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Research Article

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Kinetics of potassium release from soil amended with clinoptilolite zeolite and maize stalks biochar

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ABSTRACT
A laboratory incubation experiment in a completely randomized design with three replications was carried out for 90 days to test the effect of zeolite and biochar application to calcareous sandy loam soil on potassium forms distribution and its release rate. The treatments included (1) Absolute control (C), (2) 10 g kg^{-1} zeolite (Z1), (3) 20 g kg^{-1} zeolite (Z2), (4) 10 g kg^{-1} biochar (B1), and (5) 20 g kg^{-1} biochar (B2). After incubation period, the concentrations of soluble, exchangeable, and non-exchangeable K and the release rate of K to 0.01 M $CaCl_2$ during 200 min (10 successive extractions for soil samples of 20 min for each using $CaCl_2$ solution) were determined. Results showed that zeolite application increased the soluble and exchangeable K concentrations. However, amending soil with biochar had a positive effect on all K forms. Addition of zeolite or biochar increased the cumulative K release. The parabolic diffusion, power function and Simple Elovich models described the kinetics of K release to $CaCl_2$ solution well from all the soil treatments. Zeolite and maize stalks biochar may have an effective role in improvement of K availability and release in the calcareous sandy loam soil as well as may aid in increasing the ability of this soil to supply the different crops with K.
Keywords: Potassium, Zeolite, Biochar, Kinetics.

INTRODUCTION
Potassium (K) is one of the essential elements for plant growth and it is involved in many physiological processes in plant such as photosynthesis, regulation of the energy compounds and enzymes synthesis, carbohydrates translocation, water relations and the response of plant salt stress, drought stress, cold stress in addition to biotic stresses (Oosterhuis et al., 2014). As reported by Sharma et al. (2006), the total K in soil varied from 1.1 to 3.1%. The available potassium (Soluble + exchangeable K) ranged from 250 to 500, 105 to 358 and 100 to 300 mg kg^{-1} for Egyptian clay, sandy and calcareous sandy soils, respectively, (Abd el hadi, 2004). The soluble K in soil solution ranges from 0.1 to 2% of the total soil K and slowly available K for plant (exchangeable + nonexchangeable K) ranges between 1 and 10% of the total K (Havlin et al., 1999). When the K levels in soil solution become low, the exchangeable K releases and replenishes this decrease. The exchangeable K ranges between 3.12 and 141.18 mg /100g in Egyptian soils; it is higher in the fine texture soils compared to it in the coarse textured ones (Abou El-Roos, 1972, Abd-El Hamid, 1983, Hassan, 1985). Calcareous soils are characterized by high pH, CaCO3 dominance, and low availability of P, K and micronutrients for plants so it is important to increase the availability of these nutrients in these soils (Taalab et al., 2019). Potassium dynamics, distribution, release, and fixation in soil may be affected by application of some organic and inorganic soil amendments (Sparks, 1987 & Jalali, 2011 & Najafi-Ghiri, 2014). Potassium release is an important process for K supplying ability of the soil in long-term cropping systems. Some soil amendments such as zeolite able to change the equilibrium between the different forms of K and influence K release from soils (Rezaei and Movahedi Naeini, 2009, Najafi-Ghiri, 2014 and Najafi-Ghiri and Owliaie, 2019).

Zeolite minerals are crystalline hydrated aluminosilicates of alkali or alkaline earth metals, have three-dimensional rigid crystalline network structure, tectosilicates with a system of channels, cavities and pores (Ming and Mumpton, 1989). Zeolites have a high cation exchange capacity (CEC), and the negative charge resulted from through the isomorphus substitution of Si^{+4} with Al^{+3} in structural tetrahedra is balanced by cations (e.g., Na^+, K^+, NH^+_4, Ca^{+2} and Mg^{+2}) (Perrin et al., 1998). Natural zeolite has higher affinity for NH^+_4 and K^+ relative to Na^+ or Ca^{+2} and Mg^{+2} because of the dimension of interior channels (0.40 – 0.72 nm diameter) and the negative charge location and density in the structure (Ming and

