

Research article

Assessing potassium release in natural silica sand from novel K-enriched sewage sludge biochar fertilizers

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ABSTRACT

Enrichment of biochars to produce slow nutrient release fertilizers with minimal losses to the environment is a promising strategy. However, the release of potassium (K) from biochar-based fertilizer produced from sewage sludge (SS) is still poorly studied. In the present 30-day incubation study, the dynamics of K release were evaluated from SS biochar-based fertilizers enriched with potassium chloride (KBF) in different forms, subjected to two levels of silica sand moisture (10 and 20%). The KBF was evaluated in the form of granules, pellets and powder, in addition to pure KCl mineral fertilizer. During the incubation period water-soluble K extractions were performed, where the K contents were adjusted to K release kinetic models. An additional experiment was performed to assess the effect of KBFs and KCl on K leaching. In general, at both moisture levels all KBFs presented a slower K release compared to pure KCl mineral fertilizer, reducing the release rate by up to 77%. The K release dynamics were affected by the type of biochar fertilizer (granule, pellet and powder) and the silica sand moisture level. The behavior of KBFs as slow-release fertilizers is strongly dependent on the silica sand moisture level. At the 10% moisture level, biochar fertilizers in the form of pellets and granules can be classified as slow-release fertilizers with the potential to increase the efficiency of K use in agriculture. Furthermore, compared to the chemical fertilizer, KBF reduced the amount of leached K, diminishing the risk of this nutrient polluting the groundwater. Our results must be further assessed in real conditions using soil as a suitable medium for agronomic and environmental evaluation. Therefore, future studies should consider the dynamic of K and other nutrients from KBFs in distinct soil types.

1. Introduction

Pyrolysis has been used in the treatment of sewage sludge (SS). The solid product of SS pyrolysis, called SS biochar (SSB), can contribute to the recycling of SS in agriculture, providing significant amounts of nutrients such as phosphorus (P), nitrogen (N), calcium (Ca) and zinc (Zn) (Paz-Ferreiro et al., 2018). However, low potassium levels (K) are normally found in SSB which may limit its use in agriculture. The SS raw material has low K contents resulting from the sewage treatment process, where K is eliminated in the form of soluble salts along with the liquid effluent (Kirchmann et al., 2016). Thus, as a consequence of the low K concentration in the SS, SSBs typically do not have adequate K contents to meet crop demands (Faria et al., 2018; Fachini et al., 2021a) and it is necessary to complement with mineral fertilizer. Previous studies with biochars from other raw materials have also reported the need to apply mineral fertilizers combined with biochars for a complete

and balanced supply of nutrients to plants (Subedi et al., 2016; Arif et al., 2017; Shin et al., 2019).

The use of nutrient-enriched biochars has increased in recent years with the objective of enabling and balancing the supply of nutrients to plants. Enriching biochars to produce slow nutrient release fertilizers is one of the strategies used in recent years (Carneiro et al., 2021). When compared to highly soluble mineral fertilizers, biochar-based fertilizers contribute to increase the fertilizer use efficiency (Lustosa Filho et al., 2020). This is possible because biochar-based fertilizers can reduce nutrient losses via leaching (Luo et al., 2021) and the use of soluble mineral sources, in addition to contributing to increased nutrient uptake and crop productivity (Borges et al., 2020).

The surfaces of biochars are mainly characterized by negatively charged functional groups, such as carboxyl and phenolic, which can contribute to greater sorption of cations such as K (Mukherjee et al., 2011). Therefore, K-enriched SSB can act as a slow-release fertilizer and

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minimize K losses by leaching. Furthermore, the organic fraction of biochar and the presence of pores in its structure can contribute to protect the more soluble fractions of the mineral fertilizer (Kim et al., 2014; Luo et al., 2021). This minimizes its losses and provides a better synchronization of nutrient release with the plant's nutritional demand, increasing the efficiency of the applied fertilizer (Adu-Gyamfi et al., 2019). Thus, the enrichment of SSB with mineral K sources may result in a slow K release biochar-based fertilizer, resolving the low supply of this nutrient with the application of pure SSB, in addition to reducing K losses from highly soluble sources. In general, weathered soils from tropical regions have low cation exchange capacity. As a consequence, K losses by leaching are common in these regions. Therefore, slow-release fertilizers are crucial for increasing K use efficiency in agriculture.

Studies on biochar-based fertilizers are relatively recent. Therefore, there is a wide variation in the methodologies used to obtain the final product (Karim et al., 2016; Farrar et al., 2018). Biochar granulation and pelleting processes are the most common for nutrient enrichment and have been used as a way to minimize the risk of dust and respiratory problems caused by the application of biochar in its powder form (Vincevica-Gaile et al., 2019). In the case of SSB, to date there are no published studies evaluating the release and availability of nutrients from SSB-based fertilizers enriched with soluble K sources. Furthermore, enrichment technologies can affect the release of nutrients by the fertilizer as well as its agronomic efficiency, and they need to be better studied. Soil is the most appropriate medium to assess nutrient release from fertilizers. However, in this preliminary study, silica sand was used as a pure medium to avoid the influence of the soil matrix on K sorption (Adams et al., 2013).

The present study sought to evaluate the dynamics of K release from K-enriched biochar fertilizers (KBFs), subjected to distinct silica sand moisture levels. For this, the following hypotheses were tested: i) K release dynamics are affected by the physical form of the KBF; ii) KBFs act as K slow-release fertilizers regardless the silica sand moisture level.

2. Materials and methods

2.1. Production of sewage sludge biochar (SSB)

The SSB was produced from SS samples collected at a sewage treatment plant (STP). This STP uses a tertiary treatment system, in which sewage decomposition is carried out in an anaerobic up-flow reactor (UASB). In this type of system, nutrients such as P and N remain in the final biomass of the SS because they are removed from the liquid effluent by a coagulation process with aluminum salts. To prepare the SSB, SS samples were air dried (to a moisture content of approximately 10%), passed through a 4 mm sieve and then pyrolyzed at 300 °C. Based on our previous study (Figueiredo et al., 2018), considering all variables together, the biochar produced at 300 °C showed the greatest nutrients availability. Pyrolysis was performed in a muffle furnace (Linn ElektroTherm, Eschenfelden, Germany) with an average temperature increase of 2.5 °C min⁻¹ and residence time of 5 h as reported by Figueiredo et al. (2018). The furnace was equipped with a mechanism to prevent oxygen flow (via a forced draft fan, assisting in the exit of gases and oil vapors from the furnace). The physicochemical characteristics of SS and SSB are presented in Table 1. The methodologies used in characterization of the SS and SSB are described in our previous work (Fachini et al., 2021b).

