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Research highlights:

- A glasshouse experiment measured the bioavailability of K in zeolite N, made by caustic reaction with kaolin.).
- The elemental compositions of plants supplied with K as K-zeolite N and soluble K fertiliser were similar except for Si being significantly more concentrated in the plants supplied with K-zeolite N.
- For non-leached pots the agronomic effectiveness of K-zeolite N as a potassic fertiliser was superior to. KCl .This may reflect the high Si concentration in plants grown with K-zeolite N combating fungal disease.

Use of a zeolite synthesized from alkali treated kaolin as a K fertiliser:

Glasshouse experiments on leaching and uptake of K by wheat plants in sandy soil

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Abstract

Zeolite N, a zeolite referred to in earlier publications as MesoLite, is made by caustic reaction of kaolin at temperatures between 80 °C and 95 °C. This material has a very high cation exchange capacity (CEC≈500 meq/100g). Soil column leaching experiments have shown that K-zeolite N additions greatly reduce leaching of NH_4^+ fertilisers but the agronomic effectiveness of the retained K^+ and NH_4^+ is unknown.

To measure the bioavailability of K in this zeolite, wheat was grown in a glasshouse with K-zeolite N as the K fertiliser in highly-leached and non-leached pots for four weeks and compared with a soluble K fertiliser (KCl).

The plants grown in non-leached pots and fertilised with K-zeolite N were slightly larger than those grown with KCl. The elemental compositions in the plants were similar except for Si being significantly more concentrated in the plants supplied with K-zeolite N. Thus K-zeolite N may be an effective K-fertiliser.

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Plants grown in highly-leached pots were significantly smaller than those grown in non-leached pots. Plants grown in highly-leached pots were severely K deficient as half of the K from both KCl and K-zeolite N was leached from the pots within three days.

Keywords: Zeolite N; Potassium fertiliser; Wheat; Glasshouse experiment; Nutrient leaching

1. Introduction

Very sandy soils may have inadequate water and nutrient retention for supporting plant growth. To improve these soils for agriculture, horticulture or turf grass, zeolite is sometimes applied as a soil amendment (Ferguson et al., 1986; Nus and Brauen, 1991; Wehtje et al., 2003; Malekian et al., 2011). Zeolite increases the CEC of soil (Suwardi et al., 1994) and reduces leaching of cationic plant nutrients (Huang and Petrovic, 1994; Malekian et al., 2011). Zeolite may also improve the physical properties of soils including water retention and resistance to water repellence (Petrovic, 1990; Xiubin and Zhanbin, 2001). Natural zeolite has been used as a slow release fertiliser providing either natural or added elements (Williams and Nelson, 1997; Carlino et al., 1998; Perrin et al., 1998a; b). However, much of the agronomic research has omitted the characterisation of zeolitic materials in relation to this application (Ming and Allen, 2001).

A synthetic zeolite referred to by the trade name MesoLite was developed to have a high ammonium (NH_4^+) exchange capacity for wastewater treatment (Thornton et al., 2007a, b) with an effective cation exchange capacity (CEC) of approximately 500 meq/100g measured by the NH_4Cl -KCl exchange method (Mackinnon et al., 2003). Recent work by Mackinnon et al. (2010) has shown that this synthetic material is zeolite N of known structure (Christensen and Fjellvag, 1997).

In our previous work (Zwingmann et al., 2009) we measured the retention of NH_4^+ by MesoLite, or zeolite N, loaded with K^+ using a soil column leaching procedure. We also showed that NH_4^+ was gradually released from NH_4^+ -exchanged zeolite N on leaching. This result suggested the potential of this material for use as a slow release nitrogen fertilizer and, by analogy, the potassic form of zeolite N may also release K^+ to plants. This property will be particularly valuable for very sandy soils where leaching of soluble fertiliser is wasteful and an environmental hazard (Carpenter et al., 1998; Ball and Sinclair Knight Merz Pty Limited, 2001). In this paper we present the results of a glasshouse experiment to measure the availability of K in potassic zeolite N to plants grown on a very sandy soil under high and nil leaching conditions. We relate this agronomic information to the characteristics of the as-produced potassic form of zeolite N.

2. Materials and Methods

2.1. Materials

Bassendean sand from Kwinana, 50 km south from Perth (Western Australia) was used for the glasshouse experiments and consists mostly of quartz. This soil is classified in the Australian Soil Classification as a podosol (Isbell, 2002). The pH (H_2O) of the soil is 5.9 and electrical conductivity (EC) 20 $\mu\text{S}/\text{cm}$ (soil: DI water = 1:5). It contains 0.28 % C and 0.05 % N, and the total K content is below the detection limit of XRF analysis (<0.01 %).

Zeolite N, or MesoLite, was manufactured by NanoChem Holdings Pty. Ltd. Queensland, Australia by caustic reaction with kaolin at a temperature between 80 °C and 95 °C (Mackinnon, 1998; Mackinnon et al., 2010). For the present study, potassic zeolite N (K-MesoLite in which the exchangeable cation is K^+) was used as a K fertiliser. A detailed description of potassic zeolite N (K-zeolite N) which has a CEC ~500 meq/100g has been published by Mackinnon et al. (2003), Zwingmann et al. (2009) and Mackinnon et al. (2010).

