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## Base cations and micronutrients in soil aggregates as affected by enhanced nitrogen and water inputs in a semi-arid steppe grassland

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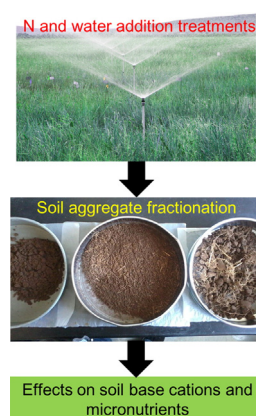
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### HIGHLIGHTS

- Higher base cations were detected in microaggregates compared to macroaggregates.
- Nitrogen addition decreased effective cation exchange capacity in macroaggregates.
- Nitrogen addition decreased Ca and Mg but increased extractable Fe, Mn and Cu.
- Water addition increased exchangeable Na while decreased available Fe and Mn.

### GRAPHICAL ABSTRACT



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### ABSTRACT

The intensification of grassland management by nitrogen (N) fertilization and irrigation may threaten the future integrity of fragile semi-arid steppe ecosystems by affecting the concentrations of base cation and micronutrient in soils. We extracted base cations of exchangeable calcium (Ca), magnesium (Mg), potassium (K), and sodium (Na) and extractable micronutrients of iron (Fe), manganese (Mn), copper (Cu), and zinc (Zn) from three soil aggregate sizes classes (microaggregates, <0.25 mm; small macroaggregates, 0.25–2 mm; large macroaggregates, >2 mm) from a 9-year N and water field manipulation study. There were significantly more base cations (but not micronutrients) in microaggregates compared to macroaggregates which was related to greater soil organic matter and clay contents. Nitrogen addition significantly decreased exchangeable Ca by up to 33% in large and small macroaggregates and exchangeable Mg by up to 27% in three aggregates but significantly increased extractable Fe, Mn and Cu concentrations (by up to 262%, 150%, and 55%, respectively) in all aggregate size classes. However, water addition only increased exchangeable Na, while available Fe and Mn were decreased by water addition when averaging across all N treatments and aggregate classes. The loss of exchangeable Ca and Mg

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## 1. Introduction

Micronutrient availability constrains net primary productivity (NPP) (Li et al., 2008, 2010), and deficiencies in soil micronutrients, including Fe, Mn, Cu, and Zn, are a problem threatening food production worldwide (Jones et al., 2013). Base cations (i.e., exchangeable Ca, Mg, K, Na) are the predominant exchangeable cations in the calcareous soils (Zhang et al., 2013). They are essential for soil buffering capacity particularly in soils affected by atmospheric acid deposition (Lieb et al., 2011), serve as good indicators of soil fertility (Zhang et al., 2013), and are critical nutrients for both plant and microbial metabolism (Cheng et al., 2010). For instance, Ca regulates plant cell signaling, cell division, and carbohydrate metabolism (McLaughlin and Wimmer, 1999), and Mg is important for photosynthesis and energy storage (Lucas et al., 2011). Biogeochemical processes may be driven by base cation and micronutrient supply; for instance, root-surface phosphatase activity is correlated with available Ca and Mg (Gabbriellini et al., 1989) and Mg and Zn availability are important for litter decomposition (Powers and Salute, 2011).

The availability of base cations and micronutrients is influenced by environmental changes, such as altered N and water availability (Treseder, 2008). The availability of base cations varies with edaphic properties, such as soil pH (Katou, 2002), organic matter fractions (Oorts et al., 2003) and soil particle sizes (Beldin et al., 2007). Prolonged N inputs generally causes soil acidification and subsequent losses of soil cations (McLaughlin and Wimmer, 1999; Cheng et al., 2010), and micronutrient availability may increase under soil acidification (Malhi et al., 1998) causing toxicity to both plants and soil microorganisms in extreme cases (Bowman et al., 2008; Horswill et al., 2008). Changes in precipitation regime and soil moisture levels may interact with inorganic N affecting soil microbial activities (Wang et al., 2015a) including the decomposition of soil organic matter (SOM) and nutrients release and their subsequent transport in the soil (Dungait et al., 2012; Nielsen and Ball, 2014). In sandy soils, increased precipitation might promote leaching of nitrate and counter-ions (such as base cations) (Ochoa-Hueso et al., 2014).