Boettinger, 2001). In a pot experiment, Thabit (2018) found that amending sandy soil with zeolite significantly increased the K uptake of maize plants and the available K (NH_4OAc- extractable K) content of soil after harvest. Natural zeolites can be used both as a K^+ carrier, some studies compared the K- leaching from KNO3 and K- saturated clinoptilolite, pointing out the advantages of the zeolite as slow- release fertilizers (Hershey et al., 1998). Biochar is a charcoal or black carbon which is prepared by pyrolysis process of biomass in no oxygen or limited oxygen supply (Jha et al., 2010). Biochar application to soil increases the soil cation exchange capacity (CEC), soil fertility, carbon sequestration, crop productivity and improves the availability of potassium in soil (Lehmann et al., 2003 and Wang et al., 2018). Addition of K-containing amendments such as biochar to soil may increase the K forms (soluble, exchangeable and non-exchangeable forms), and may affect on K release and further K fixation capacity of soil (Najafi-Ghiri et al., 2020).

This study aims to evaluate the effects of zeolite and biochar application on soil K forms and content as well as study the K release and to find the best kinetic model for description of K release from calcareous sandy loam soil amended with zeolite and maize stalks biochar.

MATERIALS AND METHODS

Maize stalks biochar production:

Maize stalks were oven dried at 60-70℃ and then cut into small pieces. The dried stalks were placed in a muffle furnace for pyrolysis under limited oxygen supply conditions at 450℃ for 30 min. Then, the produced biochar was crushed in a stainless steel mill. Chemical properties of the produced biochar are shown in Table (2).

Table 1. Energy dispersive x-ray analysis of zeolite (Thabit, 2018)

Component	%	Component	%
SiO_2	64.52	Mn_2O_3	0.16
Al_2O_3	10.82	Fe_2O_3	8.17
Na_2O	0.55	CuO	1.44
SO_3	0.43	ZnO	1.28
Cl	0.06	BaO	0.06
K_2O	4.46	MgO	0.20
CaO	2.77		

Zeolite:

The naturally occurring zeolite mineral used in this study was obtained from Alex-Zeolite Company, Egypt. The energy dispersive x-ray analysis of zeolite and some properties of zeolite were determined previously in Thabit (2018) and are shown in Tables 1 and 2, respectively. The mineralogical composition of zeolite was estimated using a Philips x-ray diffraction equipment model PW/1710 with monochromator, Cu k -α radiation (λ=1 54Å) t 40 kV 35 mA (Thabit, 2018) and the dominant mineral in this natural zeolite was clinoptilolite (85.70%) according to the x-ray diffraction (XRD) pattern.

Soil, maize stalks biochar and zeolite properties:

A sample of a calcareous sandy loam soil was collected from the experimental station farm, Faculty of Agriculture, Sohag University, Sohag, Egypt. Some physical and chemical properties of this soil are shown in Table (2). The collected soil sample was air-dried, ground, and sieved through a 2-mm sieve. The soil particles-size distribution was determined using the pipette method (Richard, 1954). The organic matter content (OM) of the soil was determined using the Walkley-Black method (Jackson, 1973) while the OM of biochar was determined using the loss-on-ignition method. Soil total $CaCO_3$ was estimated using the calcimeter method (Jackson, 1973). The pH of soil, biochar and zeolite was measured in a 1:2.5 suspension using a pH meter with a glass electrode (pH 211, Microprocessor pH meter, HANNA instruments) (Jackson, 1973). The cation exchange capacity (CEC) of the soil, biochar and zeolite were determined using 1M NH4 OAc at pH 7.0 as a saturation solution and 1M NaOAc at pH 8.2 as a replacing solution (Chapman and Pratt, 1961). The electrical conductivity (EC) of soil was measured in the soil paste extract while the EC of biochar and zeolite was measured in 1:5 extract using a conductivity meter (Orion model 150) according to Hesse (1998). The soluble K of soil, biochar and zeolite was extracted using distilled water while the available K of soil, biochar and zeolite was extracted using 1M NH4OAc (pH = 7) as described by Carson (1980). Non-exchangeable K of soil was extracted by 1M HNO_3. Potassium was measured on all extracts using flame photometer (CL378 - ELICO).

Table 2. Some physical and chemical properties of the soil under study, zeolite and biochar produced from maize stalks.