Experimental CEC values for zeolite N were determined for equilibrium exchange of ammonium ion in a 1M NH₄Cl solution (Mackinnon et al., 2010) calibrated against the Chetomontmorillonite consistent with the determination by Jaynes and Bingham (1986). The unit cell formula for zeolite N is K₁₂Al₁₀Si₁₀O₄₀Cl₂·8H₂O (Christensen and Fjellvag, 1997; Mackinnon et al., 2010). The measured CEC value for zeolite N used in these experiments is 502 meq/100g. The method used to produce this zeolite N is equivalent to reactions 1 and 2 described in Mackinnon et al. (2010).

The pH (H₂O) of K-zeolite N was 11 and EC was 4.2 mS/cm (solid: DI water = 1:5). The pH (H₂O) and EC for soil with K-zeolite N applied at the highest application rate (89 mg K/kg soil) remained close to the values for Bassendean sand, being pH 6.3 and EC 23 µS/cm, respectively. Chemical analyses of Bassendean sand and K-zeolite N are shown in Table 1.

2.2. Procedure for the glasshouse experiment

Soil (6-10 cm) was air-dried and sieved to less than 4 mm. Basal nutrients and various amounts, from 2.8 up to 89 mg/kg soil, of K-fertiliser K-zeolite N and KCl as a reference soluble fertiliser (Table 2) were added to 1.2 kg soil, mixed and placed in 13 cm diameter black plastic pots. All the nutrients were added once only before sowing except for N, which was added first before sowing and the same amount again two weeks after sowing. One group of plants was grown in leached pots and another group grown in non-leached pots. For each condition, the experiments were in triplicate. These pots were watered with de-ionised water (DI water) to the water hold by the soil at the beginning of experiments the day before sowing. Ten pre-germinated wheat (*Triticum aestivum* L.) seeds were sown per pot at 1cm depth and the pots were then placed in a glasshouse. The plants in non-leached pots were watered every day with DI water to maintain the soil at 95 % of the water hold by the soil at the beginning of experiments. The plants in the highly-leached pots were watered daily with DI water and with an amount corresponding to 5 mm excess that leached from the soil. This

watering represents a very high-leaching situation. The leachates from the highly-leached pots were collected, weighed, acidified with 1 mol/L HCl and kept in a cool room at 5 °C prior to K analysis. The pots were randomized every day. At the two leaf stage, plants were thinned to five seedlings per pot by selecting the five most uniform plants. Three weeks post-emergence, plants became affected by fungus and all the pots were treated with K-free fungicide. Twenty-five days post-emergence, the plants were cut at 5 mm from the soil and dried at 70 °C for weighing and elemental analysis.

2.3. Methods for material characterisation and leachate analysis

Elemental analysis was carried out by X-ray Fluorescence Spectrometry (Philips, PW 1730) on fused bead samples for Bassendean sand and K-zeolite N and on pressed pellets for plant material. Finely ground plant samples of approximately 0.3 g with 0.13 mL PVA solution as binder were mixed well and pressed on to a boric acid base at 350 bar using a Sietronics Autopress. Some plant samples with extremely low yield were diluted with cellulose to make 0.3 g and 0.13 mL PVA solution added as binder. C and N contents of the dried plant material were determined with a LECO CHN-1000 analyser. Due to the small amounts of plant material, XRF and C/N analysis was carried out on combined plant material from the triplicate treatments. Concentrations of K^+ in the leachates were determined by atomic absorption spectrometry (Perkin Elmer, AAanalyst 300).

3. Results

3.1. Leaching of K from highly-leached pots

Applied K leached quickly from the highly-leached pots for both K-zeolite N and KCl. Figures 1a, 1b show the amount of K leached from the soil on a daily basis and Figures 1c, 1d show the cumulative percentages of K leached over time. Higher amounts of K leached during the first days (Figures 1a, 1b) and half of the total K applied had leached from the pots between one day (blank and low K pots) and 3 days (K-zeolite N: 89 mg K/kg soil) (Figures

1c, 1d). Leaching was slower for K-zeolite N and also less K had been leached (10-30 % for K-zeolite N, 5-20 % for KCl) after 28 days.

3.2. Yield

The weight of the plants grown in non-leached pots increased with K fertiliser application rate. The plants grown with K-Zeolite N at higher application rates were heavier than those supplied with equivalent amounts of soluble fertiliser (Figure 2a). For comparison, the weights of plants grown in highly-leached pots are plotted in Figure 2b. For the highly-leached pots, the x-axis is “K applied - K leached (mg/kg soil)” at the time of harvest due to severe leaching of K (see Figures 5c, 5d). The plants in highly-leached pots were less than one eighth the weight of plants grown in non-leached pots and relatively larger yields were obtained for K-zeolite N compared to KCl where very little K was retained in the soil.