Soil aggregate structure predominantly controls SOM dynamics (Six et al., 2004) and microbial activities (Dorodnikov et al., 2009), and soil aggregate stability can serve as an indicator for grassland ecosystem health (Reinhart et al., 2015). In comparison with macroaggregates, microaggregates provide preferential sites for soil C stabilization (Wang et al., 2015b) and the SOM herein is more microbial-processed as evidenced by natural abundance stable  $^{13}\text{C}$  values (Gunina and Kuzyakov, 2014; Wang et al., 2015b). More microbial-processed SOM (i.e. more functional groups) and potentially higher mineral contents within microaggregates (Creamer et al., 2011) would purportedly provide more binding sites for base cations and micronutrients. Therefore, dynamics of soil base cations and micronutrients in aggregate scale would be a good indicator for soil health and for the potential of metal nutrients sustainability. However, studies concerning aggregate-scale distribution of base cations and micronutrients under enhanced N input and precipitation are still rarely seen.

Semi-arid steppe grasslands support diverse animal and plant species (Kang et al., 2007) and are experiencing or will experience enhanced atmospheric nitrogen (N) deposition and precipitation (Niu et al., 2009; Bai et al., 2010; Zhang et al., 2014). Previously, we demonstrated that water addition promoted the incorporation of microaggregates into macroaggregates and enhanced decomposition rates within microaggregates compared to macroaggregates, and that the addition of N depressed extracellular enzyme activities within soil

aggregates as a result of soil acidification, after 9-years in a field experiment in the semi-arid steppe grasslands of Inner Mongolia (Wang et al., 2015a,b). In this study, we investigated the changes of base cations and micronutrients within the soil aggregates. We hypothesized that (1) both concentrations of base cations and micronutrients would increase in microaggregates because of the increased abundance of adsorption sites provided by greater SOM and mineral contents therein, and (2) that increased N and water inputs would decrease base cations and increase the availability of micronutrients within soil aggregates due to soil acidification and leaching in the free-draining soil.

## 2. Materials and methods

### 2.1. Study sites and experiment design

The experiment was conducted in the Inner Mongolia Restoration Ecological Research Station (IMRERS) in the south of Duolun County, Inner Mongolia, China ( $42^{\circ}02'27''\text{N}$ ,  $116^{\circ}17'59''\text{E}$ , elevation 1324 m a.s.l). The topography of the experimental area is flat. The mean annual temperature is  $2.1^{\circ}\text{C}$ , ranging from  $-17.8^{\circ}\text{C}$  in January to  $18.8^{\circ}\text{C}$  in July, and mean annual precipitation is 379.4 mm (Xu et al., 2012). The soil is a Haplic Calcisols according to the FAO classification (IUSS Working Group WRB, 2015) with a texture of sandy loam (0–10 cm): 63% sand, 20% silt, and 17% clay (Wang et al., 2014). The chemical characteristics of the 0–10 cm depth of whole soil are given in Wang et al. (2014).

The experimental design is described in detail in Wang et al. (2014). Briefly, experimental plots were set up and run for 9 years. A split-plot design was applied with water and N addition being the two treatments. In April 2005, twelve  $8\text{ m} \times 8\text{ m}$  plots were established in each of seven treatment blocks with 1 m buffer zone between any two adjacent plots. Each block was divided into two main plots with either ambient precipitation or 180 mm water addition) as treatments. Each main plot was divided into six subplots and nitrogen treatments (urea pellets) were applied to a randomly selected subplot (dispersed on the top of the soil) (Xu et al., 2012). Two subplots were phosphorus addition treatments which were not considered in this study. Additional water (approximately 50% of mean annual precipitation) was added to the water addition plots by sprinkling 15 mm weekly during the middle of the growing season from June to August as over 65% of annually total precipitation occurs during this time. The chemical composition of the irrigation water was listed as Table S1. Nitrogen additions were split applications with half applied in early May and the other half in late June at the rates of 0 (control plots, defined as CK), 5 ( $\text{N}_5$ ), 10 ( $\text{N}_{10}$ ), and 15 ( $\text{N}_{15}$ )  $\text{g N m}^{-2} \text{yr}^{-1}$  (Xu et al., 2012). The background N inputs (atmospheric deposition plus fertilizer application) in this area are about  $5\text{ g N m}^{-2} \text{yr}^{-1}$ , so these manipulations represent 100%, 200% and 300% surplus of nitrogen compared to the background N inputs (Wang et al., 2015a).

### 2.2. Soil sampling and aggregate-size fractionation

In September 2013, top soils (0–10 cm) were sampled by compositing five randomly selected cores within each plot from four out of seven blocks. Fresh soil samples were stored at  $4^{\circ}\text{C}$  during transportation to the laboratory. Soil aggregates were isolated by a dry-sieving method according to Dorodnikov et al. (2009) to minimize the disruption in microbial activities and to prevent leaching of available nutrients by wet sieving. Briefly, fresh bulk soil samples (gravimetric water content of 10–15%) were gently passed through a 5 mm screen and transferred

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