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TITLE

Effects of Oil Field Brine Wastewater on Saturated Hydraulic Conductivity of Smectitic Loam
Soils

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ABSTRACT

Spills of brine wastewater produced during oil well drilling are occurring more frequently in the Great Plains, resulting in crop production loss on affected soil. Remediation requires removal of salt from the topsoil, which might be accomplished by leaching to subsurface horizons or subsurface drains. A laboratory study determined the effects of brine on saturated hydraulic conductivity (K_s) of four non-impacted surface soils from western North Dakota, USA. Repacked soil cores were subjected to saturated water flow, followed by one pore volume of brine. Subsequent saturated water flow leached brine from the soil and reduced K_s as much as 97% (0.086 to 0.003 cm h⁻¹) within 24 h. Effluent total dissolved solids (TDS) approached 250 000 mg L⁻¹ then declined (5 mg L⁻¹) with continued leaching, but K_s did not increase. Removal of soluble salts during leaching increased the relative sodium concentrations (ESP>55), causing clay swelling/dispersion and reduced K_s . Post-brine gypsum application (11.2 Mg ha⁻¹) to replace exchangeable sodium with calcium did not improve K_s . This evidence suggests that if subsurface drainage is used for reclaiming brine-impacted soils that special attention be given to where dispersion/swelling is occurring, leaching water quality, and closely positioning calcium amendments within the high sodium zones.

Keywords: Brine spill remediation, soluble salts, exchangeable sodium, hydraulic conductivity, clay dispersion/swelling.

INTRODUCTION

Oil production in North Dakota has increased from 100 000 barrels per day to over 1 000 000 barrels per day in less than a decade (ND Dept. of Mineral Resources 2014) due to the use of horizontal boring and hydraulic fracking technologies enabling extraction of oil and gas from the Bakken and Three Forks Formations in Montana, North Dakota, South Dakota, and Canada (USGS 2013). Along with the oil and gas, production water, also referred to as 'brine', is also produced. In 2007 in North Dakota, 3 barrels of brine per barrel of oil and 18 barrels of brine per million cubic feet of gas was produced (Clark and Vei 2009). This highly saline (predominately NaCl) produced water is allowed to separate from the oil and is stored in above-ground tanks, and then injected into salt-water disposal wells. During storage and pumping of brine to injection wells, many spills of varying severity have been reported. In 2014 in North Dakota alone, more than 432 million barrels of brine was produced (increased from <100 million in 2001), of which 71 000 barrels was spilled in 855 individual reported spills. From 2001-2014, more brine has been spilled each year than oil (ND Dept. of Health 2015; Daigh and Klaustermeier 2016).

Large brine spills, like the nearly 3 million gallons of brine that leaked from a pipeline into two creeks near Williston, North Dakota in 2015 (Jacobson 2015), attract much of the public attention. However, in addition to waterway contamination, brine usually first affects the soil where the spill occurs. The increased salt in the soil from the brine initially affects plant growth by preventing osmotic uptake of water by the plant roots or toxicity which kills the plant. This can result in complete soil sterilization for years or even decades (Murphy et al. 1988). Deterioration of soil physical properties is also affected by brine contamination. DeJong

(1982) showed dispersion and swelling of soil cores in a laboratory setting after NaCl contamination. Subsequent leaching with gypsum-saturated water was only marginally effective at restoring percolation in their study.

Current remediation techniques include either removing the soil material and replacing it with uncontaminated soil or leaching the salts out of the rooting zone. Leaching has been aided by additions of organic matter, sources of calcium, and subsurface drainage (Harris et al. 2005; Sublette et al. 2005; Carter and Fanning 1964). However, after leaching the salts, there is danger of salinization of the subsoil or, if the water table is shallow, re-salinization of the topsoil due to capillary rise. Hence, it may also be necessary to provide a means of water removal such as subsurface drainage. Remediation by incorporation of calcium and/or organic matter and leaching with excess water has been somewhat successful in reducing the salinity. However, if the salinity in the topsoil is sufficiently reduced by leaching, there may be further danger of soil physical property damage due to dispersion and/or swelling caused by excess sodium on the exchange complex (He et al. 2015; Schofield and Taylor 1961). In the current study, we use saturated hydraulic conductivity as an indication of the deterioration of soil structure from dispersion and swelling caused by excess sodium.

