodium input in TTW treatments resulted in a mean sodium content in T2 greater than that in T0 up to 0.6 m depth and greater than in the PTW treatments (Supplementary Table 1). The irrigation schedule explains the sodium accumulation because watering increased the soil moisture to reach the field capacity within the first 0.4 m depth. Because sodium is very mobile in wet soil (Ferreira et al., 2011; Pizarro, 1978), it tends to accumulate around the wet bulb. The soil was sampled within the wetted zone, and a portion of sodium may have concentrated in the 0.4-0.6 m depth, which encompasses the deepest depth of the wet bulb (0.53 m, Fig. 1) identified in field tests. A problem with applying sodium, which is highly common when using wastewater, is soil dispersion with clay platelet and aggregate swelling, changing the physical conditions of the soil (Medeiros et al., 2005b; Fonseca et al., 2007; Kiziloglu et al., 2008; Leal et al., 2009; Silva et al., 2010; Souza et al., 2010; Varallo et al., 2010; Ferreira et al., 2011; Medeiros et al., 2011; Matos et al., 2014). Therefore, several variables associated with these changes were analysed to monitor the effects of wastewater on the soil (Supplementary Table 2).

#### **Impacts on soil physical properties**

In the 0.4-0.6 m depth, EC<sub>e</sub> increased in T2 compared to T0. ESP in T2 remained higher than in T0 for the 0-0.2 and 0.4-0.6 m depths and exceeded that of the other treatments at the 0.4-0.6 m depth (Supplementary Table 2). TTW inputs promoted the accumulation of salts around the wetted zone (Fig. 1) and increased ESP and EC<sub>e</sub> in the 0.4-0.6 m depth. Salinity and sodicity were classified from soil ESP and EC<sub>e</sub>, and typical soils usually have values below 7% and 2 dS m<sup>-1</sup>, respectively (Pizarro, 1978). Thus, the treatments did not

**Table 1.** Summary of analysis of variance and means of soil pH (pH<sub>H2O</sub>), organic matter (OM), phosphorus (P), potassium (K), sodium (Na), calcium (Ca), base saturation (BS), soil electrical conductivity of the saturated paste extract (EC<sub>e</sub>), water-dispersible clay (WDC), exchangeable sodium percentage (ESP), and sand, silt, and clay levels at 30 DAE of cotton growth and before treatments (Treat.).