Property	Soil	Zeolite	Biochar
Particle size distribution Sand (%) Silt (%) Clay (%) Texture class	56.12 26.22 17.66 Sandy loam	–	–
O.M (%)	0.32	–	70.83
Total $CaCO_3$ (%)	13.30	–	–
pH (1:2.5 suspension)	7.82	7.53	8.15
CEC (cmol (+) kg^{-1})	21.33	149	54.70
EC (dS m^{-1})	3.41 (paste extract)	0.376 (1:5 extract)	5.23 (1:5 extract)
Soluble K (mg Kg^{-1})	45.85	250	5400
NH_4OAc-extractable K (mg Kg^{-1})	172.55	13400	9780
Non-exchangeable K (mg Kg^{-1})	315.00	–	–

Incubation experiment:

An incubation experiment was done in a completely randomized design with three replicates to study the effect of amending of calcareous sandy loam soil with natural clinoptilolite zeolite and maize stalks biochar on potassium forms content and potassium release kinetics from the studied soil. Plastic pots were used and each filled with 200 g of air-dried soil. The zeolite and biochar were added to soil at levels of 1% and 2% (wt/wt) and mixed thoroughly. These respective levels were equivalent to 10 and 20 g of zeolite or biochar per one kg of soil. Soil with no zeolite or biochar was considered as control (C). The moisture level of soil treatments was kept at field capacity using distilled water throughout the incubation period. The treatments were incubated at 25 ± 2°C for 90 days. At the end of incubation period, the soil treatments were air dried and analyzed to determine K forms and K release in 0.01 M $CaCl_2$ solution.

Soil potassium forms:

After incubation time, potassium forms in soil treatments were determined using different chemical extractions as follows: a- Water-soluble soil K was extracted by distilled water in 1:5 proportions. b- Available soil K (Soluble + exchangeable) was extracted using NH_4OAc (1N) at pH=7 according to Carson (1980) and the exchangeable K equals the difference between the extracted K with distilled water and that extracted with 1N NH_4OAc. c- Soluble, exchangeable and non-exchangeable K was extracted by boiling 2 g of soil with 20 ml of 1M HNO_3 solution for 25 min (Pratt, 1965) and the difference between K extracted by 1N NH_4OAc and that extracted using 1M HNO_3 gives the non-exchangeable K. All extracted K forms were measured using flame photometer (CL378 - ELICO).

Potassium release from soil:

Release analysis of potassium was performed by successive extraction of soil samples after incubation with diluted $CaCl_2$ solution according to Najafi-Ghiri (2014). Five grams of each soil sample mixed with 50 ml of 0.01M $CaCl_2$ solution and shaken for 20 min at the room temperature and then centrifuged at 4000 rpm for 10 min. The supernatant solutions obtained after the centrifugation were analyzed for K concentration using flame photometer (CL378 - ELICO) according to Jackson (1973). Then the soil was mixed with a new 50 ml of 0.01M $CaCl_2$ solution, shaken for another 20 min, and centrifuged. This extraction process was repeated 10 times. The release of K from soil samples with time was described using the following kinetic models (Sparks, 1999 and Almaroai et al., 2013):

First-order [$\ln q_t = \ln q_0 - k_1 t$]

Parabolic-diffusion [$qt = a + k_p t^{0.5}$]

Power function [$\ln qt = \ln b + kf (\ln t)$]

Simple Elovich [$qt = 1/\beta \ln (\alpha\beta) + 1/\beta \ln t$]

Where: q_t and q_0 are the amounts of K released after extraction time t (min) and at $t = 0$, respectively, k_1 is the rate constant of first-order model (min^{-1}), kp is the diffusion rate constant $(mg\ kg^{-1})^{-0.5}$, kf is the rate coefficient value ($mg\ kg^{-1}\ min^{-1}$) and a and b are constants. β is the release rate constant $(mg\ kg^{-1})^{-1}$ and α is the initial release rate constant ($mg\ Kg^{-1}\ min^{-1}$), respectively.

The Standard error (SE) of difference between measured and estimated K release was calculated as $SE=[(q-q^*)^2/(n-2)]^{0.5}$ (Zhang et al., 2000), where: q and q* represent the measured and estimated K release from soil at time t, respectively, and n is the number of measurements.