Given the rapid rate of oil development in North Dakota and the region, and the increasing number of spills recorded each year, there is some urgency to disseminate as much information as possible on the effects of oil-field brine spills so that informed decisions can be made regarding remediation of these contaminated soils. The objective of this study was to determine the effect of oil-field brine on saturated hydraulic conductivity (K_s) of a smectitic loam soil during and after contamination, and the implications for remediation.

MATERIALS AND METHODS

Four bulk soils used for this study were collected with a shovel to a depth of 30 cm from two non-contaminated locations near Mohall, North Dakota, USA. Near both locations there are long-term brine-contaminated soils. Two soils, designated B_U and B_L, were described as Barnes loam (fine-loamy, mixed, superactive, frigid Calcic Hapludoll), and were taken from upper and lower landscape positions, respectively. Soil T_U was from an upland position and was described as Tonka silt loam (fine-smectitic, frigid Argiaquic Argialboll) in the Barnes-Svea-Tonka complex. Soil S_L was Svea loam (fine-loamy, mixed, superactive, frigid Pachic Hapludoll) in the Barnes-Svea complex and was from a lower landscape position. Even though the B_U and B_L were mapped (Order 2 survey) as the same soil type, the different landscape position indicated that the two soils may be different enough to warrant keeping them separate. The soil was air-dried, crushed, and sieved through a 2-mm screen. Particle size was determined using the hydrometer method (Gee and Bauder 1979). The pH and electrical conductivity ($EC_{1:1}$) was determined on a 1:1 soil:water extract. The cation exchange capacity (CEC) was determined using the sodium acetate –ammonium acetate extraction method and analysis of sodium was done by atomic absorption (AA) spectroscopy (Soil Survey Staff 2011). The initial pH, $EC_{1:1}$ and CEC of each soil and the average bulk density and pore volumes (based on weight of saturated cores) for all of the repacked cores are listed in Table 1. No field bulk density samples were taken, hence, no attempt to replicate a specific field bulk density was made. However, Web Soil Survey (Soil Survey Staff) lists the bulk density of the Barnes-complex soils as 1.35 g cm^{-3} .

The oil-field brine used for the study was collected from a pipe supplying an injection well in the vicinity of the soil locations. Chemical analysis of the brine was conducted by Midwest Laboratories, Inc. (Omaha, NE) using EPA standard methods and indicated a conductivity of 226 dS m^{-1} with the major ions being chloride ($182\,681 \text{ mg L}^{-1}$) and sodium ($95\,738 \text{ mg L}^{-1}$). Other analyte concentrations (mg L^{-1}) included calcium ($5\,824$), magnesium ($1\,454$), sulfate ($1\,216$), phosphorus (8.5), potassium ($3\,575$), bicarbonate (238), and boron (170). The total dissolved solids (TDS) was measured by the authors by evaporation to be $308\,820 \text{ mg L}^{-1}$. The brine was mostly free of petroleum products, however, a slight oily residue was present but removed prior to starting the experiments.

Tempe cells were assembled using the modification described by Sommerfeldt et al. (1984), which included rigid stainless steel screens and filter paper discs on top and bottom to restrict swelling and retain soil particles during K_s determination. The 30-mm tall x 54-mm diameter brass rings of the Tempe cells were packed with the dried, sieved soil by incrementally adding 10-mm soil depths and tapping the sides of the assembly with a wooden dowel to aid in packing. Between each addition of soil to the ring, the previous packed layer was partially disturbed on its surface to prevent formation of a defined boundary between the layers. The assembled Tempe cells were weighed before and after soil packing to allow determination of bulk density (Table 1).

