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Fertilizing properties of potassium feldspar altered hydrothermally

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ABSTRACT

The material obtained through hydrothermal alteration of K-feldspar rock in alkaline conditions is a potential source of soluble potassium (K), but agronomic testing is needed to verify its capacity to supply K to crops. A $(\text{NH}_4)_2\text{SO}_4$ -based bioassay was used to test the capacity of the material to supply K to tomato plants (*Lycopersicon esculentum* Mill.) growing in a mixture of silt loam, peat moss, and sand. Potassium chloride (KCl) and unaltered K-feldspar rock powder also were tested for comparison. The fresh weight and K composition of the plants increased as doses of KCl or hydrothermal material increased, but not with increases in K-feldspar rock. Development of stem lesions, which develop as symptoms of K deficiency in the presence of $(\text{NH}_4)_2\text{SO}_4$, were eliminated by KCl or hydrothermal material but not by feldspar rock. A beneficial effect may occur due to the calcium (Ca) supplied or with adjustment of soil acidity by the hydrothermal material. The hydrothermal material is a K fertilizer at least as effective as KCl since it yields the same or better plant weight.

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KEYWORDS


Potassium chloride; syenite; tomato; potassium fertilization; calcium fertilization; feldspar rock; rock dust

Introduction

Tomato is one of the most common and economically important horticultural crops, ranking among the most consumed vegetables in the world. Compared to some other horticultural crops, tomatoes are potassium (K)-demanding (Boyhan et al. 2017; Kelley and Boyhan 2017; Kelley and Phatak 2017; Westerfield and Florkowska 2014). Tomato also has a high demand for calcium (Ca) to prevent the disease of blossom end rot (BER) that develops with Ca deficiency (Adams and Ho 1993; Bar-Tal and Pressman 1996; Di Candilo and Silvestri 1993; Fontes, Sampaio, and Mantovani 2000; Ho et al. 1993; Maynard, Barham, and McCombs 1957). In addition to its high demand for K, tomato was used as the test plant in the assay for the efficacy of the K-containing fertilizers in this investigation because of the unique property of tomato to develop stem lesions imparted by ammonium stress and for K to alleviate the stress (Barker 1976; Barker, Maynard, and Lachman 1967).

The most widely used sources of K and Ca for tomatoes are KCl and lime (CaCO_3), respectively. Potassium chloride is a salt with high salinity index (Rader, White, and Whittaker 1943). In tomatoes, salinity can induce BER by limiting Ca uptake due to osmotic stress and competition between K^+ and Ca^{2+} (Ho et al. 1993). Moreover, KCl is generally not allowed in organic agriculture, and organic farmers search for alternatives to KCl, although regulations vary across countries (Hollyer et al. 2013; Mikkelsen 2007). In the tropical region, where major agricultural innovation is needed to meet food-security goals, the local cost of KCl is prohibitive, and non-negligible leaching losses due to the soluble nature of this salt often are reported (Baligar and Bennett 1986; Ciceri, De Oliveira, and Allanore 2017; Khan, Mulvaney, and Ellsworth 2014; Manning et al. 2017). Cost is important considering the large tomato volumes that are produced in tropical countries such

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as India (18.3 million t), Brazil (4.2 million t) and Nigeria (2.2 million t) (FAO 2017). Large-scale use of KCl fertilizers recently has come under scrutiny due to a possibly limited efficacy of this fertilizer with several crops (Khan, Mulvaney, and Ellsworth 2014).

Alternative sources of K for agriculture include K_2SO_4 , KNO_3 , and $K_2SO_4 \cdot MgSO_4$ obtained from either mining or chemical synthesis, and less commonly $KMgCl_3 \cdot 6H_2O$ (carnallite) and $K_2Ca_2Mg(SO_4)_4 \cdot 2H_2O$ (polyhalite), obtained exclusively from mining (Ciceri, De Oliveira, and Allanore 2017; Sacks et al. 2017). Agrominerals such as K-bearing silicates, such as feldspars, also have been investigated (Ciceri, De Oliveira, and Allanore 2017; Ciceri et al. 2017; Liu et al. 2017; Manning et al. 2017; Ramos et al. 2015; Santos et al. 2017; Van Straaten 2006). Although K-feldspar rock dusts contain substantial concentrations of K, they are poorly soluble and may have little value as a fertilizer (Bolland and Baker 2000).