3.3. K and other elements in plants

The concentration of K in the plants grown in non-leached pots increased with the application rate and the amounts of K taken by plants are similar for both KCl and K-zeolite N. The concentrations of K in the plants grown in highly-leached pots are shown as a function of “K applied - K leached” (Figure 3a). The concentrations of K in the plants grown with K-zeolite N in highly-leached pots were much higher than for the same “K applied - K leached” for non-leached pots. These higher concentrations of K are probably due to the low weight of the plants in the highly-leached pots due to plant growth being reduced due to multiple nutrient deficiencies. Figure 3b (and enlargement in Figure 3c) shows that the internal efficiency of K for plants supplied with K-zeolite N in non-leached pots was higher than for plants supplied with KCl when yields were greater than 1 g/pot.

The concentrations of other plant nutrient elements for plants grown in leached and non-leached pots are shown as a function of yield (weight of the plant) in Figure 4. For all

elements except chlorine and silicon, the concentration decreased with increased plant weight due to increased plant growth reflecting the removal or reduction of K-limited plant growth conditions.

The concentration of Si in the plants grown with KCl (control) decreased with increasing yield while it increased for the plants fertilised with K-zeolite N which evidently acts as a Si-fertiliser.

According to the deficiency criteria of Reuter and Robinson (1997), concentrations of Na, S, Cu, Mn and Zn were sufficient and the concentration of Cl is low enough not to cause toxicity. However, larger plants supported by high K application rates and plants in highly-leached pots may have experienced marginal P, N, Mg and Ca deficiencies.

3.4. K mass balance

Figure 5 shows the mass balance of K for highly-leached and non-leached pots: initial total K (applied K plus blank K) is either absorbed by plants, remains in the soil or is leached from the soil in the case of highly-leached pots. For the non-leached pots (Figure 5a, 5b), the amounts of K taken up by plants from K-zeolite N were higher than for KCl for the higher K application rates. Approximately 40-50 % or more of the applied K was used by plants (Figure 5b). For highly-leached pots, most of the initial K was leached from the pots. Therefore very little K was left for plants, in particular if K was applied as KCl when more than 85 % was leached (Figure 5c, 5d). Leaching of K was slightly less when K was applied as K-zeolite N with more of the retained K being in the soil rather than in the plants at higher application rates.

3. Discussion

These results indicate that much of the K in zeolite N was released into soil solution and was available for plants. For non-leached pots, the plants grown with K-zeolite N were larger than

the plants provided with KCl, and the internal efficiency of K was higher for plants grown with K-zeolite N.

Plants grown in highly-leached pots supplied with KCl grew poorly with no relationship existing between yield and KCl application rate due to severe leaching of most K and basal nutrients at the early stage of plant growth. More than 85 % of K was leached and consequently, only a few % of initially applied K was available for the plants. Plants grown with K-zeolite N in highly-leached pots were slightly larger than for KCl but more than 75 % of K from K-zeolite N was leached from the pots leaving less than 25 % of applied K available for the plants. The source of K found in the leachate from the pots with no K fertiliser (Figure 5c) is unknown. It could have originated from the seeds, water or more likely from the soil, where the total K was below the detection limit of XRF methods, but K may be detected by AAS analysis in the leachates due to higher sensitivity of AAS technique.

These data show that K in K-zeolite N is susceptible to leaching despite being present in a non-soluble form. Presumably, K was released primarily by cation exchange in particular with NH_4^+ , Ca^{2+} , and Mg^{2+} which are major constituents of the basal nutrient solutions (Zwingmann et al. 2009). Thus K-zeolite N cannot be recommended for use on very sandy soils which are very highly-leached and which receive large additions of cation rich fertilisers. However, K-zeolite N is suitable for soils that do not experience severe leaching – the common situation for horticulture.

K-zeolite N increased the Si concentration in wheat for which Si is an essential nutrient. Several studies have shown that silicon enhances resistance of plant to both abiotic and biotic stresses, including fungal diseases (e.g. Ma et al., 2001; Rodrigues et al., 2002; Ma, 2004; Ma and Yamaji, 2006; Bacchus, 2010). In this experiment, plants were affected by a fungal disease after the third week. Plants fertilised with KCl contained less Si and were more affected than those fertilised with K-zeolite N.

This experiment showed the K availability and influence of K-zeolite N on plants during the early growth stage. The longer term influence of K-zeolite N use as a fertiliser on wheat and other plants will be the subject of further investigation.

4. Conclusions

This glasshouse experiment has shown that K from K-zeolite N is available for plants and is a superior source of K under non-leaching conditions. K from K-zeolite N is released by cation exchange particularly with NH_4^+ , Ca^{2+} , and Mg^{2+} present in the basal nutrient solution. For highly-leached soilpots, most K was rapidly leached from the pots with more than 85 % of K from KCl and 75 % of K from K-zeolite N being leached. For non-leached pots, the agronomic effectiveness of K-zeolite N as a potassic fertiliser was superior to that of a soluble KCl fertiliser. The internal efficiency of K use by plants supplied with K-zeolite N was higher than that for plants supplied with KCl. The higher yield of plants supplied with K-zeolite N may, to some extent, reflect the higher Si concentration in plants grown with K-zeolite N because the elevated plant Si helped to combat fungal disease.

