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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.

1

1	Use of a zeolite synthesized from alkali treated kaolin as a K fertiliser:		
2	Glasshouse experiments on leaching and uptake of K by wheat plants in sandy		
3	soil		
4			
5	Naoko Zwingmann ^{a,c*} , lan D. R. Mackinnon ^b , Robert J. Gilkes ^a		
6			
7	^a School of Earth and Environment, The University of Western Australia, 35 Stirling Highway, Crawley,		
8	Western Australia, Australia 6009		
9			
10	^b Division of Research and Commercialisation, Queensland University of Technology, GPO Box 2434,		
11	Brisbane, Queensland, Australia 4001		
12			
13	Abstract		
14			
15	Zeolite N, a zeolite referred to in earlier publications as MesoLite, is made by caustic reaction		
16	of kaolin at temperatures between 80 °C and 95 °C. This material has a very high cation		
17	exchange capacity (CEC \approx 500 meq/100g). Soil column leaching experiments have shown that		
18	K-zeolite N additions greatly reduce leaching of NH_4^+ fertilisers but the agronomic		
19	effectiveness of the retained K^+ and NH_4^+ is unknown.		
20	To measure the bioavailability of K in this zeolite, wheat was grown in a glasshouse with K-		
21	zeolite N as the K fertiliser in highly-leached and non-leached pots for four weeks and		
22	compared with a soluble K fertiliser (KCl).		
23	The plants grown in non-leached pots and fertilised with K-zeolite N were slightly larger than		
24	those grown with KCl. The elemental compositions in the plants were similar except for Si		
25	being significantly more concentrated in the plants supplied with K-zeolite N. Thus K-zeolite		
26	N may be an effective K-fertiliser.		

^{*} Corresponding author. *E-mail address:* naokozw@hotmail.com Present address: [°] Rio Tinto Centre for Materials and Sensing in Mining, Curtin University of Technology, GPO Box U987, Perth, Western Australia 6845, Australia

leached pots. Plants grown in highly-leached pots were severely K deficient as half of the K

29 from both KCl and K-zeolite N was leached from the pots within three days.

30

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

33

34 **1. Introduction**

Very sandy soils may have inadequate water and nutrient retention for supporting plant 35 growth. To improve these soils for agriculture, horticulture or turf grass, zeolite is sometimes 36 applied as a soil amendment (Ferguson et al., 1986; Nus and Brauen, 1991; Wehtje et al., 37 2003; Malekian et al., 2011). Zeolite increases the CEC of soil (Suwardi et al., 1994) and 38 reduces leaching of cationic plant nutrients (Huang and Petrovic, 1994; Malekian et al., 2011). 39 Zeolite may also improve the physical properties of soils including water retention and 40 resistance to water repellence (Petrovic, 1990; Xiubin and Zhanbin, 2001). Natural zeolite has 41 been used as a slow release fertiliser providing either natural or added elements (Williams and 42 Nelson, 1997; Carlino et al., 1998; Perrin et al., 1998a; b). However, much of the agronomic 43 research has omitted the characterisation of zeolitic materials in relation to this application 44 (Ming and Allen, 2001). 45 A synthetic zeolite referred to by the trade name MesoLite was developed to have a high 46

47 ammonium (NH_4^+) exchange capacity for wastewater treatment (Thornton et al., 2007a, b)

48 with an effective cation exchange capacity (CEC) of approximately 500 meq/100g measured

49 by the NH₄Cl-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

51 and Fjellvag, 1997).