Our prior work studied processing of K-bearing silicates, particularly the hydrothermal alteration of K-feldspar in alkaline conditions that leads to a complex calcium-aluminum-silicate-hydrate (C-A-S-H) mixture called for simplicity *hydrothermal material* (Ciceri, De Oliveira, and Allanore 2017). The physicochemical properties of this material suggested its use as a K fertilizer. Relatively similar materials have been tested for soil remediation and soil-pH regulation in China (Liu et al. 2017). However, no study has reported on the fertilizing and K-supplying ability of the hydrothermal material. Specifically for tomatoes, envisaged advantages are that K and Ca are administered concurrently in a roughly constant ratio from a single source (Sacks et al. 2017), *i.e.* the C-A-S-H mixture and that the alkaline properties of the material may regulate the soil pH to a level conducive for tomato growth (Ciceri, De Oliveira, and Allanore 2017; Di Candilo and Silvestri 1993).

In the current work, tomato ('Celebrity') was grown in greenhouse pots with three sources of K: (i) KCl (ii) K-feldspar rock powder (ultrapotassic syenite), and (iii) the hydrothermal material. A $(NH_4)_2SO_4$ bioassay (Barker 1976; Barker, Maynard, and Lachman 1967) was used to investigate the ability of these K fertilizers to prevent stem lesions due to NH_4^+ fertilization and to support plant growth. Results demonstrate that the hydrothermal material is an effective source of K for tomato and that the Ca available from the hydrothermal material and the neutralization of acidity further enhance the growth of the plants. Overall the material provides Ca, increases the soil pH, and does not contain chloride. Based on these results and a forthcoming techno-economic analysis that will be presented in future work, it will be possible to determine if the hydrothermal material tested here is a truly advantageous K fertilizer for organically grown crops and in regions where KCl is not readily available or affordable for use in crop production.

Methods and materials

Tomato plants (*Lycopersicon esculentum* Mill. 'Celebrity') were seeded in a peat-based medium to create 2.5-cm plugs that were transplanted in plastic flower pots (15-cm diam. x 15-cm ht.) after 43 days with a growth medium of silt loam, peat moss, and sand (7:3:2 parts by volume). Each pot contained 1500 g of the medium that was weighed. One scoop of approximately 20 g of the unfertilized growth medium before plant growth was sieved to remove gravel (ASTM E11 < 90 μm , Hogentogler & Co. Inc, Columbia, MD) and was analyzed with X-Ray Powder Diffraction (XRD) (X'pert, Malvern Panalytical Ltd., Royston, UK) for quantitative phase identification. The phases identified were quartz 590 g kg^{-1} , alkali feldspar 220 g kg^{-1} , and muscovite 130 g kg^{-1} (Supplementary Material S1). Overall the growth medium was poor in K and other nutrients and hence acidic.

Growth on treatments occurred under greenhouse conditions with temperatures controlled by fan ventilation and about 25°C by day and 22°C at night with ambient daylight in September and October 2017. The K fertilizers were added by hand mixing into the medium of each pot at transplanting (Table 1). Additions exceeding those in Table 1 were also made, and results are reported in Supplementary Table S1. The KCl was fertilizer grade; K-feldspar rock powder (ultrapotassic syenite) was the sample EBT13 SS described by (Ciceri et al. 2017), and the hydrothermal material was obtained according to the method reported by (Ciceri, De Oliveira, and Allanore 2017), using the rock EBT13 SS

Table 1. Amounts of KCl, K-feldspar rock, or hydrothermal material and amounts of total K added to medium by different amounts of fertilizer.

Fertilizer [†]	Measurement	Additions of either fertilizer or K, mg/kg			
KCl	Total fertilizer	200	400	800	1600
	Total K	100	200	400	800
	Total soluble K	100	200	400	800
K-feldspar rock	Total fertilizer	913	1827	3653	7307
	Total K	100	200	400	800
	Total soluble K	~ 0	~ 0	~ 0	~ 0
Hydrothermal material	Total fertilizer	1067	2133	4267	8533
	Total K	100	200	400	800
	Total soluble K	8	17	34	67

[†]Concentration of total K in KCl, 500 g kg⁻¹; in K-feldspar rock, 110 g kg⁻¹; and in hydrothermal material, 95 g kg⁻¹. Potassium in KCl is 100% available; K in K-feldspar rock is almost completely unavailable; and K in hydrothermal material is 8.4% available (Supplementary Table S3).

as the raw material (Supplementary Table S2 and Table S3). The treatments were in a randomized complete block design with three replicates.