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Table 1: Chemical composition of the soil and K-zeolite N

		Bassendean sand	K-zeolite N
Al ₂ O ₃	(%)	0.10	27.12
SiO ₂	(%)	96.37	32.68
TiO ₂	(%)	0.08	0.72
Fe ₂ O ₃	(%)	0.02	0.78
MnO	(%)	b.d.	b.d.
CaO	(%)	b.d.	0.23
K ₂ O	(%)	b.d.	26.05
MgO	(%)	b.d.	0.22
Na ₂ O	(%)	b.d.	0.01
P ₂ O ₅	(%)	b.d.	b.d.
SO ₃	(%)	b.d.	b.d.
V	(ppm)	b.d.	29
Cr	(ppm)	b.d.	b.d.
Ni	(ppm)	14	b.d.
Cu	(ppm)	18	34
Zn	(ppm)	b.d.	b.d.
Rb	(ppm)	4	12
Sr	(ppm)	b.d.	19
Ba	(ppm)	b.d.	88
Zr	(ppm)	35	154
Co	(ppm)	1	2
LOI		3.65	12.25
N	(%)	0.06	0.08
C	(%)	0.28	0.33

b.d.: below detection limit

Table 2: Composition of the basal nutrient solution

Nutrient element	mg/kg soil
K as KCl or K-zeolite N	0, 2.8, 5.6, 11, 23, 45, 89
N as NH ₄ NO ₃ (applied every two weeks)	96.37
Ca as CaCl ₂	40.91
Mg (as MgSO ₄)	3.94
Zn (as ZnSO ₄)	3.25
Mn (as MnSO ₄)	2.04
Cu (as CuSO ₄)	0.51
B (as H ₃ BO ₃)	0.12
Co as CoSO ₄ 7H ₂ O	0.08
Mo as Na ₂ MoO ₄ 2H ₂ O	0.08
S (as MgSO ₄ , ZnSO ₄ , MnSO ₄ , CuSO ₄)	34.20
Na (as Na ₂ MoO ₄ and Na ₂ HPO ₄)	30.44
P (as Na ₂ HPO ₄)	20.50
Cl as CaCl ₂ and	72.44 (as CaCl ₂),
KCl (only for reference pots)	Maximum addition 81mg KCl

Figure captions

Figure 1a:

Amounts of K leached from pots supplied with KCl over time.

Figure 1b:

Amounts of K leached from pots supplied with K-zeolite N over time.

Figure 1c:

Cumulative % of added K leached from the pots supplied with KCl over time.

Figure 1d:

Cumulative % of added K leached from the pots supplied with K-zeolite N over time.

Figure 2a, b:

Average plant weight for non-leached and highly-leached pots plotted vs. K applied and (K applied - K leached), respectively.

Figure 3a:

The percentage of K in plants vs. K applied - K leached for non-leached and highly-leached pots.

Figure 3b:

Internal efficiency: K in plant vs. yield for non-leached and highly-leached pots.

Figure 3c:

Enlargement of the internal efficiency curves for highly-leached pots.

Figure 4 a-k:

The concentrations of elements in plants grown in highly-leached and non-leached pots vs. yield.

Figure 5a:

The distribution of K in plants and soil for non-leached pots.

Figure 5b:

The distribution of K in plants and soil for non-leached pots expressed on a percentage basis.

