

## Salt Additions Alter Short-term Nitrogen and Carbon Mobilization in a Coastal Oregon Andisol

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Deposition of sea salts is commonly elevated along the coast relative to inland areas, yet little is known about the effects on terrestrial ecosystem biogeochemistry. We examined the influence of NaCl concentrations on N, C, and P leaching from a coastal Oregon forest Andisol in two laboratory studies: a rapid batch extraction (~1 d) and a month-long incubation using microlysimeters. In the rapid extractions, salt additions immediately mobilized significant amounts of ammonium and phosphate but not nitrate. In the month-long incubations, salt additions at concentrations in the range of coastal precipitation increased nitrate leaching from the microcosms by nearly 50% and reduced the mobility of dissolved organic carbon. Our findings suggest that coupled abiotic–biotic effects increase nitrate mobility in these soils: exchange of sodium for ammonium, then net nitrification. Changes in sea salt deposition to land and the interactions with coastal soils could alter the delivery of N and C to sensitive coastal waters.

MARINE INPUTS CAN BE IMPORTANT for element supply to terrestrial ecosystems, particularly in near-coastal areas. These inputs can occur via physical and biological processes (Farrell 1995; Polis and Hurd, 1996) and leave a potentially important but poorly documented spatial imprint on near-coastal systems. Sea salt aerosols are formed by wind action, which produces waves that crest and bubbles that break, injecting drops of seawater into the atmosphere (Lewis and Schwartz, 2004). Once airborne, some sea salt aerosols are entrained upward, whereas others fall back to the sea. Some of these aerosols make their way onto land and are deposited to terrestrial ecosystems. There has been some recognition of the influence of sea salt aerosols in cation supply to terrestrial ecosystems (Farrell, 1995; Chadwick et al., 1999) and in aquatic acidification (Wright et al., 1988; Heath et al., 1992; Sullivan et al., 1988). Based on studies in the eastern and western United States, sea salt inputs are highest within 5 to 7 km of the coast and decrease gradually beyond this distance (Valiela et al., 1998; Compton et al., 2003).

The effects of sea salt deposition may extend beyond the direct supply of chloride or cations, yet few studies link sea salts and the biogeochemistry of other elements such as C, N, and P. Variations in sea salt deposition recently have been implicated in changing dissolved organic carbon (DOC) concentrations over time in Europe and North America (Monteith et al., 2007). Heath et al. (1992) showed a pulse of nitrate in stream water during a sea salt episode in Acadia National Park, Maine, but did not address the mechanism. Green and Cresser (2008) identified a correlation between road salt additions and the form and amount of nitrogen leaching from roadside areas; however, no experimental studies have identified a similar pattern for coastal aerosols.

The Pacific Northwest coast of the United States receives some of the highest levels of chloride deposition in the United States (National Atmospheric Deposition Program/National Trends Network). Chloride in coastal Oregon soil solution and streams decreases with distance from the ocean (Bockheim and Langley-Turnbaugh, 1997; Wigington et al., 1998; Compton et al., 2003). Soil N levels and rates of N cycling are also quite high in some areas of the Oregon Coast Range because these ecosystems are shaped by historical cycles of fire and N<sub>2</sub>-fixing red alder (Perakis et al., 2011). Thus, sea salts could interact with rapidly cycling N. In a study of 27 subbasins of a coastal Oregon river, stream

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**Abbreviations:** DOC, dissolved organic carbon; DON, dissolved organic nitrogen.

nitrate concentrations also decreased with increasing distance from the ocean (Compton et al., 2003). In that study, the difference in stream N concentrations and fluxes from the inland areas to the coast is substantial, ranging from <0.1 to 3 mg N L<sup>-1</sup>, and export ranges from 2 to 31 kg N ha<sup>-1</sup> yr<sup>-1</sup>. Some of the variation was due to the presence of N<sub>2</sub>-fixing red alder, but the pattern of decreasing stream N concentrations away from the coast persisted in watersheds with very low alder and no other differences in land use. The surprising pattern of higher stream nitrate concentrations near the coast suggests that sea salt deposition may cause unexpected effects on internal N cycling and retention in Oregon coastal watersheds.