Soil depth (m)	SV	DF	Mean square							
			pH <sub>H2O</sub>	OM dag kg <sup>-1</sup>	P ..... mg dm <sup>-3</sup> .....	K ..... mg dm <sup>-3</sup> .....	Fe ..... cmol <sub>c</sub> dm <sup>-3</sup> .....	Na ..... cmol <sub>c</sub> dm <sup>-3</sup> .....	Ca ..... cmol <sub>c</sub> dm <sup>-3</sup> .....	BS %
0-0.2	Block	3	0.20	0.08	3.74	5600.58	30.26	0.0005	0.52	94.98
	Treat.	4	0.07 <sup>ns</sup>	0.12 <sup>ns</sup>	1.78 <sup>ns</sup>	1352.45 <sup>ns</sup>	32.57 <sup>ns</sup>	0.0005 <sup>ns</sup>	0.22 <sup>ns</sup>	16.38 <sup>ns</sup>
	Res.	12	0.18	0.06	2.21	7359.58	10.07	0.0005	0.25	27.11
CV (%)			6.02	16.19	61.27	34.06	14.24	21.30	13.01	6.41
Mean			7.08	1.6	2.43	251.9	22.28	0.11	3.88	81.3
0.2-0.4	Block	3	0.06	0.12	0.20	1847.78	15.71	0.002	0.17	12.45
	Treat.	4	0.16 <sup>ns</sup>	0.06 <sup>ns</sup>	0.55 <sup>ns</sup>	4722.67 <sup>ns</sup>	5.34 <sup>ns</sup>	0.0008 <sup>ns</sup>	0.28 <sup>ns</sup>	39.93 <sup>ns</sup>
	Res.	12	0.18	0.07	0.29	3952.74	6.39	0.001	0.16	34.83
CV (%)			6.71	30.93	38.16	37.86	14.45	32.73	11.99	36.64
Mean			6.4	0.9	1.4	166.1	17.5	0.11	3.4	73.6
			EC <sub>e</sub> (dS m <sup>-1</sup> )	WDC (dag kg <sup>-1</sup> )	ESP (%)	Sand (dag kg <sup>-1</sup> )	Silt (dag kg <sup>-1</sup> )	Clay (dag kg <sup>-1</sup> )		
0-0.2	Block	3	0.13	8.12	0.12	42.72	44.60	88.85		
	Treat.	4	0.12 <sup>ns</sup>	3.42 <sup>ns</sup>	0.10 <sup>ns</sup>	16.18 <sup>ns</sup>	26.68 <sup>ns</sup>	31.13 <sup>ns</sup>		
	Res.	12	0.08	3.88	0.07	10.34	17.81	22.73		
CV (%)			29.84	26.08	20.58	6.18	16.42	21.43		
Mean			0.97	7.56	1.33	51.8	25.4	22.8		
0.2-0.4	Block	3	0.01	7.57	0.36	18.18	30.18	19.80		
	Treat.	4	0.02 <sup>ns</sup>	1.54 <sup>ns</sup>	0.15 <sup>ns</sup>	12.80 <sup>ns</sup>	20.08 <sup>ns</sup>	3.68 <sup>ns</sup>		
	Res.	12	0.03	2.36	0.27	5.60	10.98	7.51		
CV (%)			29.23	14.45	32.06	4.78	16.44	9.04		
Mean			0.58	10.64	1.64	49.0	20.6	30.4		

<sup>\*</sup>Significant at 5%, <sup>\*\*</sup>significant at 1%, and <sup>ns</sup>not significant based on an F test.

**Table 2.** Baseline physical, chemical, and physical-chemical soil properties, before dolomite liming and treatments.

Soil depth (m)	pH <sub>H2O</sub>	<sup>1</sup> OM dag kg <sup>-1</sup>	<sup>2</sup> P ..... mg dm <sup>-3</sup> .....	<sup>2</sup> K	<sup>2</sup> Na	<sup>3</sup> Ca ..... cmol <sub>c</sub> dm <sup>-3</sup> .....	<sup>3</sup> Mg	<sup>3</sup> Al
0-0.2	6.2	1.3	2.3	260	0.1	2.8	0.9	0
0.2-0.4	5.5	0.7	2.0	140	0.1	2.2	0.7	0
	<sup>4</sup> H+Al	<sup>5</sup> SB	<sup>6</sup> CEC <sub>e</sub>	<sup>7</sup> CEC <sub>pH7</sub>	<sup>8</sup> BS	<sup>9</sup> ASI	<sup>10</sup> B	<sup>3</sup> Cu
	..... cmol <sub>c</sub> dm <sup>-3</sup> .....	..... cmol <sub>c</sub> dm <sup>-3</sup> .....	..... mg dm <sup>-3</sup> .....	..... mg dm <sup>-3</sup> .....	..... % .....	..... % .....	..... mg dm <sup>-3</sup> .....	..... mg dm <sup>-3</sup> .....
0-0.2	2.2	4.5	4.5	6.7	67	0	0.3	1.0
0.2-0.4	2.2	3.4	3.4	5.6	61	0	0.4	0.9
	<sup>3</sup> Fe	<sup>3</sup> Mn	<sup>3</sup> Zn	<sup>11</sup> P <sub>rem</sub>	<sup>12</sup> EC <sub>e</sub>	Sand	Silt	Clay
	..... mg dm <sup>-3</sup> .....	..... mg dm <sup>-3</sup> .....	..... mg dm <sup>-3</sup> .....	..... mg L <sup>-1</sup> .....	..... dS m <sup>-1</sup> .....	..... dag kg <sup>-1</sup> .....	..... dag kg <sup>-1</sup> .....	..... dag kg <sup>-1</sup> .....
0-0.2	23.7	10.8	0.8	35.2	0.3	52.0	25.7	22.3
0.2-0.4	24.8	3.9	0.4	30.6	0.2	49.6	20.1	30.3
	<sup>13</sup> Ma	<sup>14</sup> Mi	<sup>15</sup> TP	<sup>16</sup> ρ <sub>b</sub>	<sup>17</sup> FC	<sup>18</sup> PWP		
	..... m <sup>3</sup> m <sup>-3</sup> .....	..... m <sup>3</sup> m <sup>-3</sup> .....	..... m <sup>3</sup> m <sup>-3</sup> .....	..... kg dm <sup>-3</sup> .....	..... m <sup>3</sup> m <sup>-3</sup> .....	..... m <sup>3</sup> m <sup>-3</sup> .....		
0-0.2	0.1315	0.2924	0.4239	1.61	0.2746	0.1917		
0.2-0.4	0.1115	0.3035	0.4150	1.57	0.2549	0.1672		