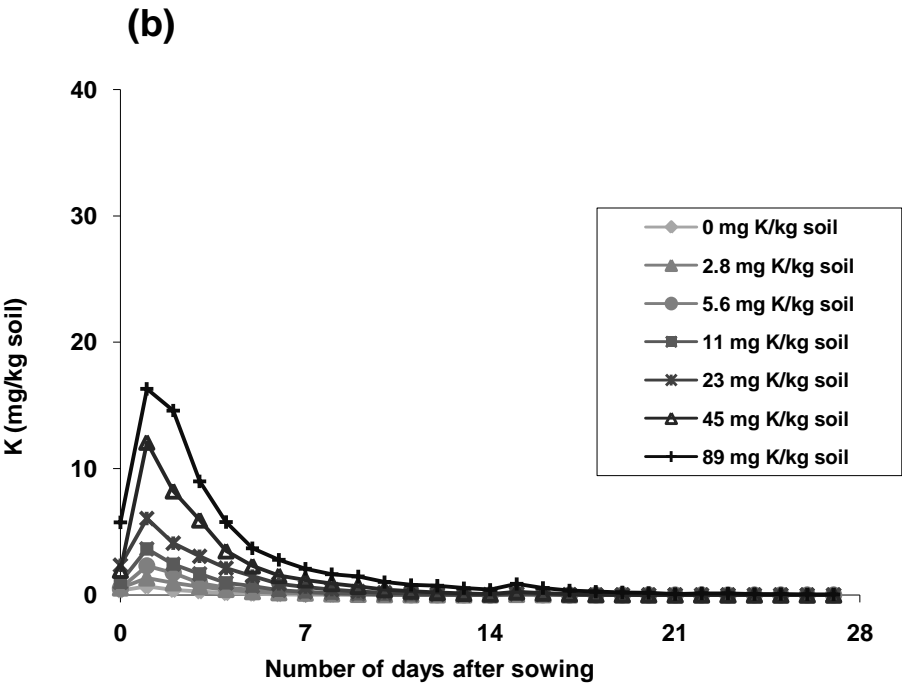
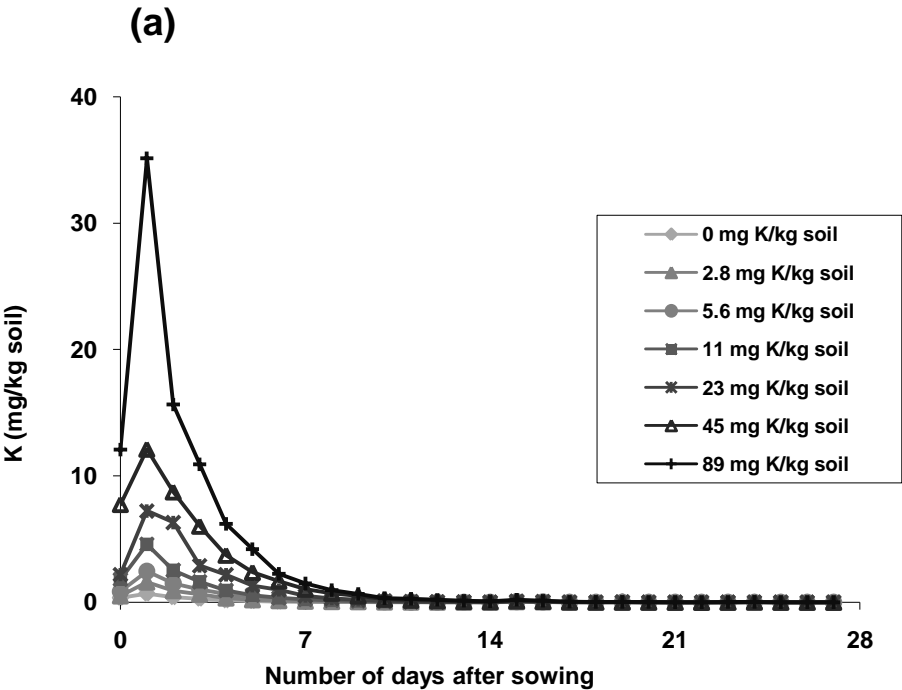
Figure 5c:

The distribution of K leached, K in plants and K remaining in soil for highly-leached pots.

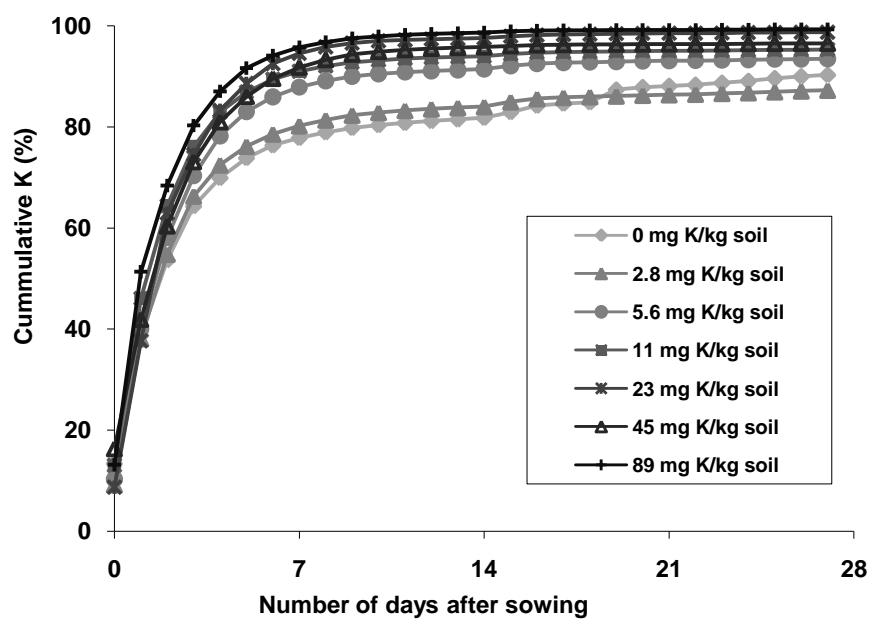
Figure 5d:

The distribution of K leached, K in plants and K remaining in soil for highly-leached pots expressed on a percentage basis.