A manifold of plastic tubing and shutoff valves was used to allow K_s determination on 10 samples simultaneously. The cores were saturated from the bottom using a Marriott bottle reservoir of degassed distilled water. After saturation of the soil, the water flow was stopped and the outlets on the bottom of the Tempe cells were temporarily plugged. The saturated cells

were weighed and the water-filled pore volume was calculated (Table 1). While the manifold was switched to the tops (inlets) of the cells, care was taken to eliminate all air bubbles in the water supply lines and Tempe cells. The water level of the reservoir was set to 200 mm above the base of the soil and water flow was started. Outflow was collected and measured at known time intervals until steady state flow was observed and K_s calculated. After reaching steady state flow, the water reservoir was replaced with the brine and one pore volume was applied (measured outflow volume). After brine application, water was again applied to the cells until relatively constant flow was observed. Three replications were performed in this manner. On separate replications of cores, the Tempe cells were opened after brine application and 2.55 g of powdered gypsum was incorporated into the top 5 mm (a rate of 11.2 Mg ha^{-1}) and the cores were again leached with water. Each soil was replicated three times for the non-gypsum treatments and two times for the gypsum treatments. Additionally, three replications of each soil were leached with water only to provide a non-brine check. Leachate quantity and quality samples were taken at the successive stages during the experiment; pre-brine application (PRE), during the brine application (BR), multiple times post-brine application (POST1, POST2, ..., POSTn), and if applicable, multiple times after gypsum application (GYP1, GYP2, ..., GYPn).

A subsample of each leachate sample was measured into an accurately weighed beaker and dried at 60°C . The resultant evaporite solid was weighed and the concentration of TDS was calculated. At the conclusion of leaching, the Tempe cells were dismantled and the soil was removed and oven dried. The exchangeable sodium percentage (ESP) was determined by first washing 5 g of soil with 3 successive washes with 33 ml of 60% ethanol (shaken, centrifuged,

decanted) to remove excess salt, then extracting the exchangeable sodium with ammonium acetate with subsequent atomic adsorption flame spectrophotometry analysis (Doll et al. 1989).

Statistical Analysis

Statistical analysis was performed using JMP (JMP, Version 10). Analysis of variance (ANOVA) and Tukey's t-test were used to determine means and standard deviations and to determine significant K_s and TDS differences between soils at a given stage and between stages for a given soil. Although the Goodness of Fit method in JMP indicated that the K_s values were not normally distributed, which is the assumption made when using a t-test, the differences discussed below were highly significant (p-values <0.0001) and should nevertheless be an indication of differences between mean values.

RESULTS

Brine saturation

Figure 1 shows the average K_s and TDS data for the four soils leached with one pore volume of brine with no subsequent gypsum application. Time series charts of K_s (Fig. 1a) show the changes in K_s relative to elapsed time. In Fig. 1b, the data was also segregated into general leaching stages; pre-brine application (PRE); during brine application (BR); and multiple post-brine application stages (POST1, POST2) to present significant differences between and within leaching stage and soil. During the initial water-leaching phase (PRE), the average K_s (Fig. 1b) was between 0.06 and 0.08 cm h⁻¹ for T_U, T_L, and B_L soils and 0.28 cm h⁻¹ for B_U, indicating adequate water flow through all of the soils. These values are similar to the values from the non-brine check cores, which were leached with water only (Fig. 2a). During the application of 1

pore volume of brine (BR), the K_s was not significantly different ($\alpha=0.05$) from PRE K_s . However, once distilled water was again introduced to the system (POST) and leaching was continued for an average of 15 h (Fig. 1a), the K_s dropped to 0.009 cm h^{-1} for S_L and 0.006 cm h^{-1} for B_U, a 97% reduction. After approximately another 27 h, the K_s was 0.002 cm h^{-1} for all soils. There was a very strong significant difference ($p\text{-value}<0.0001$) between the PRE K_s and the POST1- and POST2-stage K_s , which took <18 h after the brine source was removed to fully manifest for all soils. No significant differences in K_s between soils for any given leaching stage were measured except B_U for the PRE and BR stages (Fig. 1b).