<sup>1</sup>OM: organic matter, colorimetric determination; <sup>2</sup>Extractant: Mehlich-1; <sup>3</sup>Extractant: KCl 1 mol L<sup>-1</sup>; <sup>4</sup>Estimator: pH SMP; <sup>5</sup>SB: sum of bases; <sup>6</sup>CEC<sub>e</sub>: effective cation exchange capacity; <sup>7</sup>CEC<sub>pH7</sub>: cation exchange capacity at pH 7.0; <sup>8</sup>BS: base saturation; <sup>9</sup>ASI: aluminium saturation index; <sup>10</sup>Extractant: BaCl<sub>2</sub>; <sup>11</sup>P<sub>rem</sub>: remaining phosphorus, from P in the equilibrium solution; <sup>12</sup>EC<sub>e</sub>: electrical conductivity of the saturated paste extract, 1:2 water/soil ratio; <sup>13</sup>Ma: macroporosity; <sup>14</sup>Mi: microporosity; <sup>15</sup>TP: total porosity; <sup>16</sup>ρ<sub>b</sub>: soil bulk density; <sup>17</sup>FC: field capacity; <sup>18</sup>PWP: permanent wilting point.

change the soil salinity class, similar to the results found by Silva et al. (2010) and Varallo et al. (2012). However, sequential applications can impair the soil mainly because of sodium input. The EC<sub>e</sub> decreased by 45.3% and 27.6% in the 0-0.2 and 0.2-0.4 m depths, respectively, at the end of the cycle compared to the baseline (Table 1), most likely because of plant nutrient uptake. The ESP increased by 30.8% and 15.2% in these depths, respectively, mainly because of Na<sup>+</sup> increase in the soil exchange complex. Regarding particle size (sand, silt, and clay), there was no change with depth (Table 2 and Supplementary Table 2) as a result of the advanced degree of weathering and oxidic mineralogy of the Latosol (Oxisol), which predominantly consists of iron and aluminium oxy-hydroxides, such as hematite, goethite, and

gibbsite. The texture is sandy clay loam in the topsoil and changes to clay loam in the 0.6-0.8 m depth (Embrapa, 2013). Latosols (Oxisols) rarely change within short time periods, and a soil structural change is more likely a result of increased clay dispersion with sodium-rich irrigation water (Almeida Neto et al., 2009; Matos et al., 2014). T4 showed a lower water-dispersible clay (WDC) value compared to T0, with a higher flocculated clay value observed up to 0.6 m depth (Supplementary Table 2). The higher input of solids – suspended solids, such as oils and greases, colloidal solids, and dissolved solids – from PTW accelerate surface soil sealing and also translocate the colloidal and dissolved fractions of solids through the profile, promoting clay flocculation with binding cations, making bridges between

**Table 3.** Wastewater (WW) requirement, effective rainfall (rain), supplementary net irrigation depth (SN), and total irrigation depth applied to experimental plots during a cotton cycle.