2.2. Production of K-enriched biochar fertilizers

Details of the production and characterization of KBFs are described in our previous study (Fachini et al., 2021b). In summary, fertilizers were produced from a physical mixture of SSB, KCl and pregelatinized corn starch (binding agent). The raw materials were mixed to obtain a final mass of 1.0 kg for each fertilizer. Considering the concentrations of total nitrogen (TN) and total P₂O₅ in the SSB (Table 1), the final

Table 1

Characteristics of sewage sludge (SS) and sewage sludge biochar (SSB).

Property ^a	SS	SSB
pH (CaCl ₂)	6.4 ± 0.05	6.5 ± 0.02
Moisture content (%)	10.4 ± 0.11	8.4 ± 0.03
Volatile matter (%)	55.7 ± 0.32	47.2 ± 0.49
Ash (%)	32.4 ± 0.17	38.1 ± 0.18
Fixed carbon (%)	1.5 ± 0.19	6.3 ± 0.64
OC (%)	18.3 ± 0.11	18.3 ± 1.06
TC (%)	25.1 ± 0.17	27.1 ± 0.08
TN (%)	3.9 ± 0.17	4.3 ± 0.13
P ₂ O ₅ total (%)	4.7 ± 0.07	5.5 ± 0.08
K ₂ O total (%)	0.05 ± 0.01	0.04 ± 0.01
Ca (g kg ⁻¹)	6.0 ± 0.03	6.9 ± 0.04
Mg (g kg ⁻¹)	1.40 ± 0.2	1.54 ± 0.01
S (g kg ⁻¹)	1.30 ± 0.4	1.62 ± 0.01
Fe (g kg ⁻¹)	17.5 ± 1.06	20.0 ± 2.1
B (mg kg ⁻¹)	26.4 ± 3.0	31.2 ± 1.1
Mn (mg kg ⁻¹)	88.6 ± 4.2	100.0 ± 11.3
FA (g kg ⁻¹)	35.0 ± 0.85	33.7 ± 0.82
HA (g kg ⁻¹)	14.9 ± 0.55	9.3 ± 1.60
HU (g kg ⁻¹)	179.1 ± 0.58	229.4 ± 8.08
SA (m ² /g ⁻¹)	21.90	27.80
PV (cm ³ /g ⁻¹)	0.096	0.102

^a Average values ± standard deviation (n = 3); OC = organic carbon; TC = total carbon; TN = total nitrogen. FA = fulvic acid; HA = humic acid; HU = humin; SA = specific surface area; PV = pore volume.

concentration of K in KBF was set at 3% to meet legal requirements, where the sum of NPK in organomineral fertilizers should be greater than 10% (Brazil, 2018). Thus, 50 g of KCl were added to provide 3% K₂O in the fertilizers. As a binding and complexing agent, 65 g of pre-gelatinized starch were added for the production of granules and pellets. As a source of N and P, SSB was added to complete 1.0 kg of the formulation.

KBF samples were ground in an industrial device (Philco PLQ 1400), and subsequently passed through a 0.500 mm mesh sieve. The SSB + KCl + starch (KBF) formulation was subjected to granulation and pelletization processes. Furthermore, the powdered blend of starch-free raw materials was studied as KBF in a powder form.

The pellets were produced in a laboratory pelletizer (ENG-MAQ, model ENG 0200V, São Paulo, Brazil). Pellets measuring 6 mm in diameter and 1 cm long were produced. The granules were produced in a laboratory granulator composed of a cylindrical tray measuring 7 cm high by 36 cm in diameter, inclined at an angle of 70°, coupled to a motor operating at 40 rpm. Granules were passed through a set of 3 sieves with different meshes, in sequence: 4 mm, 2 mm and 1 mm. In the present study, 1–4 mm granules were selected.

2.3. Characterization of K-enriched biochar fertilizers

The specific surface area (SA), pore volume (PV) and micropore volume (MPV) of the KBFs were determined by means of N₂ adsorption isotherms at -196.2 °C in a NOVA 2200 surface area analyzer (Quantachrome Corp., Boynton Beach, FL, USA), and calculations were performed automatically by the Quantachrome NovaWin® software when applying the BET equation (Brunauer, Emmett and Teller).

The particle density (Dp) (g cm⁻³) of the granules and pellets was determined according to the British Standard Method (British, 1995). The individual volume of 10 granules and 10 pellets of the same particle size was calculated with the aid of a digital caliper, then the mass of each was determined on a precision scale. Subsequently, the Dp calculation was performed according to equation (1) (Eq. (1)):

$$Dp = \frac{m}{v} \quad (1)$$

where, Dp = granule or pellet density (g cm⁻³); m = mass of the granule or pellet (g); v = volume of the granule or pellet (cm⁻³).

The other physicochemical characteristics of KBFs are presented in Table 2. Methodologies used in the characterization of KBFs, as well as production and characterization details are described in our previous work (Fachini et al., 2021b).

2.4. Incubation study

To assess the K-release dynamics from KBFs, an incubation experiment was performed as described in Bley et al. (2017). Natural silica sand (SiO_2), with fine granulometry (<0.2 mm and >0.06 mm) according to the ABNT classification (NBR 6502/1995), was used to ensure that the released K levels resulted only from the studied fertilizers as previously adopted by Luo et al. (2021). Silica sand presented the following characteristics: apparent density (1.15 g cm^{-3}); Moisture content (0.60%); SiO_2 (97.0%); Al_2O_3 (1.50%); Fe_2O_3 (0.30%); and Other unspecified components (0.60%).

