LAND APPLICATION OF DRILLING MUD FROM OIL AND GAS EXPLORATION: AGRONOMIC AND ENVIRONMENTAL IMPLICATIONS

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Abstract: Waste from oil and gas drilling is often disposed of through land application. These studies examined the agronomic and environmental impact of applying drilling mud to agricultural land. Specifically, we investigated the best methods for increasing TPH degradation from land applied oil-base drilling mud (OBM), and potential BTEX leaching, along with salt accumulation and leaching from water-base drilling mud (WBM) applied to wheat. This was achieved by conducting field and greenhouse leaching experiments. Mixing OBM with caliche, lime, or gypsum resulted in > 90% TPH degradation 60 days after application with no decrease in plant yield. BTEX leaching from surface applied OBM was minimal over 90 days and all BTEX leachate concentrations were below EPA drinking water thresholds. Increased surface applications of OBM resulted in decreased TPH degradation rates. Initial soil EC values were high after land applying WBM to wheat. Increased rainfall decreased soil EC levels by leaching salts out of the top 15 cm where the majority of roots occur for wheat. Depending on rainfall amounts, soil EC decreased below the saline threshold in 90-300 days. On average, it required 3 cm of rainfall to lower the soil EC by 1 mS cm^{-1} for the 0-7.5 cm depth. Soil SAR values increased at each sampling day although never reaching the sodic threshold. Application of WBM had no impact on wheat yield except at the March application date. The land application of drilling mud may not have a long term agronomic and environmental impact if correctly applied. However, detrimental results to soil and plants can occur if over-applied.

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CHAPTER I

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INTRODUCTION

The oil and natural gas industry is a very important and lucrative business in the United States. Currently, the US ranks third in the world in oil and natural gas production. There are approximately 910,000 oil and natural gas wells onshore and approximately 4,900 offshore wells that together produce nearly 1.9 billion barrels of oil and roughly 23.5 trillion cubic feet of natural gas annually (American Petroleum Institute, 2011). A big part of this oil and natural gas production is occurring in the state of Oklahoma. In the year 2009, Oklahoma ranked 6th in the nation by producing 67 million barrels of oil that represented nearly 3.5% of the total US oil production. They are also ranked 3rd in the US natural gas production with production nearing 1,857,777 trillion cubic feet of natural gas which accounted for 8.6% of the total US natural gas production. The oil and natural gas industry plays a vital role in our economy and generates $7.6 billion dollars in labor income for Oklahoma and employs 71,000 workers which is more than 3% of the states total workforce (Evans, 2009). In Oklahoma from the year 2000 through 2011 on average there were 2500 new wells drilled every year. These wells consisted of oil, natural gas, and dry holes. From 2009 to 2011, drilling permits and average monthly rigs in

Oklahoma increased from 2,500 to 3,732 and 94 to 180, respectively (Oklahoma Corporation Commission, 2011).

An increase in drilling ultimately leads to an increase in drilling waste products. In 1995, a study conducted by the American Petroleum Institute (API) estimated that around 150 million barrels of drilling wastes were generated on land in the United States alone (American Petroleum Institute, 2000). Drilling waste can then be further broken down into two categories, drilling fluids and drill cuttings (mud). Drilling mud is used to help cool the drill bit, maintain borehole pressure, and aids in bringing the drill cuttings to the surface where the fluids and cuttings can then be separated (Ukeles and Grinbaum, 2004). Drilling muds are comprised of a base liquid (water or diesel) and numerous solids and liquids added to the mud to allow for optimum drilling conditions. Some of the products added to the mud may be deleterious and therefore need to be handled properly (Onwukwe and Nwakaudu, 2012). If the base liquid is water, then the term “water based mud” (WBM) is used, and “oil based mud” (OBM) when diesel is the base fluid. During a typical drilling operation, the first 2,400 meters utilizes water as the drilling fluid. The following 1,500 meters and especially when the drilling bit is directed somewhat horizontally, diesel is used as the drilling fluid. On average, a typical southeastern Oklahoma natural gas well that ranges from 4,300-5,200 meters deep will produce 340 m^3 of OBM (Barker et al., 1992).

The large amounts of drilling waste produced gives rise to multiple questions about how to appropriately dispose of them. Drilling waste disposal can be achieved in several ways such as, land application, onsite burial, hazardous waste landfills, and underground injection. Depending on the circumstances, one of these disposal techniques is used to properly eliminate the drilling mud waste.