Treatment	WW	Rain	SN	Total
	mm			
T0	0.0	9.4	482.3	491.7
T1	118.5	9.4	363.8	491.7
T2	177.4	9.4	304.9	491.7
T3	110.6	9.4	371.7	491.7
T4	166.3	9.4	316.1	491.7

T0: clean water and mineral fertilizer topdressing; T1: TTW as potassium source for topdressing, equivalent to 40 kg K<sub>2</sub>O ha<sup>-1</sup> and (T2) 60 kg K<sub>2</sub>O ha<sup>-1</sup>; T3: PTW as potassium source for topdressing, equivalent to 40 kg K<sub>2</sub>O ha<sup>-1</sup> and (T4) 60 kg K<sub>2</sub>O ha<sup>-1</sup>.

**Table 4.** Total application doses (kg ha<sup>-1</sup>) of chemical constituents, total nitrogen (N), phosphorus (P<sub>2</sub>O<sub>5</sub>), potassium (K<sub>2</sub>O), sodium (Na), calcium (Ca), oils and greases (O&G), iron (Fe), and organic matter (OM), provided to the treatments (Treat.) via mineral fertilizer (MF) and wastewater (WW).

Treat.	N <sup>*</sup>			P <sub>2</sub> O <sub>5</sub>			K <sub>2</sub> O			Na		
	MF	WW	Sum	MF	WW	Sum	MF	WW	Sum	MF	WW	Sum
T0	70.0	0.0	70.0	100.0	0.0	100.0	80.0	0.0	80.0	0.0	0.0	0.0
T1	35.5	37.5	73.0	100.0	23.8	123.8	40.0	40.2	80.2	0.0	69.0	69.0
T2	20.0	44.3	64.3	100.0	35.6	135.6	40.0	60.0	100.0	0.0	103.1	103.1
T3	20.0	70.1	90.1	100.0	36.5	136.5	40.0	38.0	78.0	0.0	52.7	52.7
T4	20.0	93.5	113.5	100.0	55.0	155.0	40.0	57.1	97.1	0.0	79.3	79.3
Treat.	Ca			O&G			Fe			BDO		
	MF	WW	Sum	MF	WW	Sum	MF	WW	Sum	MF	WW	Sum
T0	410.5	0.0	410.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
T1	410.5	4.6	415.1	0.0	16.7	16.7	0.0	1.3	1.3	0.0	212.8	212.8
T2	410.5	6.8	417.4	0.0	25.1	25.1	0.0	2.0	2.0	0.0	318.1	318.1
T3	410.5	4.1	414.6	0.0	73.3	73.3	0.0	3.3	3.3	0.0	689.4	689.4
T4	410.5	6.2	416.7	0.0	110.1	110.1	0.0	4.9	4.9	0.0	1036.7	1036.7

\*Corresponds to the nitrogen available for the crop according to Matos et al. (2013); T0: clean water and mineral fertilizer topdressing; T1: TTW as potassium source for topdressing, equivalent to 40 kg K<sub>2</sub>O ha<sup>-1</sup> and (T2) 60 kg K<sub>2</sub>O ha<sup>-1</sup>; T3: PTW as potassium source for topdressing, equivalent to 40 kg K<sub>2</sub>O ha<sup>-1</sup> and (T4) 60 kg K<sub>2</sub>O ha<sup>-1</sup>.

**Table 5.** Summary of analysis of variance for soil pH (pH<sub>H2O</sub>), organic matter (OM), phosphorus (P), potassium (K), sodium (Na), calcium (Ca), and base saturation (BS) after cotton harvesting and treatments (Treat.).

