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## Moisture and Nutrient Storage Capacity of Calcined Expanded Shale

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### 1. Introduction

Expanded shale (EXSH) is an important and increasingly popular soil conditioner with several horticultural applications, including its use as a soil amendment for clay textured soils (Sloan et al., 2002), as an ingredient in plant growing media (Sloan et al., 2010) or green roof substrates (Ampim et al., 2010). It is a lightweight material produced by firing mined lumps of shale at high temperatures in a rotary kiln in a process similar to that of clay ceramics. The resulting product can be screened to create various size fractions depending on the intended use. For example, Texas Industries (TXI) of North Texas, USA produces five size fractions of expanded shale that includes the following ranges, from smallest to largest, 0.07 to 0.60 mm, 0.60 to 2.0 mm, 2.0 to 4.8 mm, 4.8 to 6.4 mm, 6.4 to 9.5 mm, 9.5 to 12.7mm, 12.7 to 15.9 mm Expanded shale aggregates are suitable as components of planting media and soil amendments because, unlike most minerals, they are porous, stable, and resistant to decomposition (Ferguson, 2005). Expanded shale is believed to beneficially modify growth media properties by enhancing overall aeration, improving water and nutrient holding and release capacities, and promoting optimum plant growth (Blunt, 1988; Dunnett and Kingsbury, 2008).

Sloan et al. (2002) found that expanded shale consistently improved overall plant performance better than quartz sand, sphagnum peatmoss and cottonseed hull when they were used as amendments for poorly-drained Austin silty clay soils suggesting its superiority as a soil conditioner for the production of horticultural crops on soils with poor tillage characteristics. In a similar way, Nash et al. (1990) found that a potting medium comprising a mixture of peat moss and expanded shale increased the growth and quality of petunia and impatiens. Smalley et al. (1993) also found that amending soils with products containing expanded shale did not hamper plant performance. Though growth index and plant dry weight of *Salvia* (*Salvia splendens*) and *Vinca* (*Catharanthus roseus*) increased with increasing fertilizer levels for all their treatments, the greatest performance of these plants were observed for treatments amended with the product containing expanded shale, granite sand and composted poultry litter. In another application, Forbes et al. (2004 and 2005) discovered that expanded shale is a potential sorbent for phosphorus in subsurface flow

wetlands because of its high hydraulic conductivity, large surface area and P sorption capacities. Forbes and et al. (2004) found that expanded shale retained  $164 \pm 110$  g P/m<sup>2</sup>/yr in pilot-scale wetlands. Regardless of the generally good experimental reports for expanded shale and/or mixtures containing it, there is a need to understand the mechanisms controlling the basic water and nutrient retention properties of expanded shale and to ascertain its suitability for horticultural and soil amendment uses because expanded shale products can vary depending on geologic origin of the raw materials and differences in the production process (Ferguson, 2005).

In the United States of America, expanded shale is manufactured by multiple companies, most of which are members of the Expanded Shale, Clay, and Slate Institute (ESCSI). Expanded shale has a third to half the weight of regular rock or sand so it is easy to transport but has enough weight to avoid being carried away by water or wind. It is inert, inorganic and therefore not expected to degrade or react with agricultural or horticultural chemicals (TXI, 2009). Given the increasing interest in using EXSH as a soil amendment or as an ingredient in growing media, it is important to understand its interaction with water and nutrients. The first step towards realizing this goal is to thoroughly understand the basic properties of the material itself. The objective of this study therefore was to evaluate the dynamics of water and nutrient adsorption by EXSH.

## **2. Materials and methods**

### **2.1 Basic chemical properties**

#### **2.1.1 pH and electrical conductivity**

Untreated EXSH (10 g) was equilibrated with deionized water (10 mL) for 60 min on a reciprocal shaker and pH was measured in the supernatant with a hydrogen-specific combination electrode. Additionally, pH of the EXSH was measured using 0.01 M CaCl<sub>2</sub> as the equilibrating solution in order to minimize the possible effect of variable background salt concentrations due to soluble components in the EXSH (ASTM D 4972-89). Electrolytic conductivity (EC) of the EXSH was measured by equilibrating 20 g of EXSH with 20 mL of deionized water for 60 min on a reciprocal shaker. Then the supernatant was filtered through Whatman No. 2 filter paper and EC was measured with a dip-type electrode. The filtered supernatant was saved for determination of soluble constituents.

#### **2.1.2 Soluble and labile elements**

EXSH constituents that dissolve easily in water are immediately available to plants and microbes when applied to soil provided they are not immobilized in the soil. To determine the amounts of soluble elements in EXSH, the supernatants saved from the EC analysis were analyzed for Na, K, Ca, and Mg by flame atomic absorption spectrophotometry. In addition to soluble elements, labile elements in the EXSH were measured by extracting 20 g samples of EXSH with 20 mL of a buffered (pH 5) 1 mol L<sup>-1</sup> acetic acid solution (Gibson and Farmer, 1986). Labile elements are not immediately soluble in water, but they are potentially available to plants and soil microorganisms when applied to soil.