Statistical analysis:

The data statistically analyzed for analysis of variance (ANOVA) using the GLM procedure in SAS software (SAS ver. 9.2, SAS Institute, 2008). The LSD at 5% significance level was calculated according to Peterson (1985) to compare the means of the studied treatments.

RESULTS AND DISCUSSION

Potassium forms: Data in Table (3) show the changes in soluble, exchangeable, and nonexchangeable K after soil amending with different levels of zeolite or maize stalks biochar and incubation period of 90 days at field capacity moisture level. Soluble K in the studied soil treatments ranged from 45.85 to 123.70 mg kg^{-1} with an average of 73.69 mg kg^{-1}. Soluble K concentration increased significantly from 45.85 mg kg^{-1} in control (C) to 52.70 and 67.40 mg kg^{-1} with application of 1% and 2% zeolite levels (Z1 and Z2), respectively.

The exchangeable K content of the incubated soil samples clearly affected by amending soil with natural clinoptiloite zeolite and biochar produced from maize stalks pyrolysis (Table 3). The soil exchangeable K ranged between 126.70 and 405.60 with mean of 242.89 mg kg^{-1}. Zeolite addition had more significant effect on the exchangeable-K increase than biochar. The concentration of exchangeable-K in soil increased significantly from 126.70 mg kg^{-1} in control (C) to 253.20 and 405.60mg kg^{-1} in soils treated with Z1and Z2 levels of zeolite at the end of incubation period. This result can be explained by the high CEC (149 Cmol+ kg^{-1}), high exchangeable K content (13.40 g kg^{-1}) of the clinoptilolite zeolite used in this experiment and it's high affinity for K ions. These results are in agreement with Rezaei and Movahedi Naeini (2009) who indicated that amending soil with zeolite increased the available K content. Also, Filcheva and Tsadilas (2002) concluded that the exchangeable K positively affected by addition of zeolite to soil.

Amending soil with maize stalks biochar increased the soluble K content significantly from 45.85 mg kg^{-1} in control to 78.80 and 123.70 mg kg^{-1} in 1 and 2 % treated soil with biochar (B1 and B2), respectively (Table 3). This large increase in soil soluble K resulted from application of maize stalks biochar may be due to it's high soluble K content (5400 mg kg^{-1}). These results matched with these reported by Oram et al. (2014), Pimenta et al. (2019) and Najafi-Ghiri et al. (2020).

Also, Amine (2016) indicated that the soluble K content of the calcareous sandy soil increased from 100.4 mg kg^{-1} to 232.7 mg kg^{-1} by application of corn cob biochar at level of 60 Mg ha^{-1}.

Application of biochar at levels of 1 and 2% caused an increase in soil exchangeable K content from 126.70 mg kg^{-1} in control treatment to 192.75 and 236.20 mg kg^{-1}, respectively (Table 3). These results are in agreement with that of Najafi-Ghiri et al. (2020). Also, Wang et al. (2018) showed that the application of bamboo residues biochar to Alfisol and Entisol soils improved the K availability, increased the soluble and exchangeable K content and enhanced K uptake within a winter wheat–maize rotation. Amin (2016) found an increment in the available K of the calcareous sandy soil from 421.3 mg kg^{-1} at control treatment to 740.6 mg kg^{-1} at the highest level of corn cob biochar (60 Mg/ha) amended soil. This indicates the efficiency of biochar in improving the K status and availability in soil because the biochar contains free potassium which do not volatilize during the pyrolysis process. In addition to the positive effect of biochar in improving the CEC and K retention of soil (Jien and Wang, 2013 & Adekiya et al., 2020).

Non-exchangeable K concentration in soil treatments ranged between 307.4 and 343.20 mg kg^{-1} (Table 3). Application of Zeolite and maize stalks biochar to the studied soil had no significant effect on the non-exchangeable K content. Amending soil with maize stalks biochar at level 2% only increased the non-exchangeable K significantly from 315.05 mg kg^{-1} in control treatment to 343.20 mg kg^{-1}. This result was Similar to that reported by Najafi-Ghiri et al. (2020) for a Typic Halpocampids clayey calcareous soil amended with zeolite at level 5%.

Table 3. Concentrations of water soluble, exchangeable and nonexchangeable K (mg Kg^{-1}) of the studied soil treatments after incubation time of 90 days.