Onwukwe and Nwakaudu (2012) conducted a comprehensive review over different techniques of disposing of drilling wastes. Three common methods of drilling waste disposal include burial, underground injection, and land application. Burial of the waste usually occurs on the site where the well was produced, known as “reserve pits”. The drill cuttings are generally put into the reserve pit and then buried after the liquid portion evaporates. This method is appealing to the producer (i.e. oil and gas exploration companies) since there is no cost in transportation of the wastes. However, burial could potentially lead to the concentration of salts, heavy metals, industrial chemicals, and hydrocarbons that could leach and contaminate drinking and irrigation water. Underground injection is used when the geological formations allow for it. Drilling fluids and cuttings are mixed together until a liquefied slurry is achieved and then it is injected into a porous, permeable formation that is sealed on the top and bottom by impermeable layers. A potential negative risk of this method is the possible pollution of freshwater via case or borehole malfunction. Land application involves actually spreading of drilling wastes onto the soil at a predetermined loading rate which is based on soil conditions. One must be careful to not apply so much as to render anaerobic conditions in the soil where the microbes will not be able to break down the waste. In Oklahoma OBM disposal is primarily land application to the soil. This allows the microbes in the soil to degrade the hydrocarbons in OBM to CO_2 and wate

LITERATURE REVIEW

OBM Components

The components that make up OBM will vary depending on what each well site demands. Some of the chemicals and components that can occur in OBM regularly are barite ($BaSO_4$), bentonite, calcium lignosulfate, lignite, diesel fuel, pipe thread lubricant, potassium chloride, sodium dichromate, and sodium hydroxide (Moseley, 1983).

Rules/Regulations

Disposal of OBM in Oklahoma is regulated by the Oklahoma Corporation Commission (OCC). The rules for OBM application are stated in the Oklahoma administrative code and register, Title 165:10-7-26 (One-time land application of contaminated soils and petroleum hydrocarbon based drill cuttings). OBM can only be applied one time to a single site. In addition, the OCC suggests that all OBM should be mixed with a stabilizer at a 3:1 ratio of stabilizer to mud prior to land application. The most common stabilizers in Oklahoma are gypsum (calcium sulfate) and lime (calcium carbonate). The OCC also has several site requirements pertaining to environmental safety such as stream and groundwater protection. Current OCC guidelines restrict OBM application to soils with an exchangeable sodium percentage (ESP) below 10% and electrical conductivity (EC) below 4 mS cm^{-1}. Soil EC is an indicator of the total dissolved solids (TDS) in the soil.

The maximum allowable slope that can be applied to is 8 percent and there must be 102 cm or greater depth to bedrock for OBM. The rules also take into consideration characteristics of the drilling mud itself: TDS, chlorides, and total petroleum hydrocarbons (TPH). Maximum soil loading limits are 6,700 kg TDS ha^{-1}, 3,900 kg Cl ha^{-1}, and 45,000 kg TPH ha^{-1}. In addition, the total amount of dry solids applied cannot exceed 224,000 kg ha^{-1}.

Potential Hazards

When applying OBM one must be aware of the potential side effects it can have on grass and crop production due to TPH toxicity, potential heavy metals, and possible naturally occurring radioactive material (NORM). If applied at too high of concentrations there is a risk of TPH toxicity to the plants and virtually complete biomass failure (Macyk and Abboud, 1994). Kisic et al. (2009) found that there were no effects on crop density when applying OBM at a rate of 5,000 mg TPH kg^{-1} soil. Soil TPH threshold levels vary depending on context and the source of literature. The Oklahoma Guardian has set a threshold of 10,000 mg TPH kg^{-1} soil as the protection limit for plants. Not only is TPH toxicity an issue but the smothering of crops can occur when applying OBM at 224,000 kg dry solids ha^{-1}.

Kisic et al. (2009) conducted a four year pot study where OBM was applied and its effects on soil heavy metal concentrations and wheat emergence, density, and yield were monitored. OBM was mixed with soil at ratios of 1:1, 1:2, and 1:3 kg kg^{-1}. In 2003, the concentration of Cd, Hg, Pb, Mo, Ar, Ni, Co, Cu, Cr, Zn, Ba, and V concentrations were

0.29, .08, 19, 0.5, 16, 28, 12, 15, 55, 69, 1423, and 40 mg kg^{-1}, respectively, in the 1:2 OBM:soil. In 2007 the same metal concentrations in the soil were 0.36, 0.07, 20, 1.1,16, 13, 14, 17, 36, 70, 1500, and 24, respectively. For all other treatments, heavy metal concentrations did not significantly decrease in the soil over a four year period after an initial application of OBM. In the 2003/2004 growing season the wheat control yielded 44.5 g pot^{-1} and was significantly higher than the average yield (26.0 g pot-1) of all the treated pots. It was found that wheat emergence and density was inversely proportional to the amount of OBM applied. Treatments that had higher amounts of OBM had lower emergence and density because the higher contamination caused formation of a thin film around the seed germ and inhibited inflow of oxygen which lead to embryo death.