In the incubation assay, silica sand was placed in 200 cm^3 plastic containers. In each container 100 g of silica sand and 5 g of each KBF were added. In addition to KBFs, pure KCl mineral fertilizer was also evaluated. The KBFs used in this study contained 3% K (Table 2). Thus, 5 g of each material was applied to silica sand to provide 1500 mg kg^{-1} of K_2O , which corresponds to 1245 mg kg^{-1} of K. To ensure the same mass of K in the treatment with mineral fertilizer, 0.25 g of KCl mineral fertilizer (60% K_2O) was added to 100 g of silica. The same KCl source used to enrich the KBFs was used, presenting analytical purity that meets the purity standards defined by the American Chemical Society (ACS).

The KBFs and KCl were placed in containers with permeable polyester mesh <0.500 mm, measuring 5×4 cm and sealed at the ends with 100% polyester thread, referred to as “sachets”. All sachets were sealed at the end with polyester thread. The sachets containing the fertilizers were placed in plastic bottles, previously filled with 50 g of silica sand and covered with another 50 g of silica sand (Fig. 1).

Two levels of moisture in silica sand were tested. The moisture level of 10% corresponds to the moisture content of sandy soils at field capacity (Laboski et al., 1998). A soil water content of 20% was also chosen to simulate a moisture level with the potential to promote K losses by leaching in sandy soils. Thus, the total mass of 100 g of silica sand was moistened with distilled water, to reach 10 and 20% water, which corresponded to 0.1 g g^{-1} and 0.2 g g^{-1} , respectively. Moisture

Table 2
Characteristics of K-enriched biochar fertilizers (KBFs).

Property ^a	KBFs		
	Granule	Pellet	Powder
pH (CaCl_2)	5.73 ± 0.05	5.77 ± 0.02	6.65 ± 0.05
Moisture content (%)	7.14 ± 0.27	4.79 ± 0.44	7.31 ± 0.8
Volatile matter (%)	51.11 ± 0.63	52.90 ± 1.22	44.72 ± 0.76
Ash (%)	39.58 ± 0.14	38.74 ± 1.09	40.86 ± 0.26
Fixed carbon (%)	1.91 ± 0.45	3.90 ± 0.04	6.60 ± 0.67
OC (%)	17.64 ± 0.55	19.48 ± 1.28	17.32 ± 0.42
TC (%)	25.31 ± 0.40	25.50 ± 1.49	21.76 ± 1.95
TN (%)	3.02 ± 0.12	3.66 ± 0.31	3.17 ± 0.51
C/N	8.25 ± 0.40	7.77 ± 0.62	6.92 ± 0.18
P_2O_5 total (%)	5.21 ± 0.14	5.22 ± 0.13	5.65 ± 0.05
K_2O total (%)	3.0 ± 0.02	3.0 ± 0.03	3.0 ± 0.04
Ca (g kg^{-1})	5.57 ± 0.001	5.57 ± 0.001	6.74 ± 1.0
Mg (g kg^{-1})	1.63 ± 0.001	1.42 ± 4.7	1.56 ± 4.3
S (g kg^{-1})	1.24 ± 0.001	1.3 ± 2.5	1.6 ± 5.0
Fe (g kg^{-1})	17.3 ± 0.001	18.3 ± 1.0	20.1 ± 6.2
B (mg kg^{-1})	25.6 ± 0.4	27.2 ± 1.1	29.6 ± 0.3
Mn (mg kg^{-1})	89.0 ± 2.2	89.0 ± 0.3	102.0 ± 1.6
FA (g kg^{-1})	33.85 ± 0.22	33.20 ± 0.32	33.21 ± 0.47
HA (g kg^{-1})	11.08 ± 0.19	11.86 ± 0.64	10.98 ± 1.83
HU (g kg^{-1})	204.83 ± 3.34	211.51 ± 6.54	176.88 ± 3.41

^a Average values $\pm$ standard deviation ($n = 3$); OC = organic carbon; TC = total carbon; TN = total nitrogen. FA = fulvic acid; HA = humic acid; HU = humin.

was monitored every 5 days by assessing the total mass of the flasks using a precision scale model AY220 (Shimadzu), and when necessary distilled water was added to maintain a constant water content in the silica sand. Flasks containing the silica sand and treatments were incubated in an incubation room equipped with a temperature and humidity controller. Incubation was conducted at an average temperature of 25°C , and the following periods were assessed: 12 h and 1, 2, 3, 4, 5, 10, 20 and 30 days after initiating the incubation. After the incubation period established for each treatment, the flasks were removed from the incubation room, separating the sachet from the silica sand. The water-soluble K content released in the silica sand mass was then determined.

2.5. Determination of the K concentrations

After removing the sachets, the mass of silica sand from the container was air-dried and homogenized. The silica sand samples were then analyzed and the K contents determined. The K was extracted in a mixture with distilled water in a 1:10 (w/v) silica sand: distilled water ratio (Wang et al., 2018).

For the extraction of K, 1 g of silica sand and 9 mL of distilled water were mixed in a 50 mL beaker, then the mixture was agitated for about 5 min at 150 rpm on a horizontal circular agitation table, model Tec-1401 (Tecnal, Piracicaba, SP). Next, the mixture decanted for 30 min until finally the extract was filtered and the K reading was taken in a digital flame photometer (Benfer PHS, São Paulo, SP, Brazil).

2.6. Evaluated treatments

The following treatments were evaluated, with three replications: 1) control: only silica sand without fertilizer addition, 2) granulated KBF, 3) pelletized KBF, 4) KBF powder and 5) powder KCl mineral fertilizer. The treatments were submitted to two moisture levels (10 and 20%) and evaluated in 9 incubation periods.

2.7. Potassium release kinetics

After the determination of K, the K release rate (%) during the incubation period was calculated according to equation (2) (Eq. (2)).

$$\text{K release (\%)} = \left(\frac{\text{Kt}}{\text{Kt0}} \right) \times 100 \quad (2)$$

where, Kt = K content released during the incubation period (mg kg^{-1} K); Kt0 = total initial K content (mg kg^{-1}) added to the sachet.