Treatment	Water soluble K	Exchangeable K	Non-exchangeable K
Control (C)	45.85	126.70	315.05
Z1	52.70	253.20	307.40
Z2	67.40	405.60	323.60
B1	78.80	192.75	329.25
B2	123.70	236.20	343.20
Mean	73.69	242.89	323.70
$LSD_{0.05}$	12.27	27.86	24.43

C = control; Z1 and Z2 = treated soil with 1% and 2% zeolite (wt/wt), respectively; B1 and B2 = treated soil with 1% and 2% maize stalks biochar (wt/wt), respectively.

Release of potassium from soil samples:

Curves of potassium release for all treatments during 200 min to 0.01 M $CaCl_2$ solution are shown in Fig. (1). It is observed that K release rate was rapid during the initial phase from the successive extraction for all treatments but it reached to a nearly constant rate after 120 min.

Table (4) shows the cumulative K release (mg kg^{-1}) to $CaCl_2$ solution during 200 min. Amending soil with zeolite and maize stalks biochar significantly increased

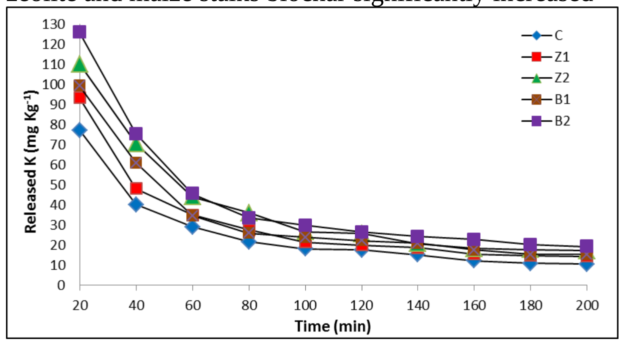

Fig. 1. Potassium release curve for soil amended with zeolite and maize stalks biochar.

the K release from all treatments comparing with un-amended soil. The cumulative of K released significantly increased from 252 mg kg^{-1} in control (C) to 308.90, 387.30 mg kg^{-1} in soil treated with 1 and 2% (wt/wt) zeolite (Z1 and Z2), respectively, and to 335.4 and 422.40 mg kg^{-1} in soil treated with 1 and 2 % (wt/wt) biochar (B1 and B2), respectively (Table 4). As regard to results showed in Tables (3) and (4), the cumulative of k released from soil treatments represents about 51.69, 50.37, 48.62, 55.83 and 60.08% the HNO_3-extractable K (Soluble + exchangeable + non-exchangeable K) for C, Z1, Z2, B1 and B2 treatments, respectively (Table 4).

Table 4. The cumulative of K released (mg Kg^{-1}) from soil treatments by 10 successive extractions with $CaCl_2$ solution during 200 min.

Treatment	Cumulative K release (mg Kg^{-1})	% of HNO_3-K that released by $CaCl_2$
Control (C)	252.00	51.69
Z1	308.90	50.37
Z2	387.30	48.62
B1	335.40	55.83
B2	422.40	60.08
$LSD_{0.05}$	22.89	-

Biochar had more significant effect on K release from soil than zeolite especially in the early period of extraction, which may be due to the quick release nature of biochar K (Angst and Sohi, 2013). In addition to the high affinity of zeolite toward K ions (Panuccio et al., 2008). As mention in Table (2), the clinoptilolte zeolite used in this study has a high ratio of exchangeable to soluble K (13400:250 mg kg^{-1}), which may explain the high tendency of zeolite to adsorb K ions. Similar results were obtained by Rezaei and Movahedi Naeini (2009), Najafi-Ghiri (2014) and Najafi-Ghiri and Owliaie (2019). Also, Najafi-Ghiri et al. (2020) indicated that the cumulative K released to 0.01 M $CaCl_2$ solution increased from 101 mg kg^{-1} for the control soil to 108, 182 and 418 mg kg^{-1} with applying of licorice root pulp, municipal waste and cow manure biochars produced at 300 ºC to the calcareous soil, and increased to 112, 200 and 486 mg kg^{-1} with addition of the biochars produced at 600 ºC, respectively.