The salinity of the leachate, as indicated by the TDS concentration for the same leaching events is shown in Figs. 1c and 1d. During the PRE stage, the TDS was quite low ($<4 \text{ mg L}^{-1}$ for all soils), indicating very little soluble salt was present in the soil to be leached out. Similar TDS values were measured from the non-brine checks (Fig 2b). The effluent TDS concentrations started to increase only slightly (not significant at $\alpha=0.05$) after addition of one pore volume of brine. This indicated that the brine had filled most of the previously water filled pore space and that no preferential flow was occurring. The TDS concentration of the leachate spiked between 225 000 and 250 000 mg L^{-1} on the POST1 sample collection, as the infiltrating water leached the salt from the soil core. As leaching continued during the POST stages, the TDS started to decline as more salts were leached out with the distilled water. The TDS for the PRE and BR stages were not significantly different for any given soil (Fig. 1d), but the TDS concentration for the POST1 stage was significantly higher than the PRE- and BR-stage TDS ($p\text{-value}<0.0001$). The POST2-stage TDS was also significantly lower than the POST1 TDS concentration. There was no significant difference in TDS between soils for any given leaching stage.

Brine saturation with gypsum application

Time series K_s and TDS data for the soils leached with 1 pore volume brine and then treated with gypsum are presented in Fig. 3. Leaching was continued for a longer time (Fig. 3a) compared to the non-gypsum treatments in order to see if the gypsum would have any remedial effect on K_s . Initial PRE stage K_s values (Fig. 3b) ranged between 0.04 and 0.08 cm h^{-1} for all soils. These values were slightly different but in the same range as all soils except B_U during the non-gypsum reps. The initial K_s for B_U are lower than for the non-gypsum reps of the study in response to the cores being packed to a slightly higher bulk density (1.31 ± 0.006 vs. 1.42 ± 0.0 , respectively). Similar to the non-gypsum treated cores, PRE and BR K_s were not significantly different within each soil and immediately started to decrease in the POST1 stage of leaching. At about 90 h, the flow of water was stopped and the Tempe cells were opened to allow incorporation of 2.5 g of powdered gypsum (equivalent to 11.2 Mg ha^{-1}) into the upper 5 mm of the soil. When leaching with water continued after gypsum application for multiple post-gypsum leachate measurement stages (GYP1, GYP2,..., GYP6) the K_s remained low, about 0.002 cm h^{-1} , until the end of the experiment at 450 h.

The leachate TDS concentrations were $< 2 \text{ mg L}^{-1}$ for all soils for the PRE stage (Figure 2d). After brine application the TDS increased slightly but was only significantly higher for S_L. Then, in the POST1 stage, only 2-4 h after the brine source was removed and water flow was started (Fig. 2c), the TDS peaked at $> 240\,000 \text{ mg L}^{-1}$ for all soils. The TDS then started to decline as more water was leached through the soils, removing much of the salt during the POST stages. After gypsum application the TDS from all soils continued to decline from $< 30 \text{ mg L}^{-1}$ to

about 5 mg L^{-1} 360 h. However, only S_L and B_U were significantly different between the GYP1 and GYP6 stages.

Exchangeable sodium

After leaching with water, the soil cores without applied gypsum were analyzed for sodium and ESP was determined (Table 2). For all soils, the ESP was significantly higher in the cores which had been saturated with brine versus those which received no brine.

DISCUSSION

The K_s was not significantly affected while the brine was being leached through the soil cores. Even after three pore volumes of brine was leached through the cores, no significant change in K_s was observed during the actual brine application (data not shown). This was due to the high salinity maintaining clay flocculation and aggregate stability during brine leaching (Pearson 2003). However, K_s was reduced to about 0.004 cm h^{-1} in <20h after leaching with distilled water.