Soil depth (m)	SV	DF	Mean square							
			pH <sub>H2O</sub>	OM (dag kg <sup>-1</sup> )	P (mg dm <sup>-3</sup> )	K (mg dm <sup>-3</sup> )	Fe (mg dm <sup>-3</sup> )	Na (cmol <sub>c</sub> dm <sup>-3</sup> )	Ca (cmol <sub>c</sub> dm <sup>-3</sup> )	BS (%)
0-0.2	Block	3	0.100	0.233	2.187	1168.18	2.83	0.0018	0.148	17.12
	Treat.	4	0.379*	0.048 <sup>ns</sup>	0.701 <sup>ns</sup>	3070.25*	17.04 <sup>ns</sup>	0.005*	0.376*	95.45*
	Res.	12	0.088	0.096	1.72	721.18	7.09	0.001	0.104	19.62
CV (%)			4.48	21.55	36.97	19.36	14.90	25.30	9.15	5.80
0.2-0.4	Block.	3	0.009	0.107	0.741	733.13	1.61	0.000	0.251	24.05
	Treat.	4	0.117 <sup>ns</sup>	0.063 <sup>ns</sup>	0.107 <sup>ns</sup>	1252.13 <sup>ns</sup>	5.89*	0.004*	0.133 <sup>ns</sup>	45.33 <sup>ns</sup>
	Res.	12	0.071	0.027	0.276	981.93	1.71	0.001	0.110	33.43
CV (%)			4.14	20.26	30.65	25.37	8.84	29.46	11.47	8.46
0.4-0.6	Block	3	0.038	0.086	1.606	396.98	0.958	0.0005	0.193	15.73
	Treat.	4	0.153 <sup>ns</sup>	0.044 <sup>ns</sup>	1.437 <sup>ns</sup>	953.88 <sup>ns</sup>	1.827 <sup>ns</sup>	0.005**	0.247 <sup>ns</sup>	66.93 <sup>ns</sup>
	Res.	12	0.241	0.032	0.977	543.94	2.357	0.0005	0.341	73.69
CV (%)			8.11	38.21	70.35	30.00	10.19	19.44	20.78	13.69
0.6-0.8	Block	3	0.027	0.054	0.926	82.73	1.02	0.0005	0.113	5.13
	Treat.	4	0.117 <sup>ns</sup>	0.032 <sup>ns</sup>	1.687 <sup>ns</sup>	469.30 <sup>ns</sup>	1.07 <sup>ns</sup>	0.0005 <sup>ns</sup>	0.117 <sup>ns</sup>	62.38 <sup>ns</sup>
	Res.	12	0.223	0.017	0.826	240.73	1.34	0.0005	0.351	58.18
CV (%)			8.10	43.87	65.39	26.17	7.89	21.30	21.86	12.20

\*Significant at 5%, \*\* significant at 1%, and <sup>ns</sup>not significant based on an F test.

**Table 6.** Summary of analysis of variance for the soil electrical conductivity of saturated paste extract ( $EC_e$ ), water-dispersible clay (WDC), exchangeable sodium percentage (ESP), and sand, silt, and clay levels, after cotton harvesting and treatments (Treat.).