In our previous work (Zwingmann et al., 2009) we measured the retention of NH_4^+ by 52 MesoLite, or zeolite N, loaded with K^+ using a soil column leaching procedure. We also 53 showed that NH_4^+ was gradually released from NH_4^+ -exchanged zeolite N on leaching. This 54 result suggested the potential of this material for use as a slow release nitrogen fertilizer and. 55 by analogy, the potassic form of zeolite N may also release K^+ to plants. 56 This property will be particularly valuable for very sandy soils where leaching of soluble 57 fertiliser is wasteful and an environmental hazard (Carpenter et al., 1998; Ball and Sinclair 58 Knight Merz Pty Limited, 2001). In this paper we present the results of a glasshouse 59 experiment to measure the availability of K in potassic zeolite N to plants grown on a very 60 sandy soil under high and nil leaching conditions. We relate this agronomic information to the 61 characteristics of the as-produced potassic form of zeolite N. 62

63

- 64 **2. Materials and Methods**
- 65

66 **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 (H₂O) of the soil is 5.9 and electrical conductivity (EC) 20 μ S/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 Cheto montmorillonite consistent with the determination by Jaynes and Bingham (1986). The unit cell formula for zeolite N is $K_{12}Al_{10}Si_{10}O_{40}Cl_2 \cdot 8H_2O$ (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 μ S/cm, respectively. Chemical analyses of Bassendean sand and K-zeolite N are shown in Table 1.

89 **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 90 91 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 92 black plastic pots. All the nutrients were added once only before sowing except for N, which 93 was added first before sowing and the same amount again two weeks after sowing. One group 94 of plants was grown in leached pots and another group grown in non-leached pots. For each 95 condition, the experiments were in triplicate. These pots were watered with de-ionised water 96 (DI water) to the water hold by the soil at the beginning of experiments the day before 97 sowing. Ten pre-germinated wheat (Triticum aestivum L.) seeds were sown per pot at 1cm 98 99 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 100 the beginning of experiments The plants in the highly-leached pots were watered daily with 101 102 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. Twentyfive 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

111 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 112 material. Finely ground plant samples of approximately 0.3 g with 0.13 mL PVA solution as 113 114 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 115 116 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 117 material, XRF and C/N analysis was carried out on combined plant material from the 118 triplicate treatments. Concentrations of K⁺ in the leachates were determined by atomic 119 absorption spectrometry (Perkin Elmer, AAanalyst 300). 120

121

122 **3. Results**

123 **3.1. Leaching of K from highly-leached pots**

124 Applied K leached quickly from the highly-leached pots for both K-zeolite N and KCl.

125 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

- 127 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.

131

132 **3.2. Yield**

The weight of the plants grown in non-leached pots increased with K fertiliser application rate. 133 The plants grown with K-Zeolite N at higher application rates were heavier than those 134 supplied with equivalent amounts of soluble fertiliser (Figure 2a). For comparison, the 135 weights of plants grown in highly-leached pots are plotted in Figure 2b. For the highly-136 leached pots, the x-axis is "K applied - K leached (mg/kg soil)" at the time of harvest due to 137 severe leaching of K (see Figures 5c, 5d). The plants in highly-leached pots were less than 138 one eighth the weight of plants grown in non-leached pots and relatively larger yields were 139 140 obtained for K-zeolite N compared to KCl where very little K was retained in the soil.

141

142 **3.3. K and other elements in plants**

The concentration of K in the plants grown in non-leached pots increased with the application 143 rate and the amounts of K taken by plants are similar for both KCl and K-zeolite N. The 144 concentrations of K in the plants grown in highly-leached pots are shown as a function of "K 145 applied - K leached" (Figure 3a). The concentrations of K in the plants grown with K-zeolite 146 N in highly-leached pots were much higher than for the same "K applied - K leached" for 147 non-leached pots. These higher concentrations of K are probably due to the low weight of the 148 plants in the highly-leached pots due to plant growth being reduced due to multiple nutrient 149 deficiencies. Figure 3b (and enlargement in Figure 3c) shows that the internal efficiency of K 150 for plants supplied with K-zeolite N in non-leached pots was higher than for plants supplied 151 with KCl when yields were greater than 1 g/pot. 152

153 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

155 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

157 conditions.

158 The concentration of Si in the plants grown with KCl (control) decreased with increasing

159 yield while it increased for the plants fertilised with K-zeolite N which evidently acts as a Si-

160 fertiliser.