#### **2.1.3 Calcium carbonate equivalent**

Calcium carbonate equivalent (CCE) is the measure of a material's ability to neutralize acidity relative to pure calcium carbonate. The comparison is useful for determining how a

material will affect soil pH when mixed with soil. The CCE of EXSH was estimated by suspending 10 g of expanded shale in 50 mL deionized water and then titrating with 0.1 mole L<sup>-1</sup> HCl to a pH 7 endpoint (modified from AOAC, 2005). The amount of pure CaCO<sub>3</sub> required to neutralize an equal amount of acidity was calculated and then CCE was determined by dividing the weight of calculated pure CaCO<sub>3</sub> by the actual weight of EXSH.

*Cation Exchange Capacity (CEC):* Methods that consist of extracting and summing exchangeable cations are not suitable for expanded shale because the manufacturing process produces significant amounts of Ca, Mg, K, and Na oxides that readily dissolve in water. On the other hand, expanded shale is essentially devoid of elements that were present in low concentrations in the original material and were subsequently volatilized during the heating process. One such element is nitrogen. Therefore, the ammonium saturation method was used to measure the CEC of the expanded shale (Chapman, 1965). For this method, the exchange complex of prewashed EXSH was saturated with NH<sub>4</sub><sup>+</sup>, followed by replacement and extraction of NH<sub>4</sub><sup>+</sup> with Na<sup>+</sup>. The extracted NH<sub>4</sub><sup>+</sup> was then quantified using a standard method consisting of alkaline steam distillation, boric acid capture, and titration with standard acid.

## 2.2 Water uptake

Water uptake experiments were designed to evaluate uptake and retention of water by individual EXSH aggregates rather than a bulk amount of the material (Fig.1). The volume of water measured in this manner includes only the internal pores of each EXSH aggregate. It should be noted that measuring the water holding capacity of a bulk volume of EXSH would give a higher value because it would also include water held not only in the internal pores, but also in the pore spaces created between adjacent aggregates.

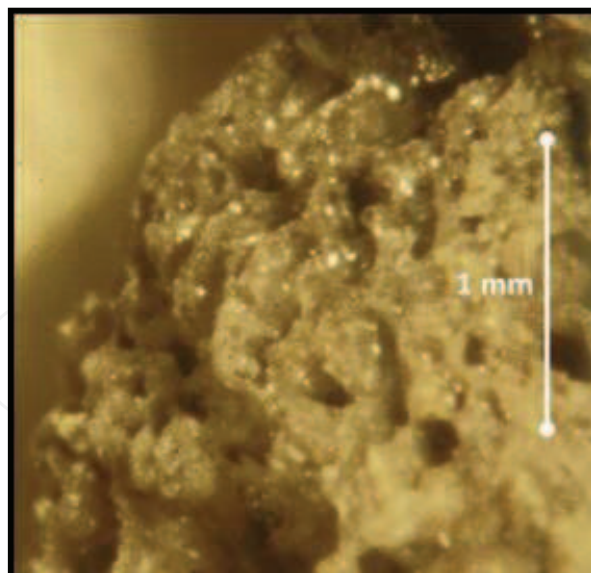


Fig. 1. Magnified view of a single expanded shale particle showing a highly porous exterior surface with range of pore sizes continuing into the interior of the particle

### 2.2.1 Maximum water holding content

To measure maximum water holding capacity, a quantity of EXSH was submerged in water for 72 h in order to completely saturate individual aggregates. At the end of that time, EXSH

was removed from the water, weighed to determine saturated weight, and dried in an oven at 105°C for 48 hours or until a constant weight was obtained. The maximum water holding content was calculated as the difference between the saturated weight and the oven-dry weight divided by the oven-dry weight (w/w).

### 2.2.2 Water adsorption rate

A laboratory experiment was designed to quantify the rate at which water was absorbed by individual EXSH aggregates. Randomly selected individual EXSH aggregates were spread onto water-saturated filter paper. The filter paper was maintained in a saturated condition by the use of paper wicks connected to a water reservoir. This allowed unrestricted diffusion of water from the saturated filter paper into the EXSH aggregates through the point of direct contact. EXSH was left on the water-saturated filter paper for periods ranging from 2.5 minutes to 72 hours. After the specified time, EXSH was removed from the filter paper and oven-dried to determine water content.

### 2.3 Nutrient release from fertilizer-treated expanded shale

A bulk quantity of EXSH was soaked overnight in deionized water to remove soluble nutrients inherent to the material and then oven-dried for 24 h at 60°C. After drying, a quantity of this pre-washed EXSH was nutrient-loaded by soaking for 48 h in a soluble fertilizer solution containing 0.56 g L<sup>-1</sup> NH<sub>4</sub>-N, 1.0 g L<sup>-1</sup> NO<sub>3</sub>-N, 2.44 g L<sup>-1</sup> urea-N, 1.76 g L<sup>-1</sup> P, and 3.32 g L<sup>-1</sup> K. Upon removal from the nutrient solution, the EXSH was rinsed with water to remove surficial deposits of nutrient solution and then allowed to air dry. Untreated and nutrient-loaded EXSH were mixed in ratios of 100:0, 75:25, 50:50, 25:75, and 0:100 (v/v) to create growing media with increasing proportions of nutrient-loaded EXSH. In order to assess the bioavailability of nutrients in the fertilizer-treated EXSH growing media, each blend was sequentially extracted six times with water and the water extracts were analyzed for selected fertilizer-added nutrients. More specifically, a 25 g quantity of each EXSH treatment was placed in a 125 mL flask, immersed in 40 mL of deionized water, and agitated on a reciprocal shaker for 30 min. Then the supernatant was filtered through Whatman No. 2 filter paper and analyzed for NH<sub>4</sub>-N, NO<sub>3</sub>-N and orthophosphate-P. This process was repeated five more times for a total of six extracts.