Next, the K release data (%) was submitted to non-linear regression analysis to determine the K release dynamics over time. Different models were tested to best represent the data, using the SigmaPlot 12.5 program. According to the values of the R^2 coefficient and the P value (<0.0001), the best model to represent the K release data was the first-order exponential equation (Eq. (3)).

$$y = a(1 - b^x) \quad (3)$$

where, y = K release (%) and x = incubation time (days).

2.8. KBF as a slow-release fertilizer

The fitted equation (Eq. (3)) was used to determine the release dynamics of the fertilizers. According to the Association of American Plant Food Control Officials (AAPFCO, 1997), a slow- or controlled-release fertilizer is a fertilizer containing a plant nutrient in a form which delays its availability for plant uptake and use after application, or which extends its availability to the plant significantly longer than a reference ‘rapidly available nutrient fertilizer’ such as ammonium nitrate or urea, ammonium phosphate or potassium chloride. In addition, according to regulations of the European Committee for Standardization (CEN),

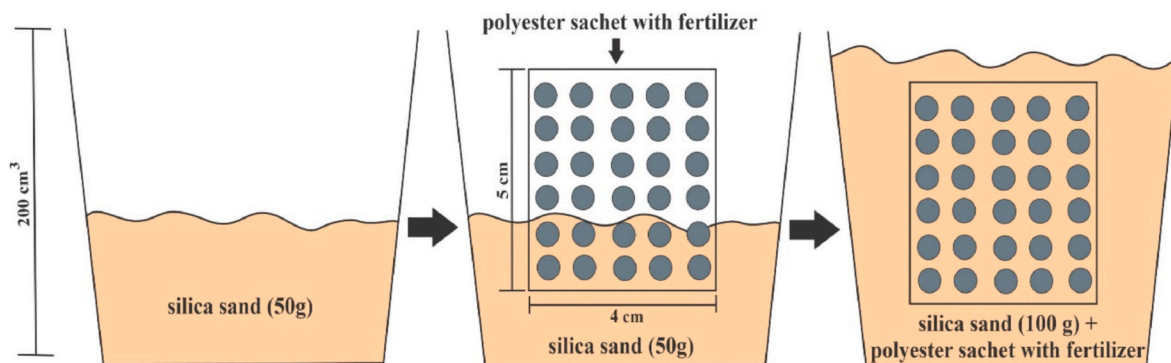


Fig. 1. Schematic representing the 200 cm³ plastic containers with 100 g of silica sand and polyester sachet containing the fertilizer.

fertilizer may be described as slow-release if the nutrient or nutrients declared as slow-release meet, under defined conditions, each of the following criteria (Trenkel, 2010): i) no more than 15% released in 24 h; ii) no more than 75% released in 28 days.

2.9. Column leaching experiment

Leaching of K from fertilizers (KBFs and KCl) in natural silica sand was assessed during a 6-day trial. The column leaching experiment, with 3 replications, was carried out in polyvinyl tubes (20 cm length x 5.0 cm diameter), with a barrier made of filter paper fitted near the base (Yuan et al., 2016). A valve was inserted at the base for the control of leachate collection (Fig. 2). The columns were packed with 375 g of silica sand. The same amount of KBF and KCl used in the incubation experiment was added into the columns (5 g of KBFs and 0.25 g of KCl). Distilled deionized water was added gently across the surface of the column. At the start of each experiment, the columns were saturated by the addition of 130 mL of distilled deionized water, and then they were immediately drained. Each column was leached once a day with 30 mL of the distilled deionized water, and a total of 180 mL of water was added to the columns over the 6-day experimental period. This volume corresponds to the average precipitation in February in the Central region of Brazil. The leachates were filtered via filter paper (JP42, Prolab) and the K concentration was determined using a digital flame photometer (Benfer PHS, São Paulo, SP, Brazil). The effect of the treatments (fertilizers) on the cumulative amount of leached K was tested by a one-way ANOVA, followed by the Tukey test ($P < 0.05$).

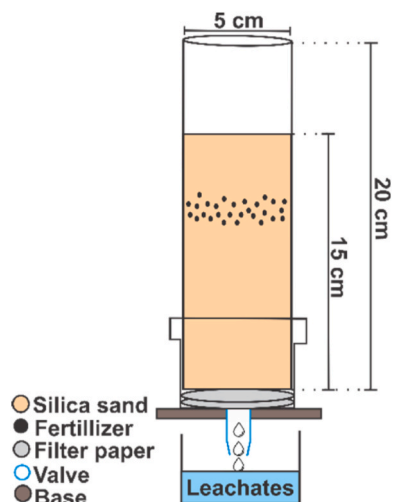


Fig. 2. Schematic of the experimental setup used in the column leaching test.

3. Results and discussion

3.1. K release kinetics

The dynamics of K release from KBFs and pure KCl at the two different moisture levels is shown in Fig. 3. For all fertilizers, the first-order exponential model adequately described the K release process with high significance ($P < 0.0001$) and a R^2 value greater than 0.85. The first-order kinetic equation also provided the best correlation to describe the release mechanism of K into water and soil in the work conducted by Pang et al. (2018).

For the two moisture levels, 10% (Figs. 3A) and 20% (Fig. 3B), all KBFs showed slower K release compared to pure KCl fertilizer. Among the KBFs, the K release rate followed the order of powder > granule > pellet, regardless of the moisture content. For the 10% moisture level, the KCl fertilizer showed a rapid K release, totaling 1078.4 mg kg⁻¹ (86.6%) of K released after the first day (Fig. 3A). Afterwards, the release remained constant until the end of the solubilization process. On the other hand, the enriched fertilizers showed slower K release than KCl, suggesting that KBF fertilizer has the ability to control K release. The enriched fertilizer types also presented different K release rates in the initial incubation period. Until the fourth day of incubation, the release was 501.4, 605.2 and 703.0 mg kg⁻¹ of K for the pellet, granule and powder, respectively. In the final incubation phase, at 30 days, KBF in the pellet, granule and powder form released only 54, 65 and 78% respectively, while the KCl fertilizer released 100% of K by the twentieth day of incubation.