161 According to the deficiency criteria of Reuter and Robinson (1997), concentrations of Na, S,

162 Cu, Mn and Zn were sufficient and the concentration of Cl is low enough not to cause toxicity.

163 However, larger plants supported by high K application rates and plants in highly-leached

164 pots may have experienced marginal P, N, Mg and Ca deficiencies.

165

166 **3.4. K mass balance**

Figure 5 shows the mass balance of K for highly-leached and non-leached pots: initial total K 167 168 (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 169 amounts of K taken up by plants from K-zeolite N were higher than for KCl for the higher K 170 application rates. Approximately 40-50 % or more of the applied K was used by plants 171 (Figure 5b). For highly-leached pots, most of the initial K was leached from the pots. 172 Therefore very little K was left for plants, in particular if K was applied as KCl when more 173 than 85 % was leached (Figure 5c, 5d). Leaching of K was slightly less when K was applied 174 as K-zeolite N with more of the retained K being in the soil rather than in the plants at higher 175 application rates. 176

177

178 **3. Discussion**

These results indicate that much of the K in zeolite N was released into soil solution and wasavailable 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 181 with K-zeolite N. 182

Plants grown in highly-leached pots supplied with KCl grew poorly with no relationship 183 existing between yield and KCl application rate due to severe leaching of most K and basal 184 nutrients at the early stage of plant growth. More than 85 % of K was leached and 185 consequently, only a few % of initially applied K was available for the plants. Plants grown 186 with K-zeolite N in highly-leached pots were slightly larger than for KCl but more than 75 % 187 188 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 189 fertiliser (Figure 5c) is unknown. It could have originated from the seeds, water or more likely 190 from the soil, where the total K was below the detection limit of XRF mehtods, but K may be 191 detected by AAS analysis in the leachates due to higher sensitivity of AAS technique. 192 193 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 194 NH_4^+ , Ca^{2+} , and Mg^{2+} which are major constituents of the basal nutrient solutions 195 196 (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. 197 However, K-zeolite N is suitable for soils that do not experience severe leaching - the 198 common situation for horticulture. 199 200 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 201 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 203

disease after the third week. Plants fertilised with KCl contained less Si and were more 204

affected than those fertilised with K-zeolite N. 205

202

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.

209 **4.** Conclusions

This glasshouse experiment has shown that K from K-zeolite N is available for plants and is a 210 superior source of K under non-leaching conditions. K from K-zeolite N is released by cation 211 exchange particularly with NH_4^+ , Ca^{2+} , and Mg^{2+} present in the basal nutrient solution. For 212 highly-leached soilpots, most K was rapidly leached from the pots with more than 85 % of K 213 214 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 215 KCl fertiliser. The internal efficiency of K use by plants supplied with K-zeolite N was 216 higher than that for plants supplied with KCl. The higher yield of plants supplied with K-217 zeolite N may, to some extent, reflect the higher Si concentration in plants grown with K-218

219 zeolite N because the elevated plant Si helped to combat fungal disease.

220

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228

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- 304

		Bassendean sand	K-zeolite N
AI_2O_3	(%)	0.10	27.12
SiO ₂	(%)	96.37	32.68
TiO ₂	(%)	0.08	0.72
Fe_2O_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_2O_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
Ва	(ppm)	b.d.	88
Zr	(ppm)	35	154
Со	(ppm)	1	2
LOI		3.65	12.25
Ν	(%)	0.06	0.08
С	(%)	0.28	0.33

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

b.d.: below detection limit

Nutrient element	mg/kg soil
K as KCI 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 $Na_2MoO_4 2H_2O$	0.08
S (as MgSO ₄ , ZnSO ₄ , MnSO ₄ , CuSO ₄)	34.20
Na (as Na_2MoO_4 and Na_2HPO_4)	30.44
P (as Na_2HPO_4)	20.50
CI as CaCl ₂ and KCI (only for reference pots)	72.44 (as CaCl ₂), Maximum addition 81mg KCl

Table 2: Composition of the basal nutrient solution

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.