Kinetics of K release:

In this study, four kinetic models were used to compare the K release from soil treated with different levels of zeolite and maize stalks biochar. Table (5) shows the calculated determination coefficients (R^2) and standard error of the estimation (SE) for the four kinetic models. The amounts of K released from soil by $CaCl_2$ solution were poorly described by the first- order equation (R^2 ranged from 0.8337 to 0.8657) (Table 5). However, the R^2 and SE values proved that the parabolic diffusion, power function and Simple Elovich were the best models to describe the kinetics of K release. These models were applied successfully also by Jalili (2007) reported a successful application of these models to for description of K release from some calcareous soils in Iran. Rezaei and Movahedi Naeini (2009) indicated that the K release from zeolite may be described well by Elovich and power function models.

Table 5. Determination coefficient (R^2) and standard error of the estimate (SE) of kinetic models for different soil treatments.

Treatment		Parabolic diffusion		Power function		Simple Elovich		First-order	
		R^2	SE	R^2	SE	R^2	SE	R^2	SE
Control	C	0.9954	4.09	0.9949	0.028	0.9897	6.12	0.8610	0.148
Zeolite	Z1	0.9967	4.24	0.9957	0.026	0.9880	8.13	0.8657	0.146
	Z2	0.9912	8.81	0.9871	0.047	0.9946	6.91	0.8337	0.168
Biochar	B1	0.9950	5.69	0.9909	0.038	0.9894	8.24	0.8529	0.153
	B2	0.9945	7.47	0.9907	0.041	0.9908	9.64	0.8505	0.156

The constants for parabolic diffusion (*kp* and *a*), power function (*kf* and *b*) and simple Elovich ($1/\beta$ and $1/\beta$ ln ($\alpha\beta$)) models were calculated (Table 6). The diffusion rate constant (*kp*) in the parabolic diffusion equation and the release rate coefficient (*kf*) in the power function equation are used to estimate the relative release rate (Olama et al., 2010). The *kp* values for K release increased from 17.944 (mg kg^{-1})$^{-0.5}$ in control (C) to 22.00, 27.90, 23.75 and 29.75 (mg kg^{-1})$^{-0.5}$ (Table 6). The constant b value of power function equation increased from 17.68 to 21.10, 24.63, 23.03 and 29.55 with amending soil with 1 and 2 % of zeolite and biochar, respectively (Table 6). Dang et al. (1994), Reyhanitabar and Karimian (2008) and Zahedifar et al. (2010) suggested that an increase in α (initial release rate constant) and/or a decrease in β (metal release constant) in the simple Elovich model would increase the reaction rate. Data in Table (6) showed that the α value increased from 9.225 mg kg^{-1} min^{-1} in control treatment (C) to 10.935, 13.735, 12.143 and 15.513 mg kg^{-1} min^{-1} in Z1, Z2, B1 and B2 treatments, respectively. However, the β values decreased from 0.0129 (mg kg^{-1})$^{-1}$in control (C) to 0.0107 and 0.0083(mg kg^{-1})$^{-1}$ with application of zeolite at levels 1 and 2%, respectively, and to 0.0098 and 0.0078 (mg kg^{-1})$^{-1}$ in the amended soil with 1 and 2% of maize stalks biochar, respectively. This reflects the significant role of zeolite and maize stalks amendments in increasing K release rate from the treated calcareous sandy loam soil.

Table 6. Kinetic models parameters (parabolic diffusion, power function and simple Elovich) for K release from treated soil with zeolite or maize stalks biochar amendments.

Treatment	Parabolic diffusion		Power function		Simple Elovich			
	a	*kp*	*b*	K_f	$1/\beta$ ln $(\alpha\beta)$	$1/\beta$	β	α
C	3.7298	17.944	17.675	0.5073	-164.46	77.337	0.0129	9.225
Z1	2.4126	22.000	21.101	0.5113	-203.14	94.672	0.0107	10.935
Z2	1.8217	27.896	24.626	0.5281	-262.13	120.780	0.0083	13.735
B1	4.6958	23.747	23.030	0.5109	-217.93	102.360	0.0098	12.143
B2	8.0967	29.746	29.550	0.5078	-271.31	128.340	0.0078	15.513

CONCLUSION

Application of zeolite and biochar may be important for management of soil K fertility. Amending the calcareous loamy sand soil with zeolite and maize stalks biochar amendments clearly increased the available K content and improved the K release ability of soil, thus the K supplying power of soil.

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