The observed landscape pattern of decreasing stream nitrate away from the coast led us to ask if sea salts increase nitrate mobility in soil. Our goal was to address this question in a controlled laboratory setting using the range of salt deposition levels occurring in the field. We expected that sodium chloride would increase the availability of ammonium, as shown previously (Duckworth and Cresser, 1991), but further hypothesized that nitrate mobility would be greater in salt-treated mineral soils. We examine the effects of NaCl additions mimicking sea salt deposition on soil chemistry, focusing on leaching of soil N, C, and P, in a soil type that is widely distributed across our study watersheds.

We designed two experiments to examine the rapid vs. month-long effects of sea salts on soil N cycling. Short-term extractions were conducted to isolate the rapid, largely chemical interactions between sea salts and extractable N. We also established soil microcosms and subjected them to repeated leaching for 1 mo. These two experiments allowed us to examine the immediate vs. month-long effects of sea salts on the interactions between soil exchange processes and microbial activity and provide insight into the mechanisms of N mobilization. If nitrate mobility was higher immediately in both experiments, then an abiotic exchange reaction between nitrate and chloride might explain the increase. If nitrate mobility and leaching only increased in the longer-term experiments, then interruption of microbial N mineralization-immobilization dynamics would be the primary explanation. Our study uses well mixed soils in microcosms to examine whether N mobility in a mature forest mineral soil responds to increases in salt concentrations near and above levels received in deposition. The goal of this study was to determine whether salt additions, in the concentrations range observed in deposition, could alter release of soil C, N, and P from the soils of our study area.

## Materials and Methods

The study area is in Cascade Head Experimental Forest, part of the Siuslaw National Forest (45°02'50" N, 123°54'21" W), approximately 2 km north of Otis, OR, and 7 km from the Pacific Ocean. The elevation is 210 m above sea level, and the mean annual temperature is 10°C. Mean annual bulk precipitation is 2500 mm, most of which falls as rain between October and June (USEPA, unpublished data). The slope is approximately 12%. We examined soils from a mature sitka spruce-western hemlock (*Picea sitchensis*-*Tsuga heterophylla*) forest along the Oregon Coast. The stand is dominated by sitka spruce, western hemlock, and scattered Douglas-fir

(*Pseudotsuga menziesii*), originating after the stand-replacing Nestucca fire of the mid-1800s (Harcombe et al., 1990). The soil is classified as an Andisol of the Tolovana soil series (USDA, 1997), which are isomesic Typic Fulvudands formed in colluvium from sandstone. These are deep, well drained soils with a loam texture at the surface. The Tolovana series covers approximately 21% of the Salmon-Neskowin basin and is the most prevalent soil series in the basin. Atmospheric wet deposition of N in Alsea, Oregon, approximately 75 km to the southeast in the Oregon Coast Range, is among the lowest observed in the conterminous United States (averaging <0.7 kg ha<sup>-1</sup> yr<sup>-1</sup> from 1980 to 2003), whereas wet atmospheric deposition of Cl<sup>-</sup> is among the highest measured (averaging 23.8 kg ha<sup>-1</sup> yr<sup>-1</sup> from 1980 to 2003) (National Atmospheric Deposition Program; obtained April 2005; <http://nadp.sws.uiuc.edu/>). We collected soils from a site inland from the coast 7 km to avoid sites with a history of extremely high NaCl loading observed closer to the coast (>100 kg Cl<sup>-</sup> ha<sup>-1</sup> yr<sup>-1</sup>, inferred from Na and Cl export) (Compton et al., 2003).

