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Research article

Closing the loop on human urine: Plant availability of zeolite-recovered nutrients in a peat-based substrate

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ABSTRACT

Recycling mineral nutrients from household wastewater is a central step in the development of a circular economy based society. The objective of this study was to evaluate plant availability of mineral elements and plant performance in a peat substrate containing nutrient-enriched zeolite (NEZ) obtained by nutrient recovery from human urine in a source separated wastewater system. Substrate content of potentially available mineral nutrients was determined by $CaCl_2$/DTPA-extraction during a 12 weeks incubation experiment for 20:80 (R20) and 30:70 (R30) volume % of NEZ:sphagnum peat, limed R20 (R20L), and 20:80 vol% of unloaded zeolite:sphagnum peat (Z20). Plant availability of mineral elements from R20, R20L, R30 and Z20 was compared with conventionally fertilised sphagnum peat (P100) for sunflower (*Helianthus annuus* L.) cv. 'Topolino' in a pot experiment. Recovery of nutrients in a potentially available form in the R20 substrate after 12 weeks was 3% (K), 23% (N, P), 34% (Mg) and 90% (S). Liming increased the recovery of mineral N to 39%, suggesting that nitrification was an important driver for the release of NH_4^+. For R20, estimated recovery of urine-derived N in sunflower shoots was 30–36%. Shoot biomass was similar in R20 and in conventionally fertilised peat (P100). However, P100 plants had more leaves and flowers+buds. Initial addition of ammonium phosphate or supplemental fertilisation with a complete nutrient solution increased flower+bud number in R20. For the NEZ-treatments, Cu and B shoot concentrations were in the low or marginal range while Zn and Mn were high or in excess. Shoot growth and nutrient uptake of sunflower were highly restricted in the unloaded zeolite control (Z20). We conclude that 20% NEZ in a peat substrate was effective as a macronutrient source for sunflower, producing similar biomass as in conventionally fertilised peat. However, micronutrient balance and early P supply may need to be adjusted for optimal plant performance.

1. Introduction

Reuse and recycling of key waste streams such as municipal waste and wastewater from society are key priorities in EU's action plan for the Circular Economy (European Commission, 2015). In a circular economy, materials that can be recycled are injected back into the economy as new raw materials, thus increasing the security of supply.

While agricultural and municipal wastes and wastewater are often recycled as fertilisers, more efficient nutrient recovery methods are needed to address the challenges related to nutrient loss and environmental pollution (Buckwell and Nadeu, 2016; Mehta et al., 2015).

One of the main areas for nutrient recycling and reuse is dealing with household waste and wastewater streams. The concept of 'ecological sanitation' (Ecosan) has been established all over the world during the past two decades (Ganrot, 2012; Kirchmann and Pettersson, 1995). Ecological sanitation is based on material flow oriented recycling of nutrients from household organic waste and wastewater, spanning the full range from strictly low-tech to high-tech solutions (Werner et al., 2003). Hundreds – if not thousands – of scientific experiments and pilot projects have resulted in a number of established techniques (Mehta et al., 2015; Simha and Ganesapillai, 2017).

A main challenge related to the treatment of source separated household wastewater is human urine and its use as a liquid

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fertiliser (Karak and Bhattacharyya, 2011; Simha et al., 2017). Many problems are associated with the application of urine for agricultural purposes, such as the need for large storage volumes and long storage time for hygienisation, transports of large amounts of water as urine is a dilute salt solution, the need for special timing and spreading techniques (Ganrot, 2005, 2012), the issue of salt (NaCl) accumulation in soil or plants (Mnkeni et al., 2008), and the socio-psychology of urine handling from taboos to disgust or acceptance (Rosenquist, 2005; Simha et al., 2017).

Human urine can, however, be treated with different methods to solve the abovementioned problems (Ganrot, 2005; Simha and Ganesapillai, 2017). One of the most successful urine treatment processes is the high P, and partial N, recovery as struvite [$Mg(K,NH_4)(PO_4)*6H_2O$] (Antonini et al., 2012; Le Corre et al., 2009; Lind et al., 2000). These studies all conclude that by recovering P as struvite from human urine, the volume is drastically reduced, hygienisation is improved, and the crystals (solids) obtained can directly be used in agriculture or also stored for later use. Handling and spreading can be made with readily available agricultural equipment. In a greenhouse evaluation, urine-derived struvite was an efficient fertiliser covering the magnesium demand and more than 50% of the phosphorus requirement of Italian ryegrass and maize (Antonini et al., 2012). However, struvite recovery is not fully addressing the N recovery from urine, because only a small part of the ammonium content is captured in struvite (Ganrot, 2005; Ronteltap, 2009).

Some natural minerals and materials can be used as nutrient adsorbents. Zeolites are aluminosilicate minerals with particularly high and selective ion exchange properties (Ganrot, 2012; Mumpton, 1999; Sangeetha and Baskar, 2016). Since the mid 90′s, the use of zeolites for N recovery from source separated systems in decentralised household wastewater treatment plants has been intensively studied, especially for urine, showing promising results for the combined usage of zeolites with other wastewater nutrient recovery methods (Ganrot, 2005, 2012). A combination of struvite precipitation and zeolite adsorption was investigated for plant nitrogen and phosphorus availability under controlled climate conditions (Ganrot et al., 2007, 2008).