The K bioassay with (NH₄)₂SO₄ was performed as follows (Barker 1976; Barker, Maynard, and Lachman 1967). After transplanting tomatoes to pots on day 43 from seeding, daily additions of 100 mL of 0.04 mol L⁻¹ (NH₄)₂SO₄ (fertilizer grade) and ~0.009 mol L⁻¹ NH₄H₂PO₄ (fertilizer grade) were started and continued for 29 days for all pots, thereby generating lesions. The NH₄H₂PO₄ was added to ensure adequate phosphorus nutrition. On day 72, the (NH₄)₂SO₄ treatment was stopped since lesion development was intense with the unfertilized treatment (Barker 1976; Barker, Maynard, and Lachman 1967). Plants were harvested on day 82, 83, and 84, one replicates at a time by cutting at soil-level in the pots. After cutting, plants were weighed for fresh weight, and each triplicate was averaged. Mature, healthy leaves were selected; young, unexpanded, top leaves and the two bottom leaves were not saved. Lesions due to (NH₄)₂SO₄ treatment were rated from visual inspection as follows: 0 (no lesions), 1 (slight development; a few scattered lesions near the bottom of stems), 2 (medium development; considerable lesions on the bottom third of stems) and 3 (severe development over entire stems). A photograph is in Supplementary Figure S2. Leaves that were saved from the plants by cutting from stems were dried in a forced-draft oven at 70°C.

After harvest, the growth medium and the dried leaves were analyzed. Potassium and Ca availabilities in the growth medium after plant growth were determined by extracting the medium with either deionized water (Ricca Chemical Company, Arlington, TX) or 0.1 mol L⁻¹ NaCl (Macron Fine Chemicals, Center Valley, PA). For each fertilizer treatment, samples of the growth medium from the three replicates were mixed together. Next, 2 g of the medium was extracted at a 1:2 solid-to-liquid mass ratio, rotating the suspension for 24 h. The vials were then centrifuged and filtered (VWR 0.45 µm, Radnor, PA). For extractions with deionized water, the pH of the filtrate was measured with a pH meter. The K and Ca elemental contents in the filtrates were measured with Inductively Coupled Plasma Mass Spectrometry (ICP-MS) (Spectrometer 7797, Agilent, Santa Clara, CA).

For each fertilizer treatment, plant leaves from the three replicates were pooled, ground, and incinerated at 500°C for 10 h in a box furnace (Mellen Microtherm, Columbia, MD). Ashes were re-ground in an agate mortar and dissolved in ~1.5 mol L⁻¹ HNO₃ (Sigma Aldrich, St. Louis, MO) for 24 h at a 1:10 (w:w) solid: liquid. The solution was filtered with syringe filters 0.45 µm (VWR, Radnor, PA) and analyzed for K and Ca by ICP-MS. For each solution, analyses were run in triplicates and averaged.

Fresh weights values are average from triplicate replications with error bars given as the standard error. Lesion ratings are average from triplicate replications with error bars given as the standard error.

Results and discussion

NH₄⁺ bioassay

The degree of NH_4^+ -generated lesions is reported (Table 2). Lesion development started on the 25th day of the $(\text{NH}_4)_2\text{SO}_4$ treatment with incipient lesions appearing with 0 KCl and all rock powder additions. No other treatments showed stem lesions. Treatments were stopped after 29 days on treatment to avoid applications of NH_4^+ that would be excessive and result in severe lesions appearing on all treatments and to allow further growth of the plants. At harvest, 10 to 12 days later, lesions were severe with 0 KCl and slight with all other KCl additions. Lesions were moderate to severe with all rock feldspar additions but were least intense at 800 mg K kg⁻¹ from the rock feldspar. Lesions with hydrothermal material additions of 100, 200, or 400 mg K kg⁻¹ medium were moderate and were absent at 800 mg K kg⁻¹. These observations confirm that the rock feldspar as such does not provide enough K to prevent NH_4^+ lesions and, hence, has low K-supplying capacity. The KCl was effective in preventing lesions at total K additions ≥ 200 mg K kg⁻¹ medium. Lesions on the plants fertilized with the hydrothermal material transitioned from a grade of 2 at 100, 200, or 400 mg K kg⁻¹ medium to 0 at 800 mg K kg⁻¹ medium. The pH rose from 4.61 to 4.86 with these additions of hydrothermal material (Figure 1).

