



## Short Communication

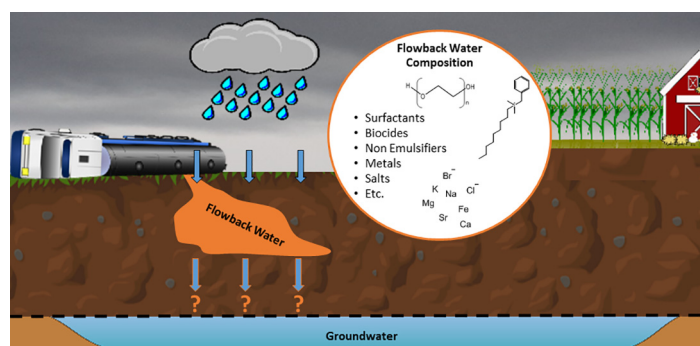
## Simulation of a hydraulic fracturing wastewater surface spill on agricultural soil

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## HIGHLIGHTS

- A spill of HFWW was simulated in bench scale soil columns.
- No surfactants were found in leachate samples.
- Transport of metals was caused by the high concentrations of salts.
- A significant decrease in the infiltration rate of the soil was observed.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Hydraulic fracturing wastewaters (HFWWs) contain synthetic organic components and metal ions derived from the formation waters. The risk of spills of HFWW that could impact soil quality and water resources is of great concern. The ability of synthetic components, such as surfactants, in HFWW to be transported through soil and to mobilize metals in soil was examined using column experiments. A spill of HFWW was simulated in bench scale soil column experiments that used an agricultural soil and simulated seven 10-year rain events representing a total of one year's worth of precipitation for Weld County, Colorado. Although no surfactants or their transformation products were found in leachate samples, copper, lead, and iron were mobilized at environmentally relevant concentrations. In general, after the initial spill event, metal concentrations increased until the fourth rain event before decreasing. Results from this study suggest that transport of metals was caused by the high concentrations of salts present in HFWW. This is the first study utilizing authentic HFWWs to investigate the transport of surfactants and their effect on metal mobilization. Importantly, a significant decrease in the water infiltration rate of the soil was observed, leading to the point where water was unable to percolate through due to increasing salinity, potentially having a severe impact on crop production.

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## 1. Introduction

The chemistry of hydraulic fracturing wastewaters (HFWWs) is highly variable and is driven by the formation waters and the synthetic

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chemical additives (fracturing fluid) used to stimulate oil and gas extraction (Abualfaraj et al., 2014; Elsner and Hoelzer, 2016; Hayes, 2009; Khan et al., 2016; Oetjen et al., 2018, 2017; Thacker et al., 2015). Debate has arisen over what the potential risks to water resources associated with accidental releases or spills of HFWW are (Taylor et al., 2011; Vengosh et al., 2014; Vidic et al., 2013). The majority of spills occur during the waste management and disposal phase or during transportation and storage (Kell, 2011). Though national data are lacking, from 2015 to 2016, the Colorado Oil and Gas Conservation Commission (COGCC) reported 543 spills of flowback or produced water (Colorado Oil and Gas Conservation Commission, 2017). The majority of these spills occurred within Weld County, Colorado (Colorado Oil and Gas Conservation Commission, 2017).

Previous studies have suggested that the synthetic chemical portion of HFWW may increase mobility of other dissolved hydraulic fracturing (HF) additives, as well as geogenic metals (Getzinger et al., 2015; McLaughlin et al., 2016). Surfactants may also increase colloid mobility by making the mineral surfaces more hydrophilic or increasing unsaturated water flow and facilitating solute transport (Sang et al., 2014). These types of co-contaminant effects, in regards to HFWW, may have important environmental ramifications in the event of a spill (Chen et al., 2017; McLaughlin et al., 2016; Rogers et al., 2017; Sang et al., 2014). However, the studies that have investigated the environmental fate and transport of HFWW rely on laboratory-prepared solutions that contain only a fraction of the contaminants present in HFWW (Chen et al., 2017; McLaughlin et al., 2016; Rogers et al., 2017; Sang et al., 2014). No studies have used actual HFWWs, which are considerably more complex and have different physical-chemical properties. This complexity is an important consideration when evaluating or modeling contaminant transport as a result of (simulated) HFWW spills. Understanding the fate and transport of HFWW in the event of a spill will be crucial when considering emergency response strategies, remediation methods, and the effects a spill may have on groundwater and soil quality.

This study simulated a spill of HFWW on an agricultural soil under environmentally relevant conditions using bench-scale columns. HFWW used in the spill event was collected from a hydraulically fractured well in Greeley, Colorado located within the Denver-Julesburg basin. The soil used in the columns was collected from an adjacent agricultural field and represents most abundant soil order in the United States (Agriculture, 1999). Rain events were then simulated to promote leaching, and leachates were analyzed for organic and inorganic constituents. The objectives of this study were to (1) determine the leachability of hydraulic fracturing fluid additives in soil, and (2) elucidate the potential for surfactant-enhanced metal leachability.