Figure 1



(c)



(d)

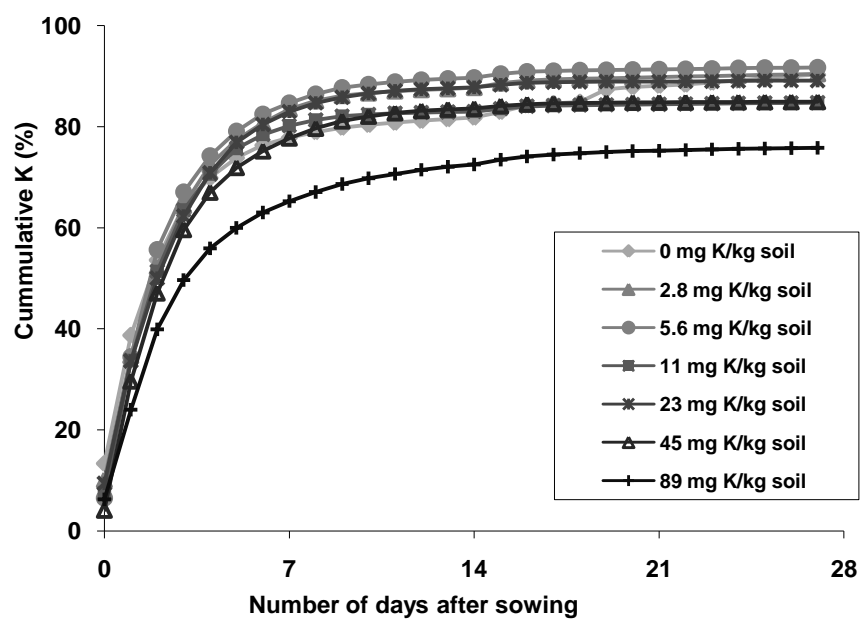


Figure 2 revised