### 2.4 Bioavailability of adsorbed nutrients

Nutrient bioavailability in a growing medium is best demonstrated by a plant's ability to extract and utilize essential elements. A greenhouse experiment was initiated to test the bioavailability of fertilizer-applied nutrients in the EXSH growing blends described above. Four replicates of each expanded shale blend that contained 0, 25, 50, 75, or 100% nutrient-loaded EXSH were placed in 500 cm<sup>3</sup> greenhouse pots. The EXSH was covered with a thin layer of acid-washed sand so that Romaine lettuce seeds (*Lactuca sativa*) planted on the surface of the growing media would have an adequate germination bed. Approximately 20 seeds were sowed onto the sand layer followed by misting with deionized water twice a day to initiate seed germination. After emergence of lettuce seedlings, the number of plants was thinned to 5 plants per greenhouse pot. Plants were watered daily with deionized water, but no additional nutrients were supplied. After one month, above ground plant tissue was harvested and dried to determine yield. Plant tissue was analyzed for macro- and microelements.

### 3. Results and discussion

#### 3.1 Basic chemical properties

##### 3.1.1 Electrical conductivity (EC)

A 1:1 water extract of EXSH produced an EC of 1.6 dS m<sup>-1</sup> (Table 1). Electrical conductivity (EC) is an indirect measurement of the soluble salt concentration of a material. Excessive salt is detrimental to plant growth. Several researchers have proposed different EC ranges that are limiting for plant growth. Mengel and Kirkby (1982) reported that EC values of less than 2 dS/m in soil will have negligible salinity effects on plant growth while Wright (1986) recommended an EC value of 2 dS/m for healthy and vigorous plant growth. In contrast, Gajdos (1997) indicated that EC values exceeding 1-3 dS/m could be detrimental to plants. In containers, however, Lemaire et al. (1985) found that only EC values greater than 3.5 dS/m were too high for vigorous plant growth. Based on published research findings (Lemaire et al., 1985; Mengel and Kirkby, 1982; Wright, 1986), the EC value measured for EXSH in this study will likely not create salinity problems when added to soil or potting media.

##### 3.1.2 pH

Table 1 shows pH values for EXSH measured in water and in a 0.01 mole L<sup>-1</sup> dilute CaCl<sub>2</sub> solution. The CaCl<sub>2</sub> solution was used to eliminate the effects of background salt concentrations in the EXSH. The dilute calcium chloride method for pH measurement is widely trusted to produce the most reliable results close to the actual pH in the root zone (Handrek and Black, 1994). As a result, the close agreement between the two values (pH 8.3 for water versus pH 8.5 for CaCl<sub>2</sub>) indicates minimal effects due to soluble salts and suggests that both values accurately represent the pH of EXSH. The measured pH values for EXSH reflect the presence of calcium, magnesium, potassium, and sodium oxides (CaO, MgO, K<sub>2</sub>O, and Na<sub>2</sub>O). These oxides were formed as a result of the high temperatures used to manufacture the expanded shale. When EXSH is exposed to water, these oxides hydrolyze to form hydroxides, which can raise pH to as high as 13 in the equilibrium solution. The fact that the equilibrium pH of the EXSH tested in this study only increased to 8.3 suggests that the material contains only relatively small quantities of reactive oxides. Expanded shale materials with higher quantities of reactive oxides would have higher equilibrium pH values.

##### 3.1.3 Calcium carbonate equivalent (CCE)

A material's capacity to neutralize acidity indicates its potential effect on pH when mixed with soil or other growing media materials, such as sphagnum peat moss, compost, sand, vermiculite, etc. Calcium carbonate equivalence can be an important value for alkaline materials, such as EXSH, that will be blended with other growing media ingredients because it predicts the how the EXSH will affect the final pH of the growing media. The calcium carbonate equivalent (CCE) shows the material's effectiveness relative to pure calcium carbonate, which is the standard material for neutralizing soil acidity. The EXSH analyzed in this study had a CCE of 0.2 to 0.3 percent. In other words, EXSH is less than 1% effective at neutralizing soil acidity as calcium carbonate limestone. This indicated that although measured pH values for EXSH were slightly above the broad range of 5.5 to 8 known to support plant growth (Bunt, 1988), it is unlikely that its addition to soil, peat moss, compost, or other growing media ingredients would result in long term increases in the pH of the

growing medium. However, Sloan et al. (2010) reported that the pH of various organic growing media, including sphagnum peat moss, pine bark, compost, and biosolids, were slightly elevated when the proportion of EXSH exceeded 30% (v/v), especially when the initial pH was less than 5.0. It is likely that EXSH will have a positive impact on strongly acidic growing media, such as peat moss and pine bark due to its alkaline pH, but that it will have only a small impact on growing media with neutral to alkaline pH values, such as compost and biosolids.