Growth medium

The pH tests after plant growth show that KCl did not change acidity of the growth medium, in agreement with common chemistry considerations (Figure 1). However, the hydrothermal material, which has a pH in water of ~ 12 (Ciceri et al. 2017), altered the pH of the growth medium, with the highest addition causing an increase of 0.25 pH unit over the unfertilized growth medium (Figure 1), a trend that is confirmed at even higher doses (Supplementary Table S1). The effect is not seen at lower doses, probably due to the buffering capacity of the growth medium.

The two extracting solutions, water, and NaCl 0.1 M were selected to measure two distinct fractions of the elements within the medium. Water extracted the soluble fraction of K and Ca. The NaCl solution extracted the soluble and the readily exchangeable K and Ca. Due to the relatively small amount of fertilizer used (Table 1), a weak extractant such as 0.1 M NaCl was chosen to avoid a nutrient release from the growth medium that would mask slight differences between fertilizer additions. Extraction results show that the release of K and Ca from the growth medium is enhanced with the addition of KCl or hydrothermal material, but such a release of K is increased greatly only

Table 2. Lesion development on tomato stems as a function of kind and amount of fertilizer applied.

Total K added, mg/kg soil	Lesion rating [†]
None	3.0 \pm 0
KCl	
100	2.0 \pm 0
200	1.0 \pm 1
400	1.0 \pm 0
800	1.0 \pm 0
K-feldspar rock	
100	3.0 \pm 0
200	3.0 \pm 0
400	2.0 \pm 0.9
800	1.8 \pm 0.3
Hydrothermal product	
100	2.0 \pm 0.3
200	2.3 \pm 0.6
400	2.0 \pm 0
800	0 \pm 0

[†]0, no lesions; 1, slight lesions; 2, moderate lesions; 3, severe lesions; \pm standard error.

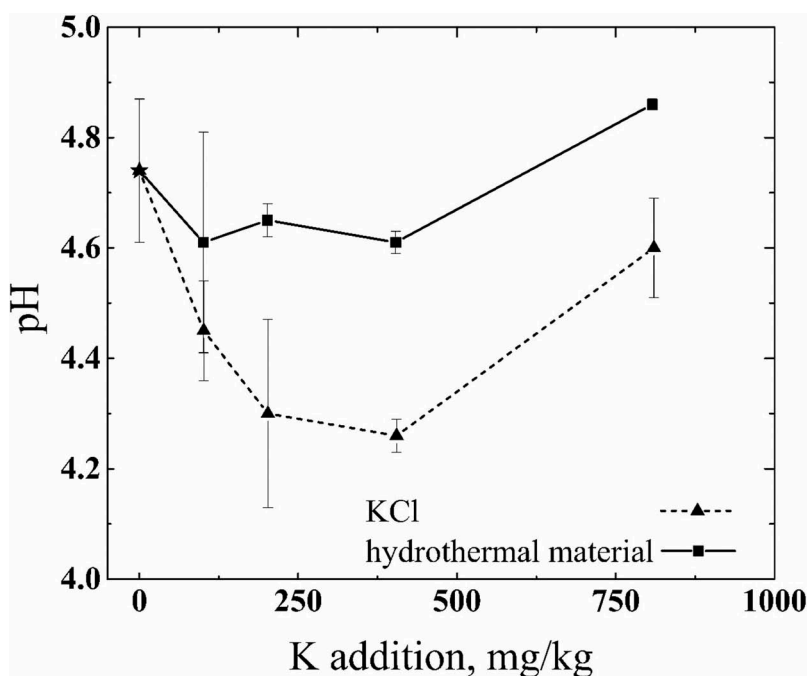


Figure 1. Growth medium pH after plant growth at different applications of fertilizer to the medium. The pH corresponding to 0 addition (star symbol) is that of the medium after plant growth without any fertilizer. Error bars are standard deviation over triplicate pH tests.