For the non-gypsum cores, the POST1 leachate samples were taken between 12-18 h after the brine source was removed. As the leachate TDS increased to $>225\,000 \text{ mg L}^{-1}$, K_s had already decreased to $0.01\text{-}0.006 \text{ cm h}^{-1}$. Similarly, for the gypsum cores, approximately 18 h after the brine source was removed, water application caused the K_s to decrease between 87-97%, depending on the soil. After 42 h, K_s had decreased another order of magnitude, to approximately $0.001\text{-}0.002 \text{ cm h}^{-1}$, and remained steady throughout the remainder of the study, even though the TDS concentration of the leachate indicated that most of the salt had been removed from the soil. The fact that K_s was not negatively affected during the brine saturation phase, but only after distilled water was applied, indicated that the high salinity

maintained the soil structure. However, the clay and organic matter exchange sites had become saturated with sodium (high ESP), which caused dispersion and swelling to occur after the salinity had been reduced (as indicated by the decrease in leachate TDS). Others have reported clay dispersion and reduced infiltration/ K_s when soil with high levels of sodium is leached with low EC waters (Abu-Sharar et al. 1987; Agassi et al. 1981; Shainberg et al. 1981; Suarez et al. 2008). In addition, soil swelling will also likely reduce water flow (Curtin 1994; He et al. 2015).

Since the project was not continued until K_s started to recover, it is unknown how long, if ever, it would take to remediate the dispersed soil. Others have indicated as many as 30 pore volumes was necessary to replace and leach sodium with calcium using a saturated gypsum solution (Jury and Weeks 1978). What is clear, however, is that once distilled water was applied to the soil surface after removal of the brine source, soil dispersion and/or swelling rapidly hindered water movement. Subsequent application of gypsum to the top 5 mm did not result in restoration of desirable water movement through the affected soil. However, it is unknown if a higher rate of gypsum would have been successful in replacing sodium and restoring the permeability of the soil but this is not likely mainly due to the low EC that results from a saturated solution of gypsum (2.2 dS m^{-1}). Although others have calculated a gypsum requirement in excess of 50 t ha^{-1} for brine contaminated soils (Ashworth et al. 1999), to limit disturbance of the soil in the Tempe cells and to be able to reassemble the Tempe cells after gypsum application, a lower rate was necessary for the current study.

Based on our results, one can hypothesize about the course of events in a field brine spill scenario. During a release of brine from a tank or pipe, the salinity of the affected soil would be very high which would maintain clay flocculation as brine continues to leach into the

soil and subsoil, thus allowing for deep percolation as long as there is a head of pressure. It also may allow a leak to remain undetected for longer periods of time since there would be less potential for surface ponding of the brine. After the pressure head of brine is removed, rainfall, irrigation, or flooding with low salinity water would likely leach some salts from the surface soil to lower soil depths before infiltration was reduced. With the reduced salinity of the soil solution and the presence of excess sodium remaining on the exchange sites, the smectitic clays would swell and dispersed clay particles would plug soil pores (Abu-Sharar et al. 1987; Curtin et al. 1994; Frenkel et al. 1978) and the surface soil would effectively be sealed. Subsequent remediation attempts using surface applied gypsum and excess water applications may be futile at that point, as the soil structure will have already been deteriorated and water movement, which is necessary to transport any sodium that may be replaced from the exchange complex with calcium, will have been drastically reduced. Once the topsoil became impermeable, no further leaching of salts from the subsoil could occur. Given the resultant degraded water movement, subsurface tile drainage would not be effective in removing additional salinity. Gypsum must be applied before the soil structure has deteriorated due to dispersion, while the saline water is still in the soil (Schofield and Taylor 1961). Of course, many variables are present in nature which could influence the process, ranging from heterogeneous flow influenced by frozen soils (Schotanus et al. 2012) and undisturbed soils to differences in clay mineralogy. While all scenarios cannot be discussed, the vast extent of Barnes-complex soils (>2.1 million ha in ND) and the fact that across eco-regions, North Dakota soils are dominantly smectitic and superactive ($\text{CEC/clay} > 0.6$), suggests that soils across much of the state would be impacted similarly after a brine spill.