Parameter	Measured Value	
	Mean	SD†
Electrical conductivity, dS m <sup>-1</sup>	1.6	(± 0.2)
pH		
(1:1 - water)	8.25	(± 0.62)
(1:1 - 0.01 mol L <sup>-1</sup> CaCl <sub>2</sub> )	8.47	(± 0.21)
Calcium carbonate equivalent (CCE), %	0.2 - 0.3	
Water holding capacity, %	37.8	(± 1.35)
Cation exchange capacity (CEC), cmole kg <sup>-1</sup>	2.75	(± 0.15)
Extractable elements, mg/kg		
Water soluble		
Sodium (Na)	55.0	(± 6.5)
Potassium (K)	64.2	(± 6.0)
Magnesium (Mg)	29.0	(± 2.8)
Calcium (Ca)	88.3	(± 10.6)
Acid soluble§		
Sodium (Na)	87.3	(± 5.3)
Potassium (K)	126.8	(± 21.0)
Magnesium (Mg)	660	(± 40)
Calcium (Ca)	843	(± 59)

† Standard deviation of the mean. Shown in parentheses in table

§ 1 mol L<sup>-1</sup> sodium acetate buffered to pH 5 with acetic acid.

Table 1. Basic chemical and physical properties of expanded shale (ExSh)

### 3.1.4 Soluble and labile elements

Expanded shale was extracted with water to measure the concentration of soluble elements and with acidified sodium acetate to measure potentially plant-available (labile) elements. Concentrations of water-soluble sodium (Na), potassium (K), magnesium (Mg), and calcium (Ca) in EXSH were small and similar in magnitude (Table 1). Measured concentrations for these elements are by far lower than guideline values established for cultivation which are < 857 mg/kg for Ca, <111.3 mg/kg for Na and <150 mg/kg for Mg (Fischer and Penningsfeld, 1979; Fisher and Meinken, 1995) and are consistent with the low EC values measured. When EXSH was extracted with acidified sodium acetate, only slightly higher amounts of Na and K were extracted, whereas the amounts of Mg and Ca were increased 20-fold and 10-fold, respectively. The data suggests that the heating process used to manufacture the expanded shale in EXSH produces small amounts of Na, K, Mg, and Ca salts that are easily soluble in water, plus larger amounts of Mg and Ca oxides that will dissolve in weak acid (pH 5). Potassium, Mg, and Ca are essential plant nutrients. Therefore addition of EXSH to soil will

provide small amounts of immediately plant-available K, Mg, and Ca plus larger amounts of more slowly available Mg and Ca. However, the inherent concentrations of these elements in EXSH are too low to significantly affect plant nutrition – either beneficially or detrimentally when it is added to soil.

### 3.2 Water uptake

Two facets of water uptake by EXSH were investigated: 1) the maximum water-holding capacity and 2) the rate of water uptake. The maximum water holding capacity provides information on the total porosity in EXSH aggregates. After soaking in water for at least 72 hr, the EXSH tested in our study contained 37.8% water (Table 1). This value includes the amount of water in EXSH when 100% of the pores were saturated plus the small amount of water on the surfaces of its aggregates. The water-holding capacity of EXSH shows that it can be a significant source of water storage in soil. However, a typical silt loam soil will have a greater available water holding capacity than EXSH. Therefore, EXSH would be an appropriate soil amendment for soils that retain excess water and exhibit poor drainage characteristics. Sloan et al. (2002) reported that 3 to 6 mm diameter expanded shale, similar to that used in this study, effectively increased the root mass of pansy plants (*Viola x wittrockiana*) compared to un-amended clay soil during an excessively wet growing season.