Soils were collected on 12 June 2002 by sampling the 0- to 15-cm depth in the mineral soil using a soil corer (5.5 cm diameter; *n* = 10 cores). Soils from the 10 cores were moist sieved to remove gravel (>2 mm), then all 10 cores were mixed together and homogenized. This field moist bulk sample was stored at 4°C until initiating each experiment. We used the same bulk soil sample in both experiments so that the variation between samples would be largely due to the variation in the treatment rather than to field variations in soil properties, which can be substantial. Soil from each core was analyzed separately for N and C by automated combustion (Carlo-Erba combustion analyzer; CE Elantech, Lakewood, NJ). Surface soils used in this experiment contained 12.9% C (SD ± 4.3) and 0.7% N (SD ± 0.1), with a C/N ratio of 17.8 g g<sup>-1</sup> (SD ± 2.6). The pH in water was 4.93 (SD ± 0.22).

For the batch experiments, 10.00 g (±0.05) of the bulk fresh soil sample was placed in 120-mL cylindrical specimen containers. We then added 100 mL of a treatment solution to each sample. Treatment solutions were 0.01, 0.1, 1, 10, or 100 mmol L<sup>-1</sup> NaCl. For comparison, seawater is approximately 600 mmol L<sup>-1</sup> NaCl, so the highest concentration of added salts is one sixth of seawater. Chloride levels in deposition near the coast were estimated from annual volume-weighted streamwater concentrations, which range from 0.06 to 0.64 mmol L<sup>-1</sup> in stream sampling locations 0.6 to 17.6 km from the coast (Compton et al., 2003). The mid-range of concentrations used in the batch extractions was similar to the concentrations in deposition. We did not use distilled water alone in extractions because of previous experience of the first author with restricted leaching from soils treated with very low ionic strength solutions that may not be common in soil solution. Three replicates and a blank (no soil) were prepared for each treatment level. The containers were placed on a reciprocating shaker for 7 h and filtered through 0.45-µm filter paper (GFA/E; Whatman Inc., Kent, UK). Filtered solutions were stored at 4°C for less than 48 h until analysis for nitrate, ammonium, and total dissolved N by automated colorimetry (Lachat Instruments, Hach Co., Loveland, CO); chloride and phosphate by ion chromatography (Dionex Corp., Sunnyvale, CA); and DOC by UV-persulfate (Tekmar-Dohrmann; Teledyne

Technologies Inc., Thousand Oaks, CA). The laboratory detection limit for ammonium and nitrate is 0.002 mg L<sup>-1</sup>. Total dissolved N was determined by persulfate digestion; dissolved organic N (DON) was calculated as total dissolved N minus nitrate N and ammonium N. Values of the blank (no soil) were used to correct the extracted values by the levels in the salt solutions without soil.

For the leaching experiments, field moist soil, equivalent to 20 g of dry weight, was placed in a bottle-top filter microcosm (termed here microcosm), using a similar approach to Nadelhoffer et al. (1990). Three replicates were used per soil and treatment level. Microcosms were lined with glass wool and 0.45- $\mu$ m filter paper (GFA/E; Whatman). Microcosms were leached by adding 100 mL of one of four solutions: 0.01, 0.1, or 1 mmol L<sup>-1</sup> NaCl or 1 mmol L<sup>-1</sup> RbCl. Solutions were added to the microcosms and immediately extracted using a vacuum pump. We used sodium chloride because sea salt is dominated by these two ions. Rubidium has a stronger affinity for the exchanger than sodium (McBride, 1994), and therefore an equivalent molarity is more effective than sodium at displacing cations from the exchanger. Comparing the response of these two cations might provide some insight into the mechanism (ion exchange vs. other processes). This leaching process was repeated at Days 6, 13, and 27 of the experiment. Between leachings, we kept the microcosms in the dark at 27°C and maintained moisture content at original field moisture ( $\pm$ 0.1 g) by checking every few days and adding deionized water where needed. Leachates were stored at 4°C until analysis for nitrate, ammonium, and total dissolved N as described above. Statistical analyses (ANOVA followed by Tukey multiple comparisons) were conducted using SYSTAT 10.