by KCl and of Ca is enhanced only with the hydrothermal material (Figure 2). At 800 mg K kg⁻¹ medium, the soluble or readily available K (water extraction) is increased by a factor of 13 over the unfertilized growth medium with the addition of KCl, yet remains virtually unchanged by the addition of the hydrothermal material. At this dose, the exchangeable K determined with 0.1 mol L⁻¹ NaCl extraction shows an increase by a factor of 11 over the unfertilized growth medium for KCl. As with the water extraction, no increase of NaCl-extractable K occurs with the hydrothermal material at this dose. At 800 mg K kg⁻¹ medium, the soil fertilized with KCl shows increases of approximately 1.5 and 3 times in water-soluble and NaCl-extractable Ca, respectively. In contrast, the soil fertilized with the hydrothermal material shows increases of 13 and 2.4 times in water-soluble and NaCl extractable Ca, respectively. The increase in available Ca observed with KCl addition is believed to be due to Ca exchange from the native minerals in the medium promoted by the addition of K⁺ ions introduced with the KCl fertilizer. Unfertilized samples show Ca/K molar ratios of approximately 1 and 6 for water and NaCl extractants, respectively. For samples fertilized with KCl, these ratios are similar but decrease with increasing K, being 1 and 3 at 400 mg K kg⁻¹ medium and 0.1 and 2 at 800 mg K kg⁻¹ medium. Samples fertilized with the hydrothermal material display nearly constant Ca/K molar ratios, across doses, of approximately 15 and 13 for water and NaCl extraction, respectively.

Plant growth

The fresh weight at the end of the growth period as a function of the fertilizer application are reported in Figure 3. The rock powder promotes plant growth above the level of the unfertilized test, confirming results on leek (*Allium ampeloprasum* L.) reported by (Manning et al. 2017).

However, the fresh weight remained significantly lower than with either KCl or the hydrothermal material. The KCl applications showed a standard response curve, where higher doses