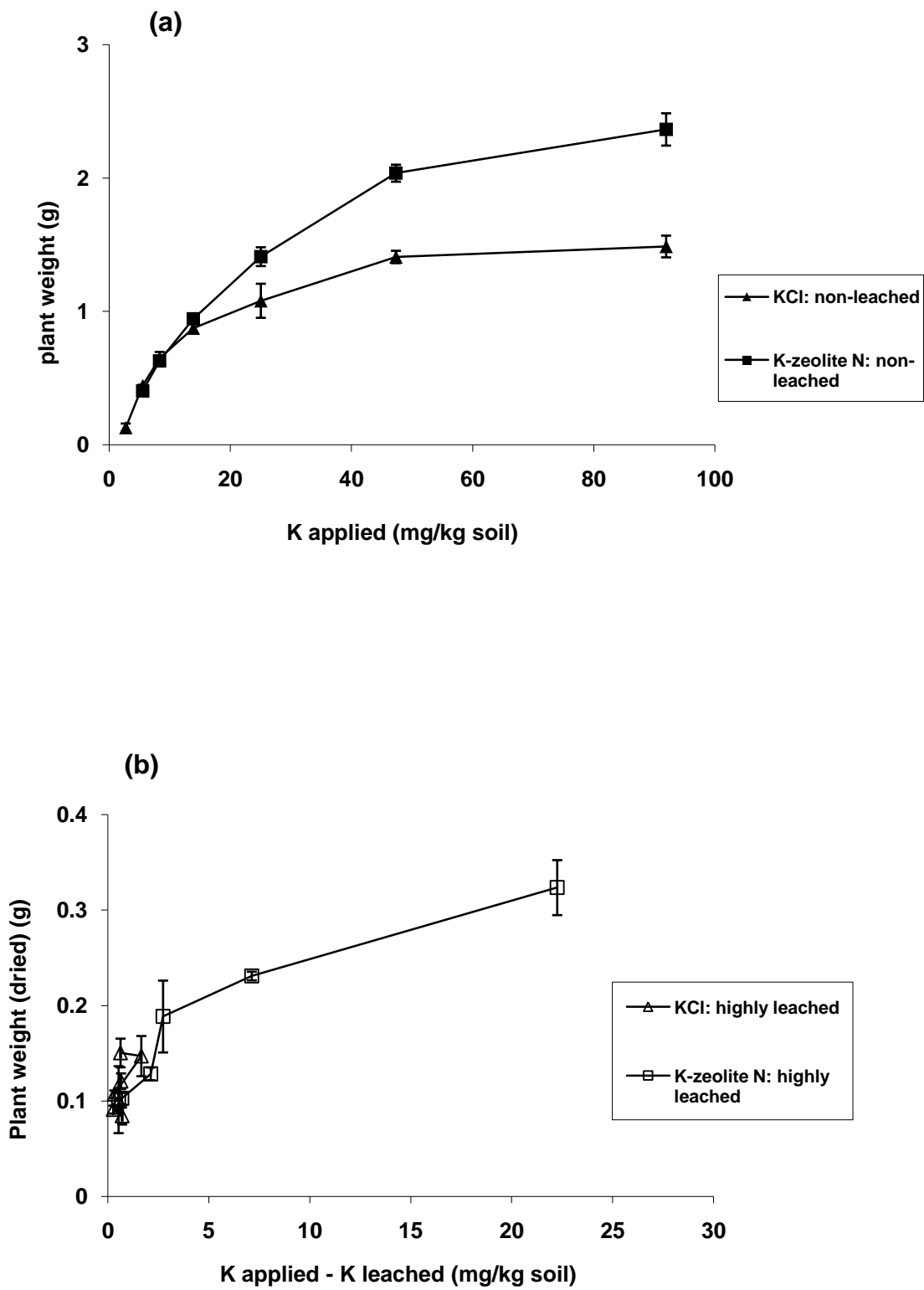
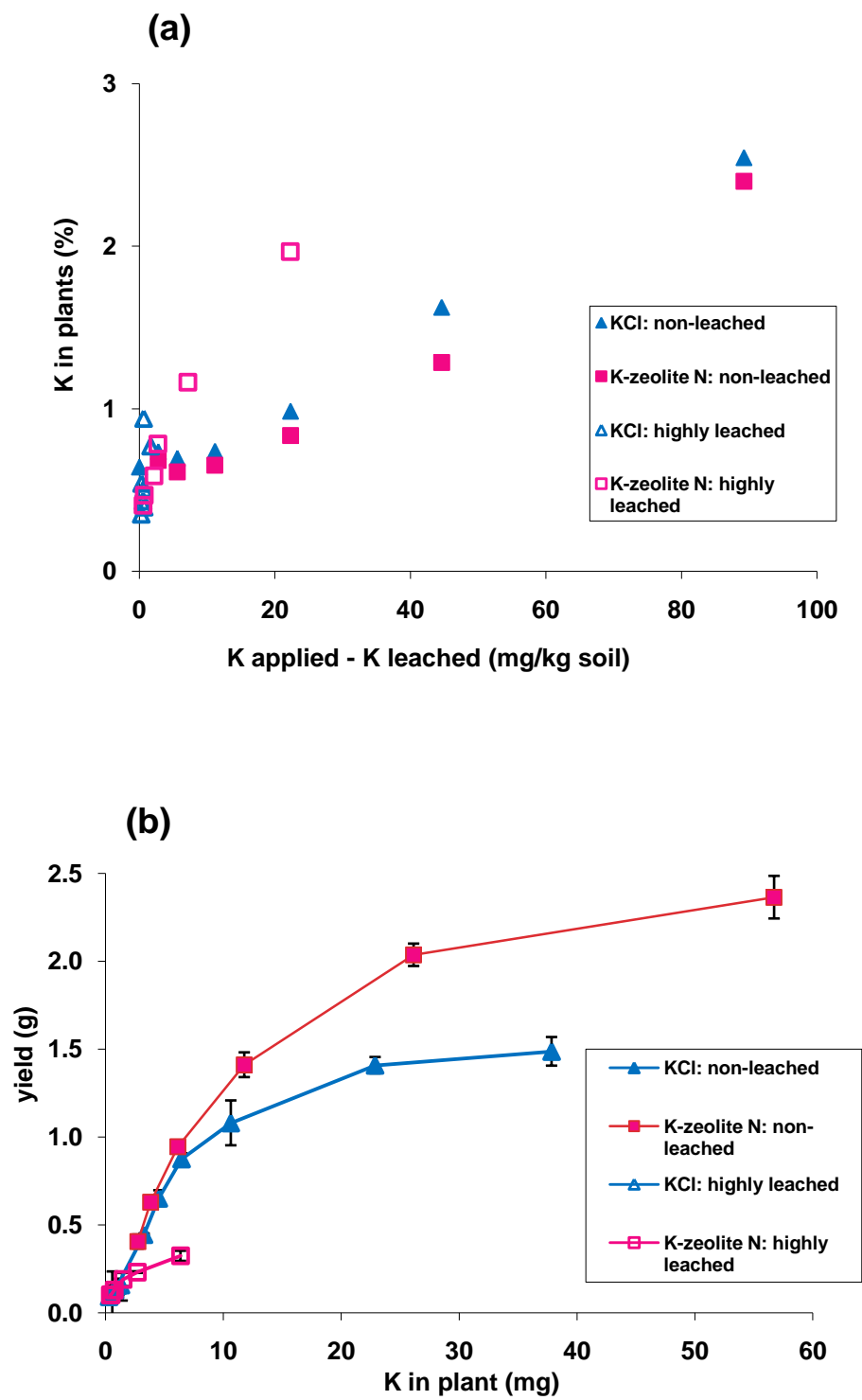