## Results

The immediate effect of the NaCl additions was to mobilize ammonium from the exchanger. Increasing salt concentrations did not yield an immediate increase in nitrate release in the batch extractions (Fig. 1), indicating that rapid exchange of chloride for nitrate was not occurring in these soils. The highest concentrations of salts resulted in the lowest nitrate release in the short-term extractions. The lack of an immediate effect of chloride on nitrate indicates that rapid, direct exchange (abiotic) of chloride for nitrate did not occur in the batch extractions. Phosphate declined significantly with salt additions in the batch extractions.

In the month-long incubations, salt additions significantly increased nitrate and ammonium release and decreased DOC leaching (Fig. 2). Total nitrate release increased with NaCl concentration. Salt additions released more ammonium during the first week of the incubations, particularly in the RbCl treatment. There was an important ini-

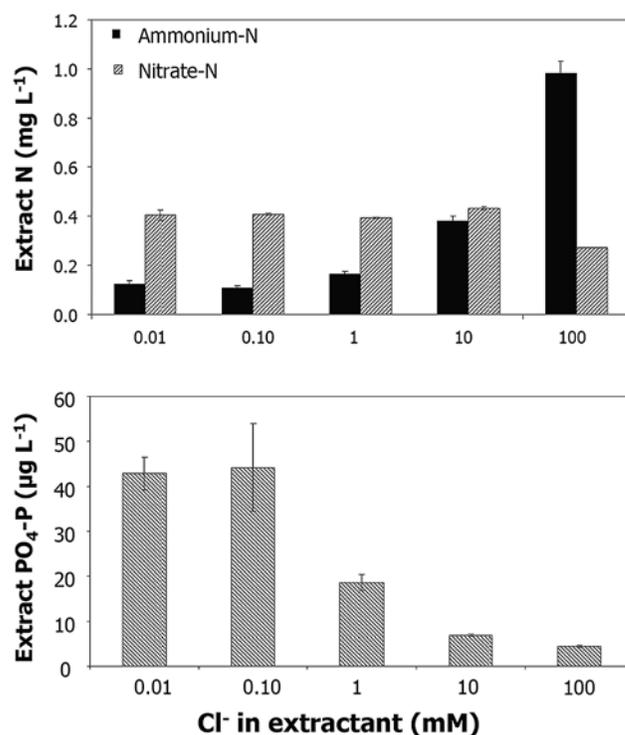


Fig. 1. Ammonium, nitrate, and phosphate concentrations released during short-term batch extractions of 0 to 15 cm soil with varying concentrations of NaCl extractant. Error bars represent SD.

tial stimulation of ammonium leaching, similar to the batch extractions, but nitrate was the dominant form of N in leachate. Phosphate and DON leaching also declined with salt additions, but the effects were not significant. The mobilization of nitrate remained elevated in the high salt treatments