While drilling for oil and natural gas, there is a potential risk of producing drilling mud that has been contaminated with NORM. Zielinski and Otton (1999) conducted a review of NORM concentrations in produced water in oil field sites throughout the United States. They found that the NORM element radium (Ra) was usually found in Barite (BaSO4) scale. This scale precipitates out from the produced water due to changes in temperature, pressure, and salinity as it is brought to the surface and is normally found in oil field pipe casing, fittings, tanks, and equipment that have come into contact with the produced water for an extended period of time. It was also determined that Ra was more soluble in chloride rich waters. The main concern for the release and transportation of Ra is when applying NORM waste to an organic rich soil that has high amounts of sulfate reducing bacteria. In the presence of sulfate reducing bacteria, barite solubility will increase, allowing Ra to become free to leach into ground water.

Application and Degradation of OBM

A study conducted by Wellman et al. (2001) found that hydrocarbon concentrations of 5,000 mg kg^{-1} in the soil degraded the most completely when mixed with manure on a 20% dry weight ratio when compared to manure at a 10% and 5% dry weight ratio. They found that hydrocarbon degradation in the 20% manure and soil mixture reached 81% compared to 32% degradation in soil alone. However, one cannot rule out the possibility that the decrease in hydrocarbons might be partly due to sorption to manure, thereby preventing hydrocarbons from being extracted. Penet et al. (2004) discovered that microbes degraded the hydrocarbons faster if they were already predisposed to the diesel compared to microbes which were in a treated sludge from a wastewater plant. Both Wellman et al. (2001) and Penet et al. (2004) found that microbes degraded the straight chained hydrocarbons faster than the branched chain hydrocarbons.

Maletic et al. (2009) conducted a study that examined total hydrocarbon and mineral oil degradation and leaching in the soil. Initial total hydrocarbon and mineral oil concentrations in the soil were 41,400 and 27,600 mg kg^{-1}, respectively. It was found that hydrocarbons and mineral oils in the soil degrade via first order kinetic degradation equations. It was determined that hydrocarbon degradation and mineral oil degradation were greater in the top most portion of the soil due to excess oxygen availability for the microbes. Leaching of hydrocarbons and mineral oil to the lower depths of the soil was ruled out because there was no increase in hydrocarbon and mineral oil concentrations in the subsurface.

Macyk and Abboud (1994) conducted a field study where they applied five different rates of diesel invert mud drilling waste; 0, 1.0%, 2.0%, 3.0%, and 4.0% oil in the soil-waste matrix. Forage production and degradation of the oil was monitored over a three year period. It was determined that more oil degraded when applied on a cultivated soil at the 2.0% or lower oil application rates. There was an increase in oil degradation when multiple additions of diesel waste were added to the soil compared to the single application. Forage yield was highest on the control (no amendment) followed by the 1.0% and 2.0% oil application rates, and practically no yield on the 4.0% addition.

It was determined by Bjorklof et al. (2008) that when observing aged petroleum hydrocarbons (PHC) such as lightweight fuel oils and lubrication oils in the soil, degradation rates were twice as high under aerobic conditions as anaerobic conditions. When oxygen was not limited, PHC degradation was greater than 10 mg PHC kg^{-1} soil day^{-1} when the initial soil PHC concentration was below 2,000 mg kg^{-1}. It was found that PHC degradation rates were linear and dependent upon the dissolution rate of contaminants in the soil water phase.

A field study was conducted by Cansfield et al. (1978) that monitored hydrocarbon degradation over an 833 day period on a Red River Clay soil. The experimental plots had 9.45 Mg of hydrocarbons ha^{-1} applied along with 336.3 kg N ha^{-1} and 48.9 kg P ha^{-1} to promote microbial degradation. The oil content of the soil after application was 1.45% of the air dried soil weight. After 833 days soil samples were taken from the plots and then allowed to air dry. Forty grams of air dried soil was then taken and extracted three times with 175 mL of tetrahydrofuran. The extract was then fractionated into saturates, monoaromatics, diaromatics, polyaromatics and polar compounds, and high molecular weight materials. Percent degradation of the fractioned extracts was 54.6, 50.0, 57.1, 44.4, and 11.1, respectively. Total degradation was 50.35 %.