CONCLUSIONS

Soil cores were packed with Barnes-complex soils from western North Dakota and subjected to oil-field brine application. Infiltrating brine did not affect hydraulic conductivity of the soil, but subsequent water application caused swelling and dispersion, which caused a drastic reduction in K_s as salinity was reduced but ESP remained high. Surface application of 11 Mg ha^{-1} of gypsum after brine application had no effect on K_s or leachate TDS over the course of the study. Given the current extent of and potential for future oil-field development in North Dakota, South Dakota, Montana, and Canada, brine spills are likely to continue to impact highly productive soils such as the Barnes series used in the current study. Environmental professionals and landowners should be educated about the impacts of brine spills and remediation challenges. Based on this work, spill remediation attempts using surface applied gypsum, flooding with low to moderate salinity water, and leaching to subsurface drains must occur prior to the reduction of EC by natural or anthropogenic events. While this project demonstrated degradation of soil structure when water was applied directly after brine, many variables may be present under actual field conditions that were not addressed with this particular study. Future brine spill-related research should include laboratory and field scale experiments to investigate different amendments, unsaturated flow, undisturbed soil, and frozen soils.

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Table 1. Initial soil and repacked soil core properties. The pore volume, calculated based on particle density=2.65 g cm⁻³ and bulk density=1.40 g cm⁻³, was 32.4 cm³. n=2 for texture, pH, EC, and CEC. n=5 for core bulk density and pore volume. l, loam; sl, sandy loam.

Soil	Sand	Silt	Clay	Lab texture	pH _{1:1}	EC _{1:1}	CEC	Core bulk density	Core pore volume
	%	%	%			dS m ⁻¹	cmol[c] kg ⁻¹	g cm ⁻³	cm ³
T_U	41	40	19	l	7.46	0.53	11.22	1.41 ±0.04	29.02 ±0.23
S_L	38	43	19	l	6.78	0.69	12.58	1.39 ±0.04	29.68 ±1.08
B_U	42	41	17	l	7.50	0.50	9.42	1.36 ±0.06	29.50 ±0.90
B_L	53	37	9	sl	7.38	0.84	10.32	1.36 ±0.01	30.56 ±2.15

± values are standard deviation.

Table 2. Exchangeable sodium percentage of soils after leaching experiments.

Soil	Brine applied pore volumes	Exchangeable sodium %
T_U	0	0.9
	1	62.8
S_L	0	1.0
	1	59.8
B_U	0	1.0
	1	56.6
B_L	0	1.0
	1	55.5

FIGURE CAPTIONS

Figure 1. Time series charts of average a) saturated hydraulic conductivity (K_s) and c) total dissolved solid (TDS) concentration of the leachate for the four soils (T_U, Tonka upland; S_L, Svea lowland; B_U, Barnes upland; B_L, Barnes lowland) leached with one pore volume brine and receiving no gypsum. Vertical lines through data points are 95% confidence intervals. Charts by leaching stage (PRE, pre-brine water application; BR, during brine application; POST, post-brine water application) of average b) saturated hydraulic conductivity (K_s) and d) total dissolved solid (TDS) concentration of the leachate for the four soils leached with one pore volume brine and receiving no gypsum. Means with different lower case letters are significantly different between leaching stage for a given soil. Means with different upper case letters are significantly different between soils for a given leaching stage. Corresponding leaching stage is also indicated across top of time series chart.

Figure 2. Time series charts of average a) saturated hydraulic conductivity (K_s) and b) total dissolved solid (TDS) concentration of the leachate for the four soils (T_U, Tonka upland; S_L, Svea lowland; B_U, Barnes upland; B_L, Barnes lowland) leached with water only. Vertical lines through data points are 95% confidence intervals.

Figure 3. Time series charts of average a) saturated hydraulic conductivity (K_s) and c) total dissolved solid (TDS) concentration of the leachate for the four soils (T_U, Tonka upland; S_L, Svea lowland; B_U, Barnes upland; B_L, Barnes lowland) leached with one pore volume brine and receiving 11.2 Mg ha⁻¹ gypsum. Vertical lines through data points are 95% confidence

intervals. Charts by leaching stage (PRE, pre-brine water application; BR, during brine application; POST, post-brine water application) of average b) saturated hydraulic conductivity (K_s) and d) total dissolved solid (TDS) concentration of the leachate for the four soils leached with one pore volume brine and receiving 11.2 Mg ha^{-1} gypsum. Means with different lower case letters are significantly different between leaching stage for a given soil. Means with different upper case letters are significantly different between soils for a given leaching stage. Corresponding leaching stage is also indicated across top of time series chart.