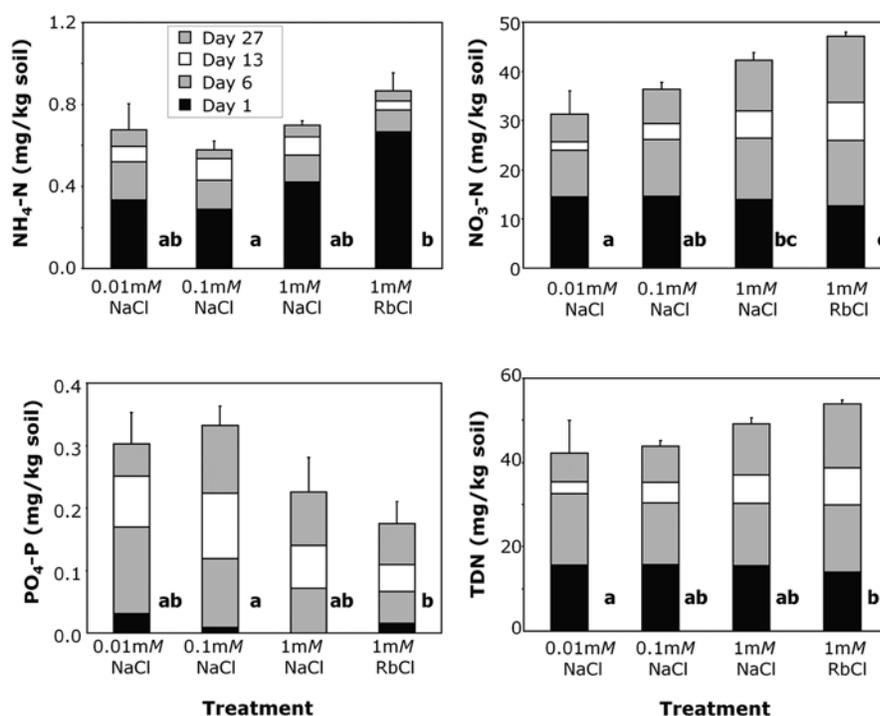


Fig. 2. Cumulative leaching of ammonium, nitrate, phosphate, and total dissolved nitrogen during month-long (27-d) incubations of 0 to 15 cm soil in microcosms. Small letters indicate significant differences in leaching between treatments ( $p < 0.05$ ).

throughout the experiment, with the greatest production occurring in the last week of the incubation, indicating that the effect of salt addition on net nitrate production is maintained beyond an initial disturbance.

Comparison of the results of RbCl vs. NaCl extractants provides some insight into the mechanism. Rubidium has a stronger affinity for the exchanger than Na (McBride, 1994). Over the month-long period, the RbCl treatment released the most ammonium and nitrate because ammonium concentrations were much higher in the 1 mmol L<sup>-1</sup> RbCl treatment than in the 1 mmol L<sup>-1</sup> NaCl treatments. Slightly more nitrate was released in the longer-term incubations leached with RbCl vs. NaCl at the same molarity. Thus, the mechanism appears to be related to the displacement of ammonium by the accompanying cation on the exchanger.

## Discussion

Our work establishes the occurrence of a salt-driven N release in a controlled setting and provides insight into the mechanism of N mobilization in soils subjected to salt inputs in the range of atmospheric deposition. We varied concentrations of NaCl for simplicity rather than using a sea salt mixture. Our assumption was that because these two ions dominate seawater, they would cause the dominant effects. Sulfate or other ions in seawater could have an impact, but we were not able to assess these impacts with our current design. In addition, we acknowledge that short-term laboratory incubations with reconstructed soil columns do not represent field conditions. Our goal was to determine whether salt additions, at levels observed in the field, could alter N mobilization in a controlled setting.

Early work by Hahn et al. (1942) found no effect of KCl on nitrification; they stated that “generalization is impossible, since this influence appears to depend on the conditions under which experiments are conducted.” We agree with this statement particularly if different studies were designed to test different drivers. Roseberg et al. (1986) and Groffman et al. (1995) added microbial substrates (ammonium or glucose) to create optimal conditions and thus are not directly comparable to ours. Both substrates could lead to the result of no impact (if ammonium is equally available across all treatments). Duckworth and Cresser (1991) found that salt stimulated ammonium release from organic soils but did not affect nitrate release, but organic soils have plentiful DOC and redox conditions to reduce nitrate. Other factors that play a role include concentrations and forms of salt, counterion, time of contact, initial soil pH, and previous exposure to Cl<sup>-</sup> (Roseberg et al., 1986; Duckworth and Cresser, 1991; Hale and Groffman, 2006).

Our results appear to be more consistent for net N mineralization. For similar soils in coastal Washington, Heilman (1975) found that, although KCl additions reduced nitrification in short-term soil incubation, net N mineralization was strongly stimulated by the addition of KCl. The increase in mineralization more than offset the reduction in nitrification (decrease of 10 mg kg<sup>-1</sup> in net nitrification vs. an increase of 50 mg N kg<sup>-1</sup> in net mineralization). Green and Cresser (2008) found that salt additions displaced ammonium and may have increased net N mineralization, consistent with our findings.