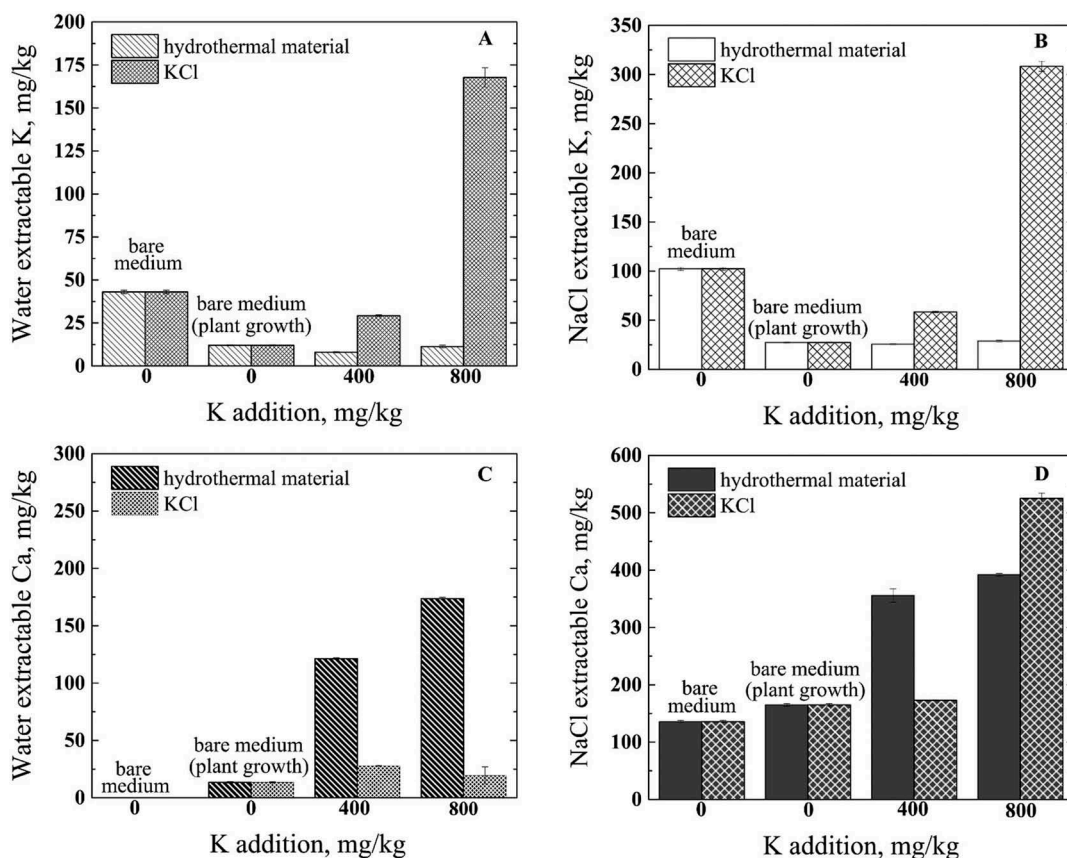


Figure 2. Potassium and Ca availability from the growth medium after plant growth for applications of fertilizer. Extraction with H_2O is given for K and Ca in (a) and (c), respectively; extraction with $0.1 \text{ mol L}^{-1} \text{ NaCl}$ is given for K and Ca in (b) and (d), respectively. Bare medium corresponds to the unfertilized medium as such with or without plants growing in the medium. Error bars are standard deviation over triplicate extraction tests.

(>400 mg K kg^{-1} medium) may result in plateau or even suppression of plant growth, a response that has been associated with Cl^- toxicity or K^+ competition with other cations (Fontes, Sampaio, and Mantovani 2000; Xu et al. 2000). The hydrothermal material resulted in an enhanced growth that exceeded that occurring with KCl at applications from 200 to 800 mg K kg^{-1} medium and with higher applications (Supplementary Table S1).

Leaf composition

The average K content is higher for KCl ($\sim 34 \text{ g kg}^{-1}$) than with the hydrothermal material ($\sim 28 \text{ g kg}^{-1}$) or the rock powder (21 g kg^{-1}) (Figure 4). The K concentration in leaves increased nearly linearly with increasing amounts of KCl added in the range of 0 to 800 mg K kg^{-1} , but no increase occurred with the rock powder or hydrothermal material (Figure 2). With amounts of hydrothermal materials added outside this range, K increased to a top value of 44 g kg^{-1} (Supplementary Table S1). These results are consistent with the suppression of lesion formation on the stems with high additions of KCl or hydrothermal material. Within the range of 0 to 800 mg K kg^{-1} , no differences among the leaf Ca concentrations occurred among the fertilizers, and no trends in Ca accumulation occurred with increasing amounts of fertilizers (Figure 4). With hydrothermal material applied outside the 0 to 800 mg K/kg ,

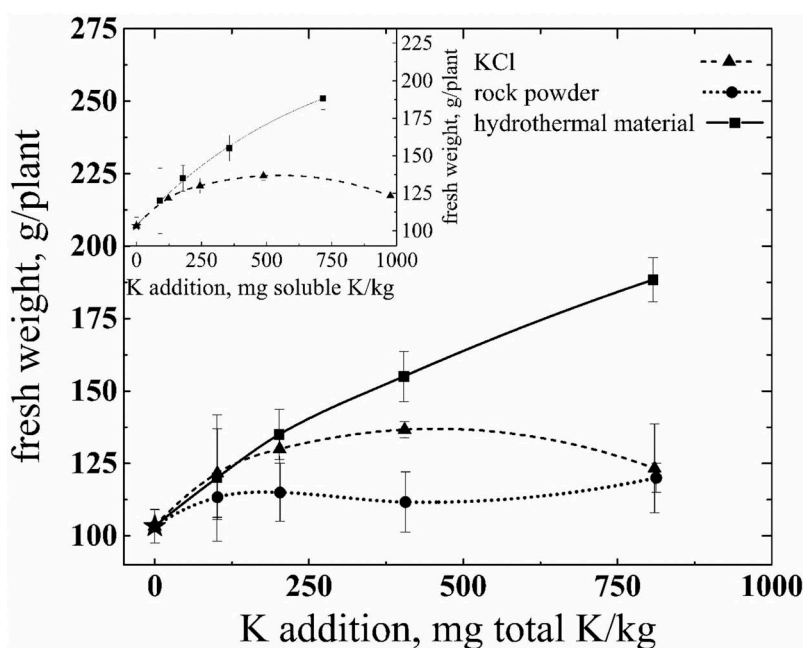


Figure 3. Fresh weights of shoots of tomato as a function of kind of fertilizer and total amount of K added to the medium. The fresh weight corresponding to 0 addition (star symbol) is that of the plant growth without any fertilizer. Inset shows the fresh weight as a function of the amount of soluble K from KCl or hydrothermal material. Error bars are standard deviation over triplicate analyses.