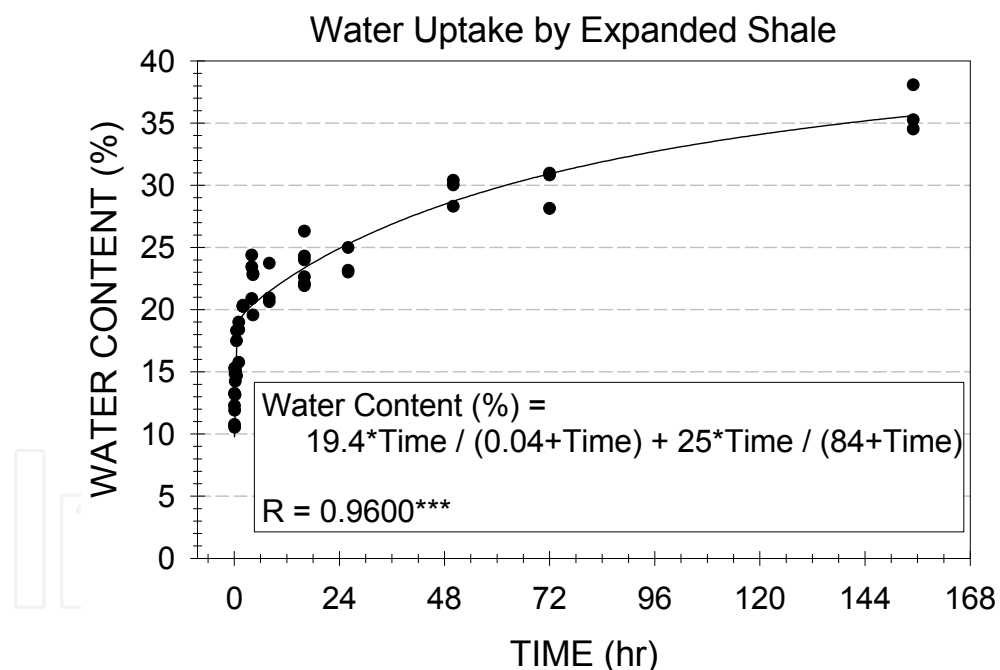


Fig. 2. Rate of water diffusion into expanded shale (EXSH) from a saturated porous medium. Data was fitted to a double rectangular hyperbolic function to create a predictive uptake equation. \*\*\* Significant at the 0.001 level of probability

The rate at which EXSH can absorb water is perhaps more important than its total water holding capacity. The water uptake rate provides information on the size of pores in the EXSH aggregates and can be related to the amount of water that will be available to plants. Figure 2 shows that EXSH absorbed 15% of its weight in water within 10 minutes and 20% within 2 hours. The water uptake rate slows considerably beyond 2 hours, but continued to



increase to a maximum of 36% at 150 hours. The experiment was designed so that water could only enter the EXSH aggregates via capillary diffusion. When an EXSH aggregate was placed in contact with a saturated porous medium, water quickly diffused throughout the aggregate's surface layer until the exterior pores of the aggregate were saturated. A magnified view of an individual expanded shale aggregate shows the exterior pores to be relatively large and evenly distributed around the surface (Fig. 1). Water is held in these pores at relatively low tension and is therefore easily available for plant uptake, but it is also susceptible to rapid evaporation. This water accounts for 15 to 20% (w/w) of the total water holding capacity of EXSH. The remaining 16 to 21% of the total water is held in the much smaller interior pores of the EXSH aggregates. This water is held at relatively high tensions and is therefore mostly unavailable for plant use.

### 3.3 Nutrient release from fertilizer-treated EXSH

The porous nature of EXSH allows it to absorb water and therefore, any chemical constituents dissolved in water will also be absorbed by the EXSH aggregate. For this experiment, EXSH was loaded with nutrients by soaking it in a fertilizer solution for 48 hours, followed by rinsing in deionized water, and then air-drying. The reason for rinsing the fertilizer-impregnated EXSH was to ensure that nutrients released from the aggregates were from the internal pores and not residual surface deposits of fertilizer. Based on results from the water uptake experiment (Fig. 2), 5 to 10% of the pore volume was not saturated with fertilizer solution because the EXSH was soaked for only 48 hours whereas 150 hours was needed to attain complete saturation. Also, it is likely that 10 to 15% of the fertilizer was removed from the exterior EXSH during the rinsing process. These two factors probably reduced the total nutrient-supplying capacity of the fertilizer-treated EXSH by 15 to 25%.

A portion of each fertilizer-treated EXSH medium containing 0, 25, 50, 75, or 100% nutrient-loaded EXSH was sequentially extracted with water to determine the kinetics of nutrient release. Nutrients extracted with water are immediately available for plant uptake. Nitrate-nitrogen ( $\text{NO}_3\text{-N}$ ), ammonium-nitrogen ( $\text{NH}_4\text{-N}$ ), and phosphate-phosphorus ( $\text{PO}_4\text{-P}$ ) were measured in all extracts. Although the fertilizer used to impregnate the EXSH with nutrients also contained urea-N, we did not measure that form of N in the water extracts. In general, the concentration of each constituent decreased with each sequential extract (Fig. 3). This demonstrates that a portion of soluble nutrients absorbed by EXSH can later redissolve and become available for plant uptake. If EXSH were present in a growing medium, individual aggregates would either absorb or release nutrients depending on the concentration gradient of the surrounding solution. In practical terms, if EXSH aggregates in the growing medium were surrounded by relatively pure water, then nutrients would diffuse from the interior pores towards the exterior pores due to a concentration gradient. In a similar but reverse process, nutrients from a fertilizer solution passed through the medium would possibly diffuse into the interior pores of the EXSH aggregates in response to the nutrient gradient. Phosphorus retention by expanded shale in constructed wetlands as reported by Forbes et al. (2004 & 2005) may have been related to this diffusion phenomenon.

Nitrate-N,  $\text{NH}_4\text{-N}$  and  $\text{PO}_4\text{-P}$  each have different binding mechanisms to solid particles in a growing medium. Nitrate-N has a negative charge and exists as a free (i.e., unbound) ion in typical soil conditions. It moves freely through the soil with soil water. Ammonium-N has a positive charge and is attracted to negatively charged exchange sites found in typical soils.