## 2. Methods

### 2.1. Soil column study design

Design for the columns was based on similar unsaturated soil-column based spill studies assessing stormwater runoff (Payne et al., 2014) and the “Demonstration of promising technologies to address emerging pollutants in water and waste water” (DEMEAU) guidelines to accurately simulate large rainfall events (Oriol et al., 2012). Three spill and three control (15.2 cm × 5.1 cm) PTFE columns were dry-packed with agricultural soil collected from a drainage ditch in an adjacent field near a stream (SI, Fig. S1). The depth of soil in the columns represented the minimum depth to the water table for the study site (United States Department of Agriculture, 2017). The soil was classified as a mollisol (aquolls) and specific soil characteristics can be found in SI Table S1. Mollisols are the most abundant soil order in the United States making up about 21.5% of the surface area (Agriculture, 1999). The soil was sieved to 0.1 cm to reduce preferential flow paths by having a column diameter-soil grain diameter ratio >40 and a representative elementary volume (REV) between 40 and 100, as required for chemical

transport experiments (Oriol et al., 2012). A single surface spill event normalized to the column areas representing a 95 percentile spill in Colorado (79.8 mL or 0.52 pore volumes) was simulated using HFWW that consisted of the first day of flowback water from a well located in Greeley, Colorado (Colorado Oil and Gas Conservation Commission, 2017). HFWWs were poured on the top of columns and flowed through columns based on gravity alone. Concentrations of known analytes in the original HFWW can be found in SI Table S2 and Oetjen et al. (2018). In control columns, the spill was comprised of an equal volume of deionized (DI) water. In a manner similar to previous studies (Ulrich et al., 2017), the infiltration of a 10-year, one hour storm rainstorm (77 mm of precipitation) for Greeley, Colorado (140 mL per rain event) was simulated via ponding once per week for seven weeks, equating to a year’s worth of precipitation for the area, and flowed through columns based on gravity alone (NOAA, 2015). Columns were allowed to drain completely between rain events. The rainwater used was collected during a precipitation event near Denver, Colorado and stored at 4 °C throughout the length of the experiment. The concentration of measured constituents in the rainwater can be found in SI Table S3. Leachate drained into amber bottles (cleaned by combustion at 400 °C) that were held in insulated sampling coolers filled with ice. After drainage was complete, samples were stored a 4 °C until chemical analysis.

A follow-up spill experiment simulating only the electrolyte portion of the HFWW was also performed under the same conditions described above. Here, a solution of sodium chloride matching the ionic strength of the HFWW was spilled onto three replicate columns to confirm the conclusions drawn from the original experiment.

### 2.2. Water quality analysis

Leachate samples were filtered through a 0.45 µm polyethersulfone membrane filter (VWR International, Radnor, Pennsylvania) and subsamples were analyzed for anions using ion chromatography (IC; ICS-900, Dionex, Sunnyvale, California) and dissolved organic carbon (DOC) concentration using a carbon analyzer (Shimadzu TOC-L, Columbia, Maryland). These filtered samples were then acidified with 200 µL nitric acid. A partial soil digestion of readily available elements was performed through an acid digestion of an aliquot of the soil (not used in the columns) following EPA method 3052 (SI Table S4). Partial soil digests and filtered water samples were then analyzed using inductively coupled plasma optical emission spectroscopy (ICP-OES; Optima 5300 DV, PerkinElmer, Fremont, California). QA included the use of Sc as an internal standard, and analysis of several certified QC standards, run after every 15–20 samples. Ferrous iron measurements were performed using Hach Method 8146.

### 2.3. High resolution mass spectrometry analysis

Leachate samples were extracted using a salt assisted liquid-liquid extraction as outlined in Oetjen et al., 2018. The extracts were then analyzed using liquid chromatography quadrupole time-of-flight mass spectrometry (LC-QToF-MS) following the approach described in Oetjen et al., 2018. Samples were screened using a library consisting of five polyethylene glycols (PEGs) PEG-EO10-14, eight benzalkonium chlorides (BACs) BAC C10 - C17, and 14 alkyl ethoxylates (AEOs) C-12 EO6-13 and C-13 EO6-11 (SI Table S2) present in the HFWW. To be considered a positive library identification, reverse fit and forward fit scores were required to be 90% or better. Additionally, precursor ion mass errors were required to be less than five (parts per million) ppm error and the intensity threshold of fragments be within 5% (Oetjen et al., 2018). Nontarget peaks were required to have an intensity >1000 counts per second (cps), a signal-to-noise ratio >10, and an intensity five times greater than the method blank.

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