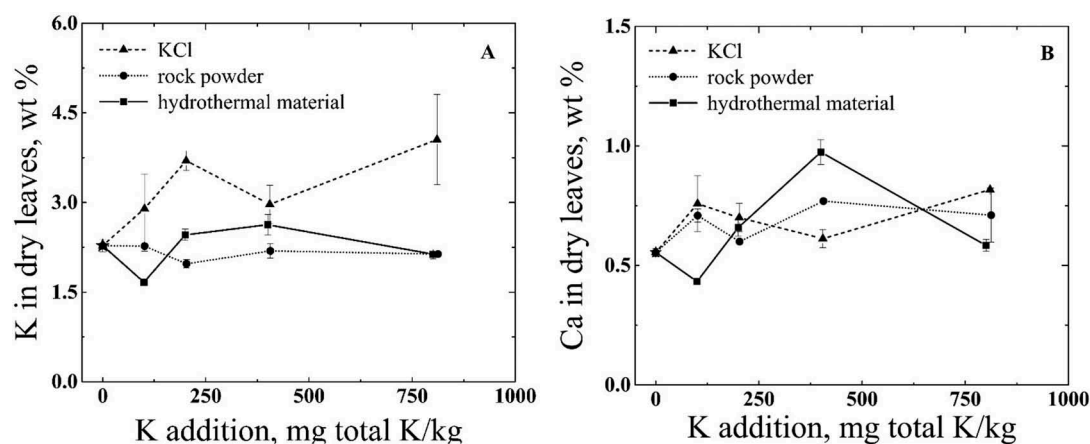


Figure 4. Potassium and Ca concentrations in leaves of tomato as functions of the kind and amount of fertilizer applied to medium. The concentration corresponding to 0 addition is that after plant growth without any fertilizer. Error bars are standard deviation over triplicate tests.

Ca in leaves increased with increasing amounts of material (Supplementary Table S1). Potassium chloride does not supply any Ca, and plants can access only that naturally provided by the growth medium (Figure 4). However, results shown demonstrate that the hydrothermal material can be a concurrent supplier of K and Ca (Figure 4). This action may be particularly important to avoid blossom-end rot of tomato and to supply Ca in general to other crops (Adams and Ho 1993; Bar-Tal and Pressman 1996; Lopez and Satti 1996; Sacks et al. 2017).

In summary, results of the bioassay show that KCl was effective in preventing lesion developments at low through high applications of K, whereas the hydrothermal material was effective at high doses. The rock powder did not prevent lesion development at any dose. At the end of the growth period, the fresh weight of the tomato plants was similar for either KCl or the hydrothermal material and increased with increasing applications of K. Plants fertilized with increasing amounts of the rock powder did not yield a significant increase in fresh weight. The pH of the growth medium was constant around 4.5 for rock powder and KCl at any application, but the hydrothermal material led to a pH 4.9 and thus had a liming effect. In the comparable range of K applications, the average K concentration in the plants was higher with KCl than with the hydrothermal material or the rock powder. The average Ca content in the plants was lower with KCl or the rock powder than with the hydrothermal material, which was the only fertilizer that supplied Ca.

Conclusion

In this work, the fertilizing properties of a K-bearing material obtained from hydrothermal alteration of K-feldspar rock (ultrapotassic syenite) in alkaline conditions were assessed performing an ammonium bioassay. For applications up to 800 mg K kg⁻¹ medium, the fresh weight obtained with the hydrothermal material equals or exceeds that of KCl. Lesion formation from ammonium-induced potassium deficiency was suppressed with all applications of KCl or hydrothermal material but only with the highest applications rock powder. At high applications, a benefit may occur with the hydrothermal material due to the synergistic effect of K and Ca supplied concurrently, as well as pH increase in the growth medium. The K concentration in the plant was higher in plants grown in KCl and increased with increased applications, whereas no increase in leaf K occurred with the rock powder. Large applications of hydrothermal material increase leaf K substantially. The solubility of the hydrothermal material (~79 g K kg⁻¹ water) is lower than for KCl (~340 g K kg⁻¹ water), but a holistic view of its properties permits to conclude that the hydrothermal material enhances tomato plant growth under the conditions of the ammonium bioassay, and therefore has the potential to be an alternative source of agricultural K where KCl is unavailable or expensive.

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