This allows it to be held more tightly in the soil and makes it less susceptible to loss through leaching. Orthophosphate-P is an oxyanion and tends to strongly bind to various soil components, primarily aluminum and iron minerals at low pH (<5.5), and calcium and magnesium minerals at high pH (>7.6).

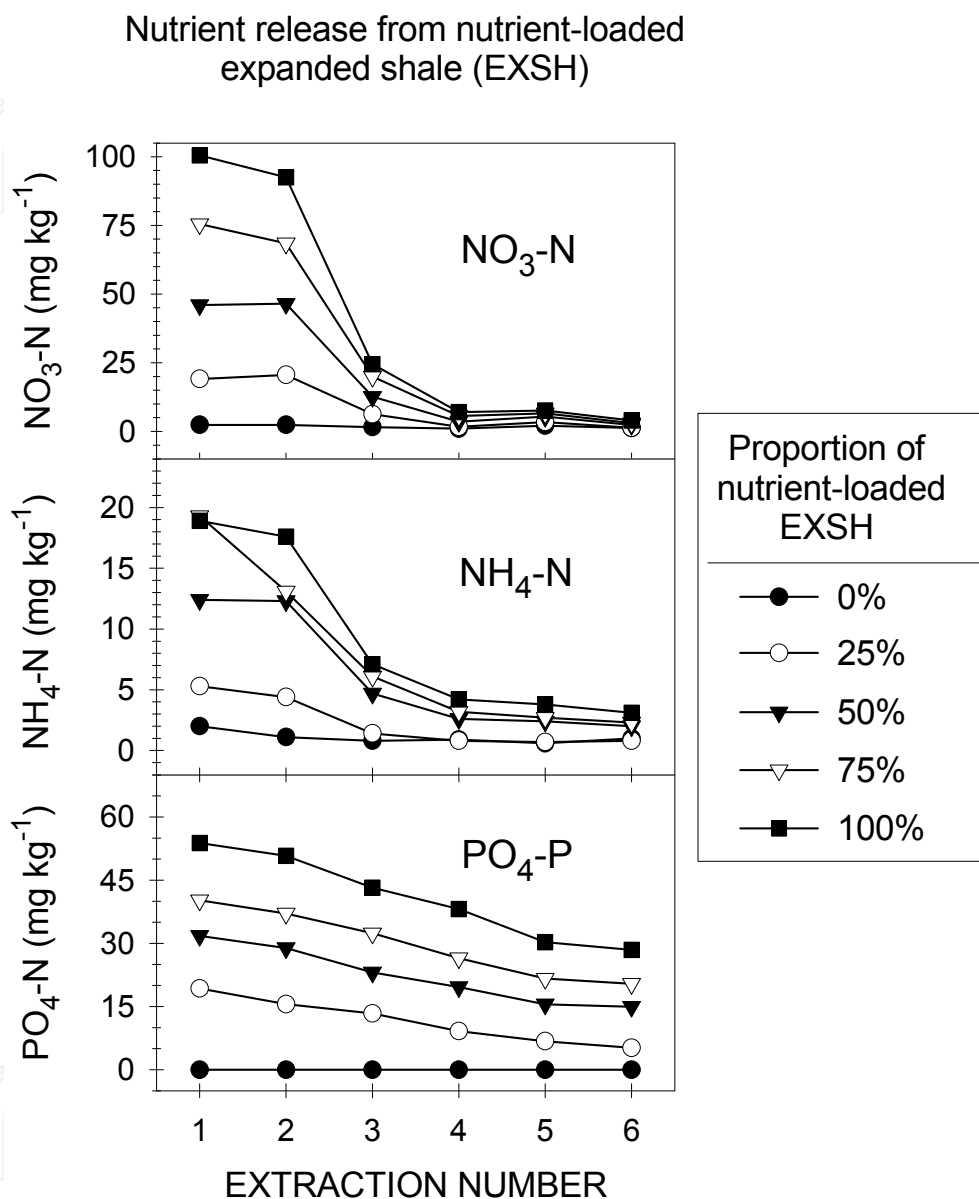


Fig. 3. Sequential extraction of NO<sub>3</sub>-N, NH<sub>4</sub>-N, and orthophosphate-P from expanded shale (EXSH) loaded with N, P, and K fertilizer

The different chemistries of NO<sub>3</sub>-N, NH<sub>4</sub>-N, and PO<sub>4</sub>-P were apparent in the sequential extraction data presented in Fig. 3. Nitrate-N was easily extracted with water. This was best illustrated with the 100% fertilizer-treated EXSH treatment. The first extraction removed 100 mg NO<sub>3</sub>-N per kg of EXSH. By the fourth extraction, the amount extracted had decreased to 7 mg kg<sup>-1</sup>, but was still slightly greater than the concentration for the untreated 0% control (i.e., EXSH without fertilizer pre-treatment). The remaining extractions continued to remove small amounts of NO<sub>3</sub>-N that were slightly greater than the control. The extraction of NO<sub>3</sub>-N

from fertilizer-treated EXSH was consistent with water uptake data that showed very rapid water uptake followed by a much slower uptake phase (Fig. 2). In the absence of any attractive force between  $\text{NO}_3\text{-N}$  and the EXSH aggregate,  $\text{NO}_3\text{-N}$  would diffuse out of the EXSH in a fashion similar to water. The rapid release of  $\text{NO}_3\text{-N}$  with the initial extractions corresponds to diffusion of  $\text{NO}_3\text{-N}$  from larger pores near the surface of the aggregate (Fig. 1). The much lower, yet significantly higher concentrations of  $\text{NO}_3\text{-N}$  released with the later extractions relative to the control suggest diffusion of  $\text{NO}_3\text{-N}$  from smaller pores deeper inside the EXSH aggregate.