Soil depth (m)	SV	DF	Mean square					
			$EC_e$ (dS m <sup>-1</sup> )	WDC (dag kg <sup>-1</sup> )	ESP (%)	Sand (dag kg <sup>-1</sup> )	Silt (dag kg <sup>-1</sup> )	Clay (dag kg <sup>-1</sup> )
0-0.2	Block	3	0.04	7.83	0.48	26.53	18.18	8.85
	Treat.	4	0.04 <sup>ns</sup>	23.65 <sup>**</sup>	0.92 <sup>*</sup>	4.80 <sup>ns</sup>	20.08 <sup>ns</sup>	17.18 <sup>ns</sup>
	Res.	12	0.02	3.65	0.18	9.20	33.48	37.98
CV (%)			28.82	18.41	24.52	5.86	26.12	23.66
0.2-0.4	Block	3	0.03	6.22	0.04	7.87	5.33	3.07
	Treat.	4	0.03 <sup>ns</sup>	20.53 <sup>*</sup>	0.99 <sup>ns</sup>	8.45 <sup>ns</sup>	7.95 <sup>ns</sup>	9.63 <sup>ns</sup>
	Res.	12	0.01	5.12	0.37	8.28	8.25	14.36
CV (%)			24.40	16.08	32.39	5.92	15.61	11.48
0.4-0.6	Block	3	0.037	2.86	0.18	8.98	4.18	10.73
	Treat.	4	0.04 <sup>**</sup>	22.57 <sup>*</sup>	0.88 <sup>**</sup>	5.83 <sup>ns</sup>	3.83 <sup>ns</sup>	5.80 <sup>ns</sup>
	Res.	12	0.005	5.52	0.12	5.86	28.23	30.73
CV (%)			19.31	16.79	19.59	5.48	27.74	15.11
0.6-0.8	Block	3	0.062	1.67	0.06	9.27	22.40	16.20
	Treat.	4	0.008 <sup>ns</sup>	8.94 <sup>ns</sup>	0.14 <sup>ns</sup>	0.83 <sup>ns</sup>	4.63 <sup>ns</sup>	6.58 <sup>ns</sup>
	Res.	12	0.015	7.38	0.12	3.39	17.86	23.91
CV (%)			37.26	17.25	19.82	4.29	23.48	12.51

\*Significant at 5%, \*\*significant at 1%, and <sup>ns</sup>not significant based on an F test.

particles (Matos et al., 2013; Souza et al., 2010; Travis et al., 2010). The average WDC decreased 37.6% compared to T0 up to 0.6 m depth, which is useful for soil. Given these results, PTW can be used to remediate soils with clay dispersion problems.

## Materials and Methods

### Experimental site, pre-treatment operations, crop and irrigation management

The experiment was implemented in April 2012 at the Copasa/Unimontes experimental site (Unimontes - Universidade Estadual de Montes Claros, the State University of Montes Claros), located next to Janaúba STP, MG, Brazil (15° 46' 12.6" S latitude, 43° 19' 13.5" W longitude, altitude of 530 m) in a pasture area that had degraded for more than five years. The Köppen climate classification is Aw, tropical with a dry winter. The soil is classified as a eutrophic Red Latosol (Oxisol) (Embrapa, 2013), and the baseline soil attributes are given in Table 2. After weeds were removed, the fields were spread with 1,500 kg ha<sup>-1</sup> of dolomite lime with effective calcium carbonate equivalent (ECCE) of 104.45% at 41 days before sowing (Ribeiro et al., 1999). On the following day, two cross subsoilings incorporated lime with rods spaced 0.5 m apart, up to 0.65 m depth, taking advantage of soil moisture content derived from 48 mm of rainfall over the previous four days. At 38 days before sowing, two cross harrowings were performed. This sequence was employed for soil loosening and levelling, lime incorporation, and weed control. On April 30, 2012, cotton was sown after the soil moisture reached field capacity (Table 2), applying 62.7 mm of clean water divided into three irrigations. The cotton cultivar used was NuOPAL BG RR (Monsanto Co. ®), an early-to-medium cycle cultivar with medium fibre and resistance to *Lepidoptera* and the herbicide glyphosate. The sowing was manual, with 20 seeds per linear metre (70% germination, 40% vigour) and 0.9 m between rows, at a depth of 0.03 m. Delinted seeds were treated with Imidacloprid 600 FS (imidacloprid, 4.5 mL kg<sup>-1</sup> of the commercial product - cp) and Cercobin 500 SC (methyl