The two moisture levels showed similar K release dynamics from the fertilizers. At the 20% moisture level, in just 1 day of incubation the KCl fertilizer released almost 90% of K, reaching 1049.30 mg kg⁻¹ of K on the second day of the experiment (Fig. 3B). From the second day onwards, release remained practically constant. At this same moisture level, the accumulated release of K by the enriched fertilizers at 30 days was 65, 77 and 83% for the pellet, granule and powder, respectively. This greater presence of water anticipated by 10 days the total release (100%) of K from the mineral fertilizer KCl (1245 mg kg⁻¹ of K), proving that the rate of K solubilization is very dependent on the soil moisture level. Increasing the moisture level from 10 to 20% also increased K release from KBFs, with increases of 19, 18 and 5% for the pellet, granule and powder, respectively. The fertilizer nutrient release mechanism depends, among other factors, on the fertilizer solubility in solution, granule physical form and soil/substrate water content (Adms et al., 2013). KCl has a high water solubility, around 300 g/L (Coelho et al., 1994). In the present work, a greater supply of water in silica resulted in a higher rate of K release from fertilizers considering the same period of time under the same conditions of temperature, substrate and fertilizer doses. According to Du et al. (2006), the highest K release rate was obtained when the fertilizer was immersed in water, followed by water-saturated silica and finally, the lowest release rate was verified when the fertilizer was released onto silica with the water content at the

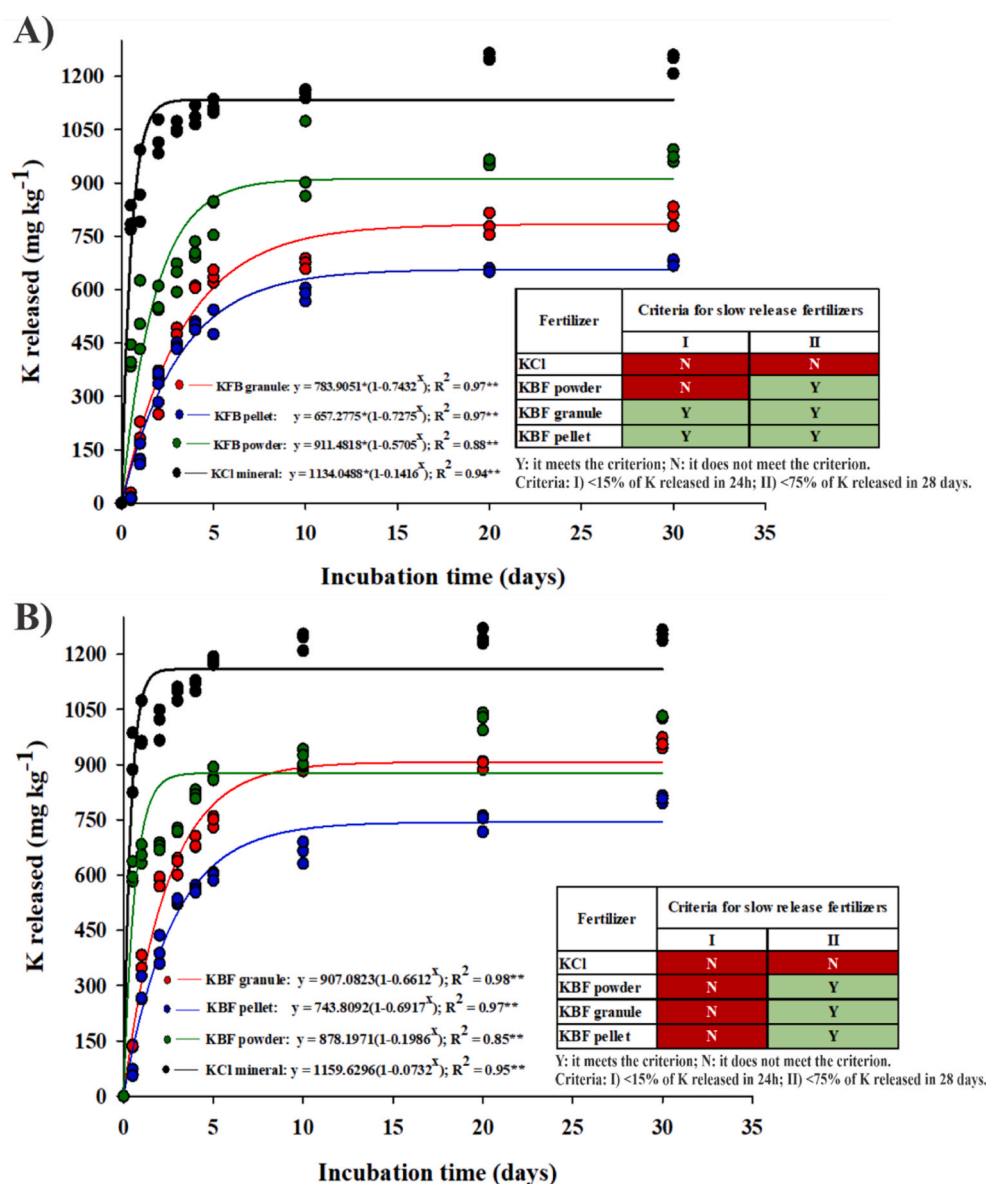


Fig. 3. K release (mg kg⁻¹) from KBF and mineral KCl over time (days) in silica sand with 10% moisture (A) and 20% moisture (B). **P < 0.0001. Tables show the criteria for fertilizer to be considered slow-release according to the European Committee for Standardization (CEN).

field capacity.

3.2. Slow-release mechanism of K from KBFs

The models used to describe the K release kinetics (Fig. 3) demonstrated that KBFs have a slower K release when compared to KCl mineral fertilizer. According to the European Committee for Standardization (CEN) regulations for slow or controlled release fertilizers, the product must not release more than 15% in 24 h, and no more than 75% in 28 days (Trenkel, 2010). In the present study, when submitted to 10% moisture the KBF both in the form of granules and pellets can be classified as a slow-release fertilizer. On the other hand, KBF in powder form, despite not releasing more than 75% in 28 days, released more than 15% K in 24 h, therefore it did not fully meet the requirements to be considered a slow-release fertilizer according to CEN classification (Trenkel, 2010).