Our work provides support for the suggestion that increases in sea salts at concentrations occurring in precipitation can reduce DOC mobilization and leaching to freshwater (Evans et al., 2001; Monteith et al., 2007). Additions of NaCl strongly reduced solubilization of DOC in the rapid and month-long experiments (Fig. 3). Increasing salinity removes DOC from solution via flocculation and sorption to mineral surfaces (Fox, 1991; Hedges and Keil, 1999). In columns of forest soils, increasing ionic strength reduced DOC leaching (Evans et al., 1988). Andisols, such as those in the Oregon Coast Range, have high sorption capacity associated with amorphous iron and aluminum hydroxides and aluminum-humus complexes, but electrolytes alter the net surface charge of these variable-charge minerals. At pH <7 (soil pH is 4.93 at this site), increasing salt strength also generates more positive surface charge (McBride, 1994) and generally decreases solution pH (Green et al., 2009), which would increase the sorption capacity of mineral surfaces for carboxyl groups of DOM. Salt additions alter the characteristics of the soil exchanger to increase anion sorption and thus reduce DOC in solution. A history of chronic salt additions may influence the nature of the relationship between salt additions and DOC (Green et al., 2009), and the fact that our soils showed a strong response to salt additions may be because we dramatically increased the salt levels. Chronic salt additions at higher levels may have less impact on DOC concentrations, as seen by Green et al. (2009).

Phosphate declined significantly with salt levels in the batch extraction and declined substantially but not significantly in the month-long extractions. This finding, along with the decrease in DOC, indicates that sorption capacity increases with ionic strength of the leaching solution. Changes in phosphate delivery are important for aquatic productivity, and this potential effect on phosphate availability should be examined further.

The effect of salts on the availability of C substrates also has important implications for N cycling. Nitrate dynamics in particular are influenced by the availability of C substrates (Hart et al., 1994); increases in bioavailable DOC have been shown to reduce nitrate along soil flowpaths (Sobczak et al., 2003). Groffman et al. (1995) suggest that glucose additions relieve chloride inhibition of microbial respiration, which indicates that the availability of DOC may be a key factor in the microbial response to salt addition. A reduction in DOC may cause a decrease in microbial immobilization of nitrate, which, combined with the large release in ammonium (Fig. 1), would yield higher net nitrification. Stark and Hart (1997) found high rates of gross N cycling rates for similar soils. Because gross rates of N cycling are often several times higher than net rates (Stark and Hart, 1997; Booth et al., 2005), a slight imbalance in gross immobilization and mineralization/nitrification rates can lead to marked changes in net mineralization and nitrification. Thus, the salt-driven reduction in DOC release here could play this role in initiating this imbalance by removing a C substrate and thus reducing immobilization. The influence on denitrification could be an important factor at larger scales. Hale and Groffman (2006) recently demonstrated that chloride can reduce potential denitrification and increase the net release of inorganic N in stream debris dams.

Interactions between C and N are critically important, given the evidence for the control of stream nitrate dynamics by DOC (Bernhardt and Likens, 2002; Goodale et al., 2005). Although not statistically significant, phosphate concentrations were substantially lower with increasing salt additions in batch extractions. A shift in the relative abundance of C, N, and P has important consequences for nutrient status of terrestrial, freshwater, and coastal aquatic ecosystems (Vitousek and Howarth, 1991; Carpenter et al., 1998).

### Summary and Implications for Coastal Watershed Biogeochemistry

Salt additions mimicking coastal salt spray increased the release of ammonium and enhanced leaching of nitrate in a coastal Oregon Andisol. Salts also reduced the mobilization of DOC. The mechanism for the salt-driven increases in nitrate leaching in our incubations appears to be a combination of abiotic and biotic processes: The increase in nitrate leaching results from displacement of ammonium from the exchanger followed by increased net nitrification and nitrate release. Sea salts can be deposited to land during storms through entrainment in heavy rains and by direct flooding with seawater surges. Increases in storm frequency that might be associated with global climate change (e.g., Hayden and Hayden, 2003) could increase the frequency and severity of sea salt episodes in coastal regions (Wright and Schindler, 1995). Interactions between sea salts and human alterations of the N cycle (e.g., fertilization or N deposition) could lead to unexpected effects on receiving waters such as N-limited estuaries and the coastal ocean. There is a clear need for a better understanding of such element interactions to predict the response to human activities and changing climate.