thiophanate, 3.0 mL kg<sup>-1</sup> cp) to prevent damage from *Aphis gossypii*, *Frankliniella schultzei*, and *Colletotrichum gossypii* var. *cephalosporioides*. In the furrow sowing, 100.0 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup> (333.3 kg ha<sup>-1</sup> of a NPK 4-30-10) was applied, complemented with 20.0 kg N ha<sup>-1</sup> (14.8 kg urea ha<sup>-1</sup>) and 40.0 kg K<sub>2</sub>O ha<sup>-1</sup> (11.6 kg KCl ha<sup>-1</sup>), according to Ribeiro et al. (1999). The crop cycle began on April 5, 2012, after more than 80% seedling emergence, and the plants were thinned to nine plants per linear metre (equivalent to 100,000 plants ha<sup>-1</sup>). The applications of wastewater and water were performed using a semi-automated drip irrigation system with a filtering mechanism composed of two sand filters with a slow filtration rate equal to 6.1 m<sup>3</sup> m<sup>-2</sup> h<sup>-1</sup> and a 120 mesh disc filter, a 2" hydrometer, a 2" retention valve, a fertilizer hydraulic injector, glycerine manometers, and pressure taps. Although only one irrigation system was used, wastewater and water were applied individually, and the tubing was cleaned between each water application, when the water was discharged into two sinks; the first sink was located next to the filtration system for backwashing, and the second was located at the end of the experimental site. A 5.9-m-long lateral row was established for each row of plants, which were spaced 0.9 m apart. Water applications were performed with non-self-compensating cylindrical Naantif® drip emitters with an inner diameter of 0.014 m and a pre-filter, at a mean flow rate (q<sub>e</sub>) of 5.87 L h<sup>-1</sup> at 204 kPa pressure, with emitters spaced every 0.4 m. Irrigation management was conducted with a two-day watering interval and aided by the software Irriplus®. The water demands (evapotranspiration) of cotton (ETc) were determined using the Food and Agriculture Organization of the United Nations' Penman-Monteith (FAO-PM) model (Allen et al., 2006) to calculate ETo. The application efficiency (Ea) was estimated to be 96.0% from assessments of field irrigation uniformity (Bernardo et al., 2006). With the ETc, Ea, and q<sub>e</sub> results, the net irrigation depth, gross water depth accumulated during the irrigation schedule, and working time requirement of the irrigation system were calculated. After the applications, the net wastewater irrigation requirements were recalculated based on the irrigation schedule. At 9 and 57 DAE, 12 hours after the last irrigation, trenches were opened at the end of three

random lateral rows, and the width of the zone wet by the emitters was determined with a measuring tape every 0.05 m of depth. The mean percentage of wetted area was calculated by dividing the maximum wetting width by the space between lateral rows (Merriam and Keller, 1978). The mean wetted area was 50%. At 133 DAE, when 55% of the bolls were open and 30% were physiologically mature, irrigation was discontinued (Beltrão and Azevedo, 2008). The water for irrigation was supplied by the network of the Companhia de Saneamento de Minas Gerais - COPASA - MG, the sanitation company of Minas Gerais, in Janaúba, Minas Gerais State, Brazil, from the Bico da Pedra reservoir. The TTW was captured by graded conduction via 200 m of PVC pipe from the second maturation pond of COPASA STP (the last treatment stage before release into the Gorutuba River) to a pump installed at the experimental site. The PTW, conducted in the same manner, was captured after the flowmeter in a preliminary treatment system.

### ***Treatments, experimental design and topdressing***

The experiment followed a randomized complete block design with five treatments and four replications, blocking homogeneous soil conditions. The crop fertigation treatments were clean water and mineral fertilizer topdressing (T0); TTW as a potassium source for topdressing, equivalent to 40 kg K<sub>2</sub>O ha<sup>-1</sup> (T1) and 60 kg K<sub>2</sub>O ha<sup>-1</sup> (T2); and PTW as a potassium source for topdressing, equivalent to 40 kg K<sub>2</sub>O ha<sup>-1</sup> (T3) and 60 kg K<sub>2</sub>O ha<sup>-1</sup> (T4). Public water supply was used to meet the remaining crop water requirement. The treatments started at 35 DAE, using the irrigation system to apply wastewater or topdressing fertilizer to the control. A total of 22 wastewater irrigations were performed between 35 and 100 DAE (average of 2.4 irrigations per week). In the control, six mineral fertilizers were applied as topdressing, supplying a total of 50.0 kg N ha<sup>-1</sup> (111.1 kg urea ha<sup>-1</sup>) and 40.0 kg K<sub>2</sub>O ha<sup>-1</sup> (69.0 kg KCl ha<sup>-1</sup>), according to Ribeiro et al. (1999). These same N and K<sub>2</sub>O application doses were used to calculate the wastewater volume applied in the treatments, except that T1 received an additional dose of 15.5 kg N ha<sup>-1</sup> as urea at 76 DAE to achieve the recommended dose.