Smaller amounts of  $\text{NH}_4\text{-N}$  were extracted from fertilizer-treated EXSH compared to  $\text{NO}_3\text{-N}$  because the fertilizer source used to pretreat the EXSH contained  $1000 \text{ mg L}^{-1} \text{ NO}_3\text{-N}$ , but only  $560 \text{ mg L}^{-1} \text{ NH}_4\text{-N}$ . However, the magnitudes of the difference between desorption of  $\text{NO}_3\text{-N}$  and  $\text{NH}_4\text{-N}$  could not be explained by fertilizer composition alone. It is likely that some of the  $\text{NH}_4\text{-N}$  was retained in the EXSH by cation exchange capacity. The CEC analysis indicated that EXSH had a small but significant CEC of  $2.75 \text{ cmol kg}^{-1}$  (Table 1), which was adequate to retain up to  $385 \text{ mg kg}^{-1} \text{ NH}_4\text{-N}$ . Other than the quantity extracted, release of  $\text{NH}_4\text{-N}$  from fertilizer-treated EXSH followed a trend similar to that shown by  $\text{NO}_3\text{-N}$  suggesting that diffusion processes were a significant factor in controlling the release of  $\text{NH}_4\text{-N}$  from the porous EXSH.

Phosphorus release curves from fertilizer-treated EXSH were distinct from those for  $\text{NO}_3\text{-N}$  and  $\text{NH}_4\text{-N}$  (Fig. 3). The amount of  $\text{PO}_4\text{-P}$  released decreased linearly with each successive extraction. There was no rapid release with the initial extractions as seen for  $\text{NO}_3\text{-N}$  and  $\text{NH}_4\text{-N}$ . This suggests that other factors, in addition to diffusion, controlled the release of  $\text{PO}_4\text{-P}$  from EXSH. The presence of calcium- and magnesium- oxides in EXSH created conditions favorable for the adsorption and/or precipitation of  $\text{PO}_4\text{-P}$ . Experience with calcareous soils has shown that dicalcium phosphate ( $\text{CaHPO}_4$ ) is commonly a first precipitation product following application of phosphate fertilizer (Sposito, 1989). EXSH contained a substantial amount of extractable Ca as well as  $\text{pH} > 8.3$  (Table 1) suggesting the possible precipitation of the relatively soluble dicalcium phosphate ( $\text{CaHPO}_4$ ) mineral. Phosphate adsorption and precipitation are reversible processes, particularly in an EXSH medium, which is much less complex than a soil medium. Dissolution and release of adsorbed and precipitated phosphorus from EXSH would occur at a slower and more constant rate than would be expected from simple diffusion due to a concentration gradient. The practical implication of these results is that phosphorus-impregnated EXSH could function as a relatively effective slow-release phosphorus fertilizer. Forbes et al. (2004 & 2005) also found that expanded shale was effective at removing soluble P from effluent water in a constructed wetland.

### 3.4 Lettuce growth in fertilizer-treated EXSH

The true test of nutrient bioavailability is whether plants are able to extract and utilize nutrients from the growing medium. For this experiment, Romaine lettuce (*Lactuca sativa longifolia*) was grown in the same fertilizer-treated, EXSH media described in the preceding sequential extraction discussion. Briefly, the growing media consisted entirely of EXSH where 0, 25, 50, 75, or 100% (v/v) had been previously impregnated with fertilizer solution. Lettuce was grown for approximately 45 days with no additional fertilizer application and then harvested to determine shoot and root yields as well as nutrient content of the aboveground tissue (Fig. 4).



Fig. 4. Increasing the proportion of nutrient-loaded expanded shale (EXSH) in the growing media from 0 to 100% resulted in significant increases in the size of Romaine lettuce

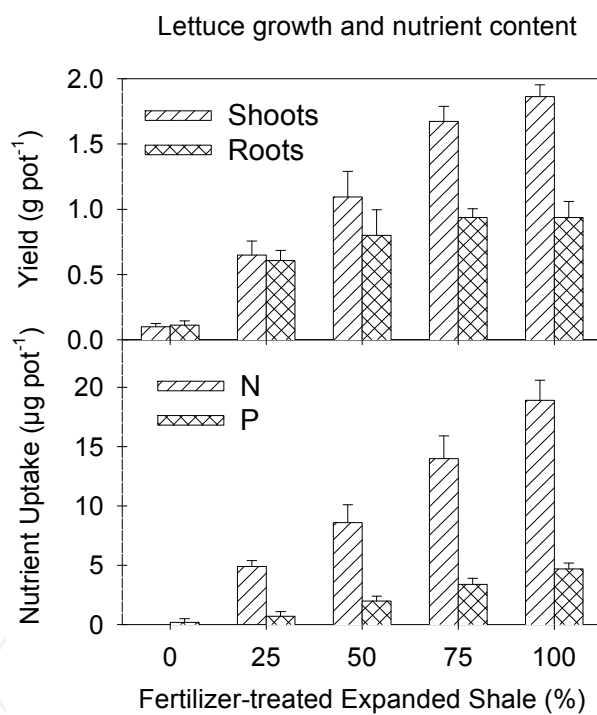


Fig. 5. Lettuce yields and total nutrient uptake for lettuce grown in fertilizer-treated expanded shale