When KBFs were subjected to 20% moisture, none of the fertilizers meet the criterion I and released more than 15% of the K in 24 h. However, in this higher moisture level, only biochar fertilizers (granule,

pellet and powder) met the second criterion of not releasing more than 75% in 28 days. Considering that biochar has different physicochemical characteristics from other raw materials used so far for the development of a controlled or slow-release fertilizer, and studies with fertilizer biochar are recent, there is a need to create new regulations for these new fertilizers that utilize biochar as a raw material.

Potassium is one of the nutrients most subject to leaching losses, especially in sandy soils (Rosolem and Steiner, 2017). As a consequence, in addition to reducing the efficiency of using this nutrient (Cheng et al., 2017), K losses can promote pollution of waterways (Borges et al., 2017). The use of a KBF can thus contribute to the slow release of K, reducing the social and environmental problems caused by the high solubility of K mineral fertilizers.

The release of K from chemical fertilizers is normally proportional to the applied dose (Adebayo et al., 2017). However, organic fertilizers (Soremi et al., 2018), organominerals (El-Mageed and Semida, 2015) and pure biochar (Wang et al., 2018) may not release K proportionally to the fertilizer dose due to complexity of K interactions with the organic and inorganic compounds of these fertilizers. The effect of KBF dose on K

release needs to be better understood.

The results of this work prove that, depending on the soil moisture level, the efficiency of enriching sewage sludge biochar with KCl is increased since this combination resulted in a K slow-release fertilizer. When the moisture is at field capacity, KBFs in the pellet and granule forms were classified as slow-release fertilizers. Recent studies with biochars from other biomasses enriched with mineral nutrient sources have also proven the effectiveness of transforming biochar into a slow-release fertilizer for which it was enriched, mainly N, P and K (Zhao et al., 2016; Gwenzi et al., 2017; Shin et al., 2019; Lustosa Filho et al., 2020; Pogorzelski et al., 2020; Carneiro et al., 2021; Luo et al., 2021). This slower release of the nutrient from a biochar fertilizer resulted in an increase in agronomic efficiency of up to 15% in clayey soils when compared to mineral fertilizer (Borges et al., 2020); and in sandy soils the agronomic efficiency was the same as that of a highly soluble mineral fertilizer (Carneiro et al., 2021). Similarly, a P-enriched biochar fertilizer was more effective in providing P to plants in clayey soil when compared to conventional high-solubility phosphate fertilizer (Pogorzelski et al., 2020).

The slower release of nutrients from biochar fertilizers, when compared to highly soluble mineral fertilizers, has been related to several factors such as: i) formation of new low solubility precipitates on the surface of the biochar (Luo et al., 2021), i.e., elements present on the surface of the biochar can bind with highly soluble nutrients from the mineral source and form new compounds with lower solubility (Carneiro et al., 2021); ii) strong electrostatic attraction between the negatively charged biochar surface and positively charged elements (Gwenzi et al., 2017; Luo et al., 2021); iii) physical protection of the soluble fraction by biochar pores (Pogorzelski et al., 2020); iv) presence of micropores, complex pore geometry and the hydrophobic nature of the biochar (Chia et al., 2015) which limits water access inside the biochar fertilizer, hindering the diffusion and dissolution of the more soluble fertilizer (Limwikran et al., 2018); and v) strong biochar adsorption capacity (Li et al., 2018). Based on these factors and considering the K release kinetics obtained in the present study (Fig. 3), a representative schematic was elaborated (Fig. 4) that seeks to clarify the mechanisms that can explain the release of K from the enriched KBFs.

Several mechanisms act simultaneously which cause the slower K release from KBFs when compared to KCl fertilizer. The kinetics (Fig. 3) obtained in the present study indicated that K release occurs in two distinct phases. The first (Phase 1) was characterized by a faster initial release of K. This behavior may be associated with dissolution of readily available and water-soluble KCl present on the surface of KBF. The

dissolution of K present on the surface can promote the formation of channels on the fertilizer surface through which water will start to diffuse into the fertilizer (Pogorzelski et al., 2020). This water diffusion can be limited and slow, due to a very tortuous path and flow and the presence of varied pores (Gwenzi et al., 2017). Next, a slower release of K begins (Phase 2). This second phase occurs due to the physical protection of the biochar pore network (Dias et al., 2018) and its hydrophobic nature (Chia et al., 2015) which hinders and limits the access of water to the minerals found inside the fertilizer or inside the biochar pores (Gwenzi et al., 2017). Furthermore, a part of the already dissolved K can be re adsorbed on the biochar surface due to electrostatic attraction and its strong adsorption capacity (Luo et al., 2021).

This behavior with two distinct phases has also been reported in other works with biochar fertilizers (Kim et al., 2014; Gwenzi et al., 2017; Shin et al., 2019; Pogorzelski et al., 2020). Biochar fertilizers are often little soluble in water and short-term availability is low (Lustosa Filho et al., 2020). However, it can increase over time due to the fertilizer dissolution process that occurs after water diffuses into the biochar fertilizer matrix and releases the nutrient through its pore structure (Luo et al., 2021). Pogorzelski et al. (2020) reported that the use of biochar as a compound in the production of fertilizers, combined or not with synthetic polymers, improves the recalcitrance and porosity characteristics of the fertilizer. Recalcitrance reduces the structural degradation of the soluble fertilizer, providing greater stability over time. Porosity, on the other hand, controls the flow of water in the fertilizer matrix, directly influencing the release of nutrients. According to Kim et al. (2014), the low surface area and reduced pore size of biochar are fundamental properties that influence the interaction between water and the soluble fertilizer associated with biochar. Another important factor to be reported is that biochar has humic substances in its composition (Table 1) that can act as chelating and complexing agents in the formulation of mineral, organic and organomineral fertilizers, forming complex molecules with metal ions. Thus, the presence of humic substances in biochar may have complexed K and contributed to a slower release of this nutrient in KBFs compared to the KCl mineral fertilizer.

3.3. Influence of granulation and pelletizing technology on K release

The results of the present study show that K release dynamics are affected by the enrichment process of KBF. The KBF in a powder form released K faster than other enriched fertilizers (Fig. 3).