### ***Sampling and wastewater analysis***

Samples of wastewater were collected monthly from the end of one of the lateral rows during applications. The samples were divided into different containers and acidified (pH < 2) with H<sub>2</sub>SO<sub>4</sub> (N), HNO<sub>3</sub> (K, Na, Ca, and Fe), and HCl (O&G). Samples for BOD analysis were placed in Winkler bottles and immediately evaluated. The pH and EC were also measured immediately after sampling. All samples were taken in polystyrene boxes with ice to a laboratory to determine total N, ammoniacal N, nitric N, total P, and total K, Na, Ca, Cu, Fe, and B, according to Apha et al. (2012). With the results of the wastewater analyses from the previous month, the wastewater fertigation requirements in the respective treatments were calculated.

### ***Variables and statistical analysis***

Within the useful plot area, four soil samples were randomly collected from the wetted zone by the emitters at a distance of approximately 0.05 m from the plant rows, from the 0-0.2 m and 0.2-0.4 m depths at 30 DAE and from the 0-0.2 m, 0.2-0.4 m, 0.4-0.6 m, and 0.6-0.8 m depths at 149 DAE (13 days after harvest). The four samples were combined to form a

composite sample for determining water-dispersible clay (WDC), soil electrical conductivity (EC<sub>e</sub>), particle size (sand, silt, and clay), soil pH (in water), organic matter (OM), P, K, H+Al, Na, Ca, Mg, and Fe, according to Donagema et al. (2011). The base sum, base saturation (BS), and exchangeable sodium percentage (ESP) were also determined. The values within each layer were subjected to analysis of variance and mean tests for within-treatment comparisons (Tukey's test,  $p \leq 0.05$ ) and to comparisons between treatments and the control (Dunnnett's test,  $p \leq 0.05$ ) using the software SAEG 9.1 (SAEG Sistema para Análises Estatísticas, Version 9.1: Fundação Arthur Bernardes - UFV - Viçosa, 2007).

### **Conclusions**

The application of preliminary treated wastewater (PTW) and tertiary treated wastewater (TTW) to reach potassium topdressing requirements for cotton did not promote chemical excesses up to 0.8 m soil depth. The application of TTW equivalent to a dose of 60 kg K<sub>2</sub>O ha<sup>-1</sup> increased sodium content up to 0.6 m depth, ESP in the 0-0.2 and 0.4-0.6 m depth layers, and EC<sub>e</sub> in the 0.4-0.6 m depth layer, which may compromise the soil quality and/or groundwater after repeated applications. The application of PTW equivalent to a dose of 60 kg K<sub>2</sub>O ha<sup>-1</sup> decreased soil pH in the top 0.2 m and water-dispersible clay up to 0.6 m depth. A general recommendation is to use PTW when providing 60 kg K<sub>2</sub>O ha<sup>-1</sup> in topdressing.

### **Acknowledgements**

The authors thank Companhia de Saneamento de Minas Gerais - COPASA, the sanitation company of Minas Gerais; Banco do Nordeste do Brasil - BNB/ETENE/FUNDECI, Brazil's northeast bank; Associação Mineira dos Produtores de Algodão - AMIPA, the Minas Gerais Association of Cotton Producers; and Fundação de Amparo à Pesquisa do Estado de Minas Gerais - FAPEMIG, the Minas Gerais State Research Foundation, for financial support and providing grants.

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