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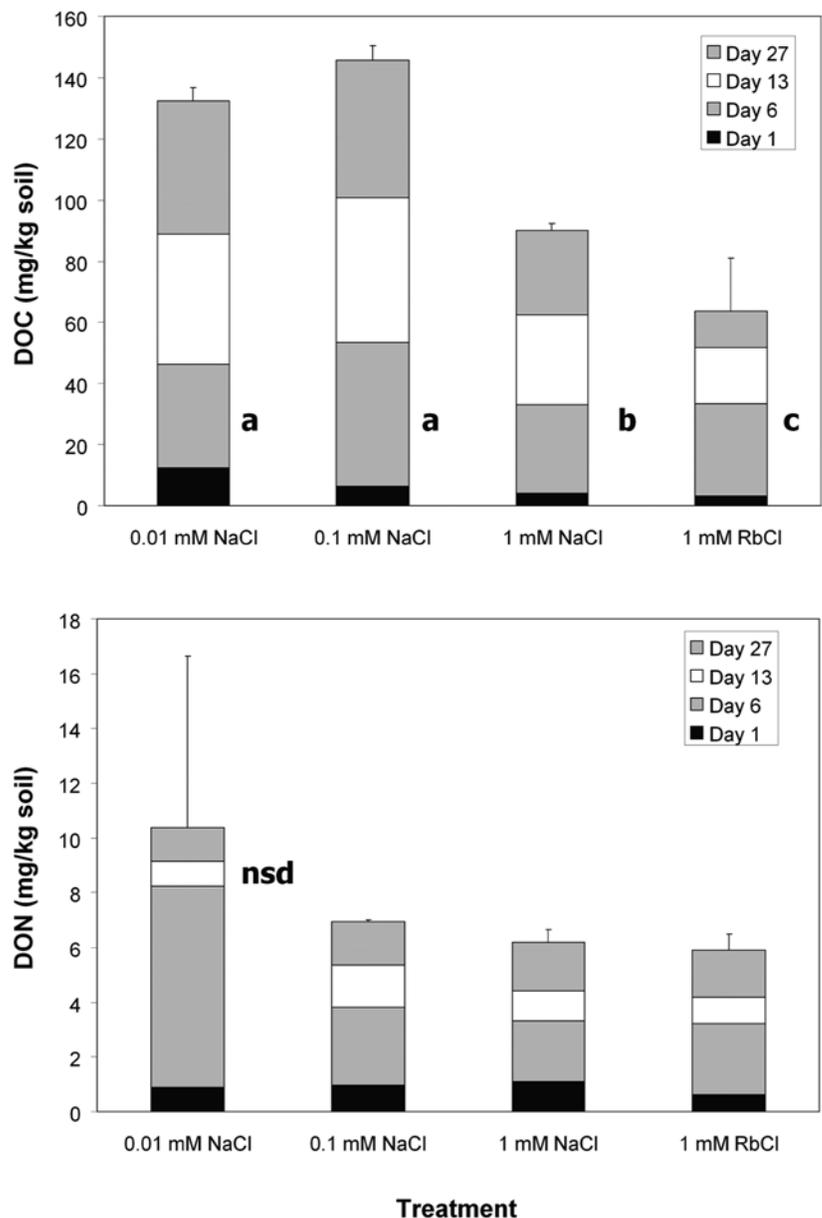


Fig. 3. Cumulative leaching of dissolved organic carbon (DOC) and dissolved organic nitrogen (DON) from the microcosms over the 27-d incubation. Small letters indicate significant differences in leaching between treatments at 27 d ( $p < 0.05$ ). nsd, no significant differences.

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