Figures S1 and S2 show the KBFs during the incubation period. Note

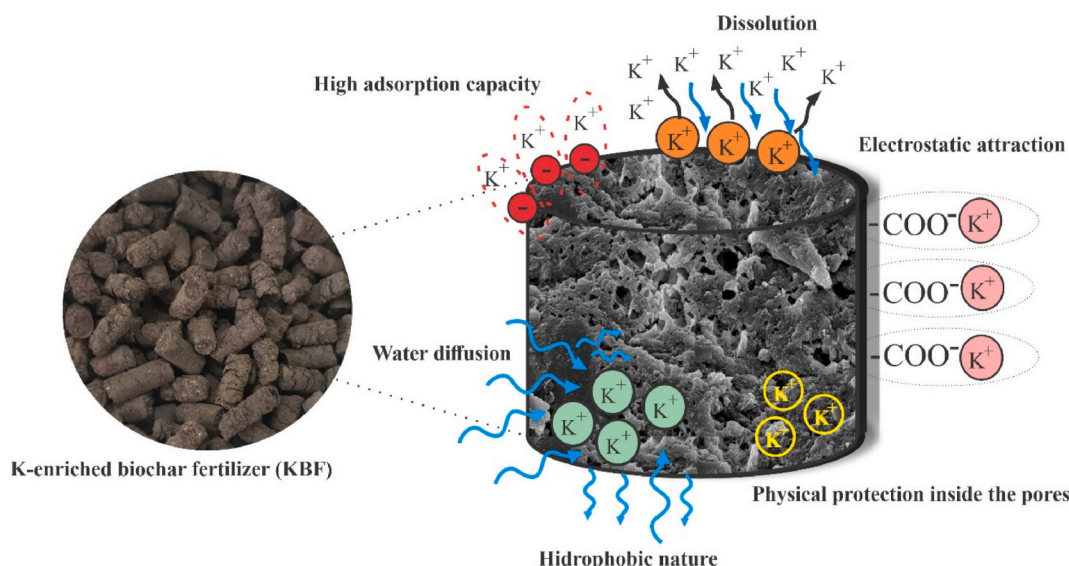


Fig. 4. Schematic representing the interactions between biochar and K that interfere with the release of K in KBFs.

that the pellets and granules in the sachets did not fall apart, i.e., they maintained their initial shapes throughout the incubation time. Even the 20% moisture level (Figure S2) was not sufficient to break up the KBF granules and pellets. This indicates that, at field capacity (moisture level of 10%), both granulation and pelletization promoted protection of the soluble K source found inside the granules and pellets. The presence of starch in the granulation and pelletization process may have contributed to the fact that the KBFs did not break up, promoting protection of the soluble K source. In a work by Kim et al. (2014), increasing the percentage of starch resulted in pellets that promoted slower nutrient release. Another work carried out with biochar fertilizer granules showed that even after 72 days of leaching the granules maintained the same structure due to the presence of starch and this resulted in lower nutrient leaching losses (Gwenzi et al., 2017).

In addition to the presence of starch, the pelletization process may also have contributed to the slower release of K by the pellets when compared to other enriched fertilizers. In general, during the pelleting process, the particles are forced against each other by the applied pressure (Reza et al., 2014), producing smaller pores and reducing the total pore volume (Kim et al., 2014). According to Pogorzelski et al. (2020), porosity controls the water flow in the fertilizer matrix, directly influencing the release of nutrients, i.e., fertilizers with greater porosity will tend to have a higher rate of nutrient release due to the greater ease of water diffusion to their interior.

Fig. 5 shows the N adsorption and desorption isotherm models described by the Brunauer – Enmett – Teller Model for the different KBFs. The isotherms of all materials were Type IV(a) with a Type H3 loop according to the IUPAC classification (Fig. 5). This indicates that in general the pores of KBFs are predominantly mesopores and macropores (Wang et al., 2016; Huang et al., 2017). The KBF in pellet form had the lowest values of SA, PV and MPV, confirming the effect of compaction promoted during pelletization. In this sense, granulation presented higher values of SA, PV and MPV than pellets, but much lower than KBF in powder form.

This reduction in total pore volume reduces the SA and water contact to solubilize the K source present in enriched fertilizers. On the other hand, the higher the PV the easier the diffusion of water into the fertilizer and its contact with the material particles, favoring solubilization of the soluble source and contributing to the release of nutrients. According to Hermawan and Adipati (2018), fertilizers with greater porosity tend to have a higher rate of nutrient release when compared to fertilizers with thicker and smoother layers. Carneiro et al. (2021) evaluated two biochar fertilizers and found that the greatest P release occurred in the biochar fertilizer that presented the highest SA, which

allowed greater fertilizer dissolution. Thus, different biochar fertilizers, even if enriched with the same mineral nutrient source but with different biochars, can result in different nutrient release rates due to different physical characteristics of the enriched fertilizer. Biochar fertilizer pellets with K, P and lignin showed slower release of K and P due to higher stability/durability promoted by lower SA and PV (Kim et al., 2014). Slower biochar N release was also observed in granules with lower PV (Chen et al., 2018). In this sense, granules presented higher values of SA, PV and MPV than pellets, but much lower than the product in powder form, which may have resulted in slower release in the order pellet < granule < powder.

KBF in a pellet form had a higher Dp than in granule form (Fig. 5). In general, a higher Dp indicates a lower percentage of voids and gaps within the material (Hu et al., 2016). This occurs because in the pelletizing process the press reduces gaps and empty spaces between the material particles (Reza et al., 2012), requiring a greater amount of raw material to form a pellet. In the granulation process, the amount of powder material needed to form a granule is smaller and there is also no pressing process, resulting in a more porous structure of the granule when compared to the pellet structure (Vincevica-Gaile et al., 2019). The Dp results (Fig. 5) once again prove the effect of pelletizing on the physical characteristics of the pellet and its effect on the K release dynamics. On the other hand, greater pore volume is observed in KBFs in the form of granules and powder, facilitating the entry of water into the fertilizer which causes greater contact with the material particles and favors a greater release of nutrients when compared to KBF in the pellet form (Fig. 3).