Coulon et al. (2005) conducted a mesocosm study looking at degradation of hydrocarbons in sub-arctic soils in relationship to temperature and the addition of fertilizer. Three temperatures were used to analyze hydrocarbon degradation (4°C, 10°C, and 20°C). Arabian crude oil and diesel were applied to the soil at rates of 28.53 and 27.33 mg g-1 dry soil, respectively. Fertilizer was applied to the soil at rates of 1.2 and 0.1 mg N and P g^{-1} dry soil. After 180 days, total alkane losses of the crude oil and diesel ranged from 77-95% whereas total poly aromatic hydrocarbons loss never exceeded 80%. It was found that the most cost effective temperature to degrade the hydrocarbons was at 10°C. Although, increasing soil temperature does increase the rate of microbial degradation of hydrocarbons it also increases the hydrocarbons solubility which leads to an increase in toxicity which could be another potential problem.

While there has been tremendous research conducted on the degradation of TPH in soils contaminated from industrial activities, there is little to no research specifically on OBM. Also, the contexts of the previously described studies are remediation, whereas the goal of land application of OBM is to dispose of the waste in a manner to prevent negative soil, water, and agronomic impacts. Barker et al. (1992) conducted a field plot study in southeastern Oklahoma looking at the effects of land farming OBM mixed with flyash. The objectives of this study were to monitor hydrocarbon, leaching, biodegradation, and metal leaching in soils. Test plots were 3.048 X 3.048 meters and 0.61 meters deep with a liner at the bottom to prevent leachate from reaching the soil and groundwater. There were five treatments which consisted of a control plot (treatment 1) that was backfilled with 50.8 cm of non-OBM amended soil, treatment 2 consisted of 38.1 cm of backfilled soil that had 12.7 cm of a 1:1 ratio of flyash and OBM applied to it. Treatment 3 consisted of 45.7 cm of backfilled soil that had 5.8 cm of OBM mixed into the top 7.6 cm. Treatment 4 consisted of 45.7 cm of backfilled soil that also had 5.8 cm of OBM mixed into the top 7.6 cm of soil and was amended with 4.5 kg of fertilizer (16-8-8). Treatment 5 consisted of 45.7 cm of backfilled soil that had 5.8 cm of OBM mixed into the top 7.6 cm of soil and was amended with 45.4 kg of fertilizer (2-1-2). A second application of fertilizer was applied to treatments 4 and 5. All plots received 1 kg of elemental sulfur and were tilled six times over the length of the study (209 days). During this study period the test plots received 65.6 cm of rainfall. The OBM used had a TPH concentration of 104,000 mg kg^{-1}, EC of 24.1 mS cm^{-1}, and a SAR of 0.5. The flyash used had an EC of 11.5 mS cm^{-1} and an SAR of 1.9. The 1:1 ratio of OBM and flyash had a pH of 11.3, EC of 12.7 mS cm^{-1}, TPH concentration of 48,543 mg kg^{-1}, and concentrations of chloride, sodium, and calcium at 121.5, 5.3, and 93.0 meq L^{-1}, respectively. There were significant amounts of barium, zinc, chromium, lead and arsenic present. TPH concentrations after 209 days for treatment 1, 2, and 3 at the 0-12.7 cm depth were <5.0, 33,310, and 24,688 mg kg^{-1}, respectively. At the 15-28 cm depth TPH concentrations for treatment 1, 2, and 3 were 42.1, 63.6, and 67.7 mg kg^{-1}, which determined that TPH did not migrate in the soil. Further analysis showed that there was no detectible TPH in the leachate throughout the study which also showed that the TPH did not migrate. At the end of the study the degradation of oil and grease for treatments 2, 3, 4, and 5 were 48, 75, 90, and 83 %, respectively. The fastest degradation rate occurred with plot 4 that was fertilized with the 4.5 kg of 16-8-8. All metals analyzed in the leachate were below the drinking water standards except for Pb measured in treatments 2, 3, and 5 at 0.32, 0.24, and 0.28 mg L^{-1}, respectively. Soil levels of lead in the 15-39 cm depth were < 1.0 mg L^{-1} and suggested that a minute amount, if any, migration had occurred. The high