Figure 3 revised



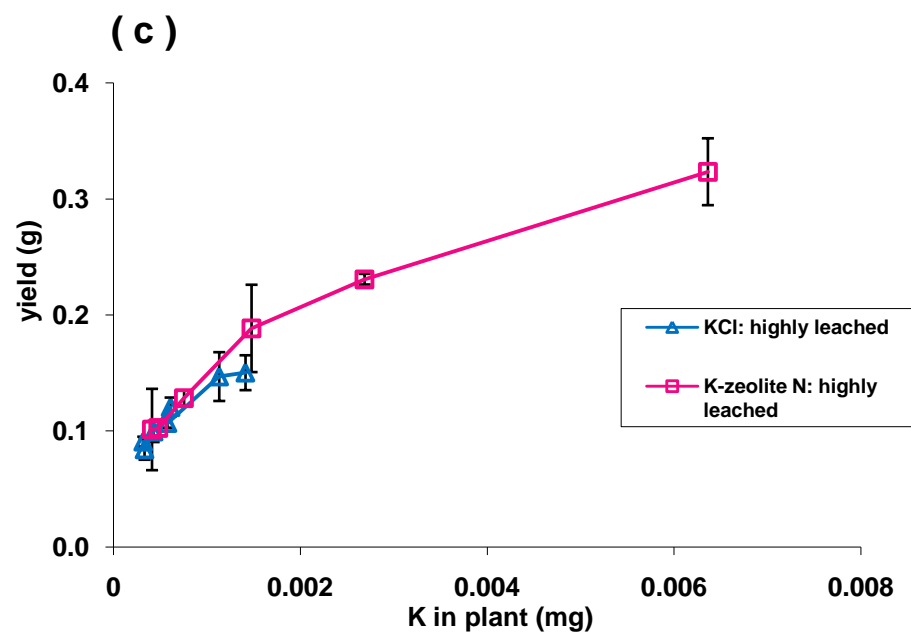


Figure 4

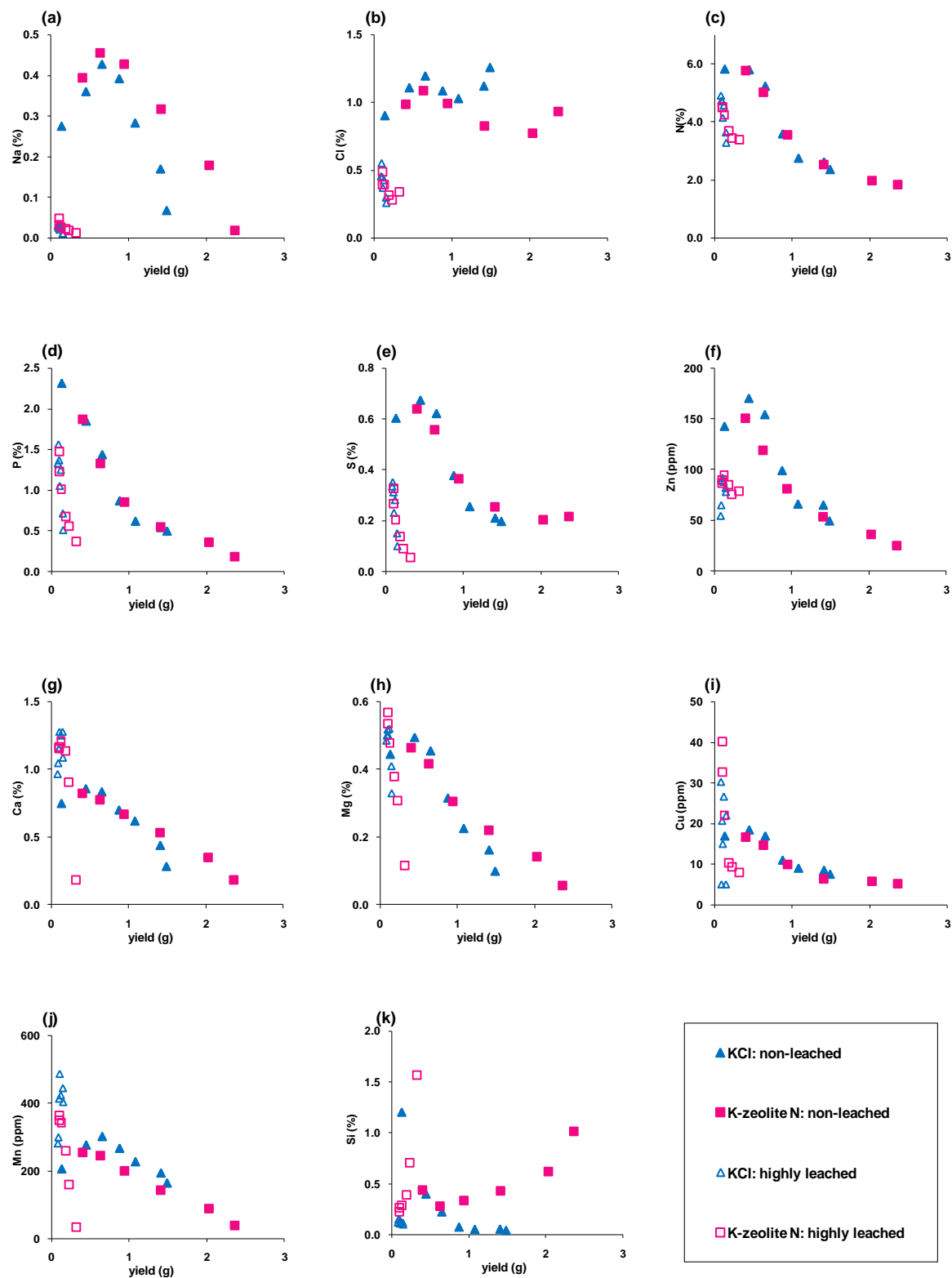


Figure 5 revised