The results of the present study indicate that enriching SSB with K sources is a promising practice for the production of a slow K release BF. The KBF exhibited excellent slow K release performance compared to the KCl mineral fertilizer, which contributes to reducing K loss through leaching and increasing the efficiency of fertilizer use. In addition to resolving the low K supply via application of pure SSB and contributing to the recycling of SS in agriculture, this work presents a sustainable alternative for the production of new SSB-based fertilizers, recycling of SS, rational use of highly soluble K mineral fertilizer and reduces Brazil's dependence on potassium fertilizer imports.

3.4. Potassium leaching from biochar-based fertilizer and KCl

Fig. 6 shows the cumulative amount of K leached over six days in the leaching column experiment. The cumulative K leached followed the order of KCl > powder = granule > pellet > control (P < 0.05). In

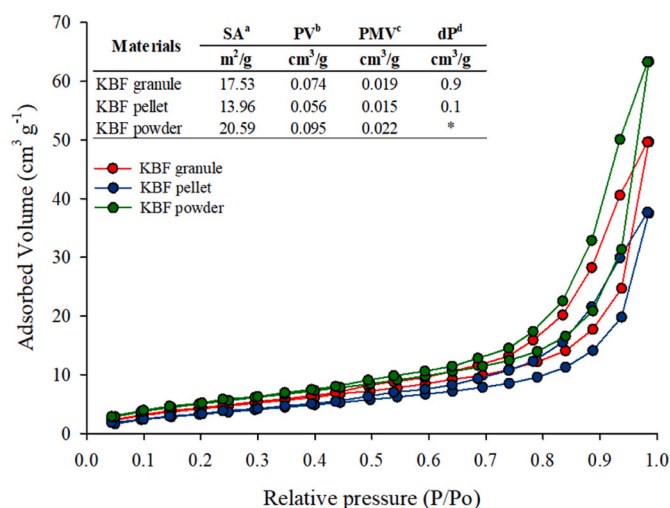


Fig. 5. Physical property values of KBFs. ^a: surface area; ^b: pore volume; ^c: micropore volume; ^d: particle density; *: not determined.

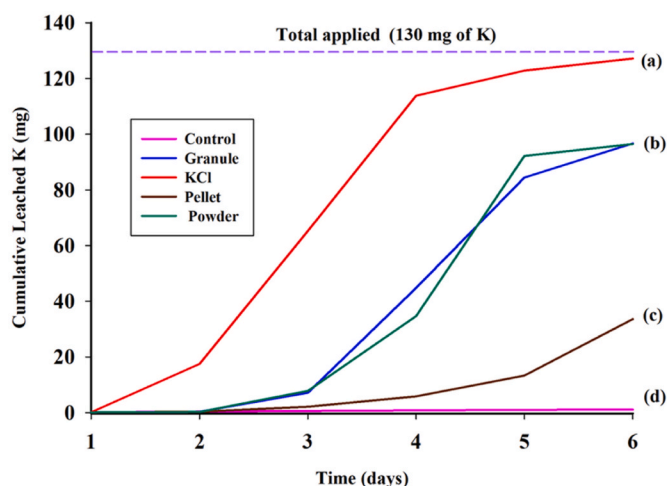


Fig. 6. The amount of K⁺ leached from biochar-based fertilizers and KCl in leaching columns filled with silica sand. Different letters indicate that the fertilizers differed significantly by the Tukey test (P < 0.05).

general, KBFs reduced the total amount of leached K compared to the chemical fertilizer (KCl). From the total amount of K applied (130 mg), 128 mg were leached from the chemical fertilizer after 6 days. Both granule and powder fertilizers showed intermediate values, leaching 96 mg of K. In 6 days, KBF in the pellet form reduced the total leached of K to 33.6 mg, showing the great potential of this type of fertilizer for reducing K leaching. Additionally, these results indicate that biochar-based fertilizers retain K for a longer period than chemical fertilizers. As mentioned previously, the lower rate of leached K in biochar-based fertilizers is due to their ability to release this nutrient slower, mainly when the pellet form was used, possibly due to the higher K retention capacity of biochar (Yuan et al., 2016). In the work by Shin et al. (2019), pelleting was efficient for reducing N and P leaching when compared to the same non-pelleted material with and without mixing with biochar. Mohammadi (2021) stated that one of the benefits of pelleting biochar is to reduce nutrient losses via the leaching process.

Due to the high solubility of conventional fertilizers, about 50–70% is lost to the environment (Trenkel, 2010), being unsuitable for sustainable agricultural ecosystems and requiring that their properties are improved (Timilsena et al., 2014). In addition, the greatest loss of K in agriculture via highly soluble fertilizer application is through the leaching process (Buresh et al., 2010). Therefore, in the present study biochar fertilizers, mainly those in pellet form, presented good properties to be used as sustainable fertilizers with minimum risk of polluting groundwater through leaching processes.

4. Conclusion

The results confirm the hypothesis that biochar-based fertilizers delay K release. In the first four days after application, KBFs reduced K release by up to 77% compared to the KCl mineral fertilizer. K release dynamics were affected by the enrichment process of KBFs. Performance of the K slow-release pellets was attributed to their physical characteristics after going through the pressing process during pelletizing, such as lower surface area, pore volume, micropore volume and particle density compared to KBFs in powder form. At the 10% moisture level, biochar fertilizers in the pellet and granule forms can be classified as slow-release fertilizers. Furthermore, compared to KCl, biochar-based fertilizers reduced the amount of leached K highlighting their function as sustainable fertilizers. The scaling-up process and economic analysis need to be better understood. All results presented in the current study are valid for the silica sand medium. For agronomic and environmental applications, an additional evaluation must be performed in soils. Therefore, future works should evaluate the performance of KBFs on K release and uptake dynamics for plants in different soil types.

Credit author statement

Joisman Fachini: Conceptualization, Methodology, Writing – original draft. Cícero Célio de Figueiredo: Conceptualization, Supervision, Writing – original draft, Writing – review & editing, Visualization. Ailton Teixeira do Vale: Writing – review & editing, Visualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jenvman.2022.115080>.

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