Results for the shoot and root biomass yield as well as total nitrogen and phosphorus content of the aboveground tissue are shown in Fig. 5. The quantity of aboveground lettuce shoots increased linearly as the percentage of fertilizer-treated EXSH in the growing medium was increased (Fig. 5). Root mass also increased with increasing proportion of fertilizer-treated EXSH, but reached a maximum at the 75% fraction. Total uptake of nitrogen and phosphorus also increased linearly with increasing proportion of fertilizer-treated EXSH (Fig. 5). The yield and nutrient uptake data demonstrate that nutrients in the fertilizer-treated EXSH medium were taken up and assimilated by plants. The level of

fertility was sufficient to support lettuce growth for approximately 45 days with no additional fertilization. Following harvest of the lettuce plants, additional lettuce seeds were planted into these same EXSH growing media treatments, but no fertilizer was added. This second planting of lettuce failed to thrive in the same way as the first planting (data not shown) suggesting that the ability of EXSH to provide a reservoir of abundant plant nutrients is limited without additional fertilizer applications. Other studies (Nash et al., 1990; Smalley et al., 1993; Sloan et al., 2002) have also reported good plant growth in growing media containing EXSH as a major ingredient. Together these observations suggest that expanded shale can retain and release sufficient amounts of nutrients for healthy plant growth either alone or in a mixture, but additional fertilization will occasionally be needed depending on the intensity of plant growth and the frequency of leaching events. EXSH is therefore suitable as an amendment for such growing media as soil, potting mixes, and green roof substrates.

#### 4. Conclusions

Expanded Shale (EXSH) used in this study had small amounts of Ca, Mg, K, and Na oxides. This was confirmed by measured pH values of  $>8$  in both water and  $0.01 \text{ mol L}^{-1} \text{ CaCl}_2$ . However, a low CCE of 0.2-0.3% suggests that EXSH is unlikely to significantly affect pH when added to a more buffered materials such as soil, sphagnum peat moss or compost. The concentrations of water soluble Na, K, Mg and Ca were small and consistent with the measured EC of  $1.6 \text{ dS m}^{-1}$ . Acid extraction of these elements from EXSH yielded higher amounts with Mg and Ca concentrations increasing by 20-fold and 10-fold, respectively, suggesting that when the product is added to a growing medium, small quantities of plant-available Na, K, Mg and Ca can be provided initially and followed later by larger amounts of Mg and Ca in a slow release fashion. However, the concentration of these elements in EXSH is insufficient to affect plant nutrition positively or negatively. The release of  $\text{NO}_3\text{-N}$  and  $\text{NH}_4\text{-N}$  from nutrient-loaded EXSH was rapid and controlled by mainly by diffusion mechanisms, although cation exchange capacity was also responsible for retention of  $\text{NH}_4\text{-N}$ . The movement of  $\text{PO}_4\text{-P}$  on the other hand was influenced by both diffusion and the presence of calcium and magnesium oxides in the EXSH. The presence of these oxides enhanced the reversible processes of  $\text{PO}_4\text{-P}$  adsorption and precipitation in the EXSH medium. In practical terms, the ability of EXSH to reversibly adsorb fertilizer P suggests EXSH may be a suitable material for use as a slow-release phosphorus fertilizer. In this study, both the shoot and root mass of romaine lettuce increased with increasing proportions of fertilizer treated EXSH in the growing medium. This demonstrates that nutrients were released by the fertilizer treated EXSH and assimilated by the lettuce to support growth. The linear relationship between total nitrogen and phosphorus uptake and the proportion of fertilizer impregnated EXSH in the growing medium confirms the ability of EXSH to release nutrients for plant uptake.

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## **Principles, Application and Assessment in Soil Science**

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Our dependence on soil, and our curiosity about it, is leading to the investigation of changes within soil processes. Furthermore, the diversity and dynamics of soil are enabling new discoveries and insights, which help us to understand the variations in soil processes. Consequently, this permits us to take the necessary measures for soil protection, thus promoting soil health. This book aims to provide an up-to-date account of the current state of knowledge in recent practices and assessments in soil science. Moreover, it presents a comprehensive evaluation of the effect of residue/waste application on soil properties and, further, on the mechanism of plant adaptation and plant growth. Interesting examples of simulation using various models dealing with carbon sequestration, ecosystem respiration, and soil landscape, etc. are demonstrated. The book also includes chapters on the analysis of areal data and geostatistics using different assessment methods. More recent developments in analytical techniques used to obtain answers to the various physical mechanisms, chemical, and biological processes in soil are also present.

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