The use of nutrient loaded zeolites for agricultural purposes, commonly known as 'zeo-agriculture', is a well-researched field since the past 40 years. Some recently published articles (Beler-Baykal et al., 2011; Fujinuma et al., 2015; Sangeetha and Baskar, 2016) describe positive effects of nutrient loaded zeolites on soil nutrient release and nutrient exchange capacity, plant growth or plant water supply regulation. Specifically, ammonium-loaded zeolite, combined with rock phosphate, has been suggested as a slow-release fertiliser for potted plant production (Pickering et al., 2002).

The aim of the present study was to investigate the possibility of using zeolite loaded with nutrients collected from human urine as a plant nutrient source in a horticultural cultivation system. Our objective was to determine plant availability of mineral elements and plant performance when a nutrient-enriched zeolite (NEZ), obtained by nutrient recovery from a source separated wastewater system by zeolite adsorption and phosphate precipitation, was incorporated into a peat-based substrate. We also wanted to assess the influence of NEZ dose and liming on mineral availability.

2. Materials and methods

2.1. Nutrient-enrichment of zeolite

Nutrient-enriched zeolite (NEZ) was produced by Again AB, Gothenburg, using hydrolysed urine collected from urine diverting toilets at the Ecocentrum Environmental Exhibition building in Gothenburg, Sweden. The NEZ (Gainutri™) was obtained by a new method based on zeolite adsorption and P precipitation techniques, using Mg as a main cation (patent no. PCT/EP2014/064361). Zeolite clinoptilolite (Rota Mining Corporation, Istanbul, Turkey) with a grain size of 0–800 μm was used as the main component of the NEZ for ammonium-N recovery. The ion selectivity of the zeolite was $Cs^+ > NH_4^+ > Pb^{2+} > K^+ > Na^+ > Ca^{2+} > Mg^{2+} > Ba^{2+} > Cu^{2+}, Zn^{2+}$ according to the producer. The mineral element composition of zeolite and NEZ is presented in Table 1.

2.2. Substrate incubation

Mineral element availability in four substrates amended with NEZ or unloaded zeolite (Table 2) was studied during 12 weeks incubation a semi-controlled climate. A coarse (5–20 mm), lightly decomposed (H3-H4, Von Post scale) milled peat was used as the main substrate component. The NEZ was mixed with the peat at 20% (R20) or 30% (R30) (v:v). For the substrate containing 20% NEZ, a treatment limed with 1000 mg L^{-1} of $CaCO_3$ (R20L) was also included. A control treatment (Z20) contained 20% unloaded zeolite only. Deionized water was added to 15% of substrate field capacity.

For each of the substrates, four 10 L batches were prepared and each batch was further divided into eight samples. Each sample was placed in a plastic bag, and the four replicate samples for each substrate composition and week were distributed in a plastic box covered with a black (inside) and white (outside) plastic sheet. The substrate samples were incubated from the 29th of June in a daylight chamber with a day/night temperature of 20/15 °C (16/8 h) and a relative humidity of 70%. After 0, 1, 3, 4, 6, 8, 10 and 12 weeks, one box was removed from the chamber and stored at 4 °C before analysis.

Substrate contents of potentially plant available (CAT-extractable) mineral elements were measured after extraction in 0.01M $CaCl_2$+0.002M DTPA according to the European standard method for growing substrates (SS-EN 13651). CAT-extractable elements, substrate compacted laboratory bulk density (SS-EN 13040), and substrate pH after 1:5 (v:v) substrate:water (SS-EN 13037), were determined by Eurofins Agro Testing Sweden, Kristianstad.

Table 1
Average mineral element contents of zeolite and NEZ, calculated from duplicate samples taken during a 6 month long NEZ production period. Analyses were performed by Eurofins Environment Testing, Sweden.

Analysis	Zeolite	NEZ	Units	Method
Dry matter (DM)	86	93.3	%	105 ± 5 °C[a]
pH	8.9	8.6	–	H_2O[b]
N	<500	2650	mg kg^{-1}	Kjeldahl[c]
NH_4-N	<100	2300	"	40% NaOH[d]
Ca	27 000	24 500	mg kg^{-1} DM	aqua regia[e]
K	16 500	16 000	"	"
Mg	6250	7600	"	"
S	135	1300	"	"
P	155	340	"	7M HNO_3[f]
Cu	3.6	4.3	"	"
Ni	7.7	9.5	"	"
Zn	33	39	"	"
Cd	<0.095	0.08	"	"
Pb	46	52	"	"
Hg	<0.047	<0.048	"	"
Cr	2.8	4.0	"	"

[a] SS-EN 12880:2000SS.
[b] SS-EN 15933:2012.
[c] SS-EN 13342.
[d] Standard methods 1998, 4500 mod.
[e] ISO 11466/EN13346 mod.
[f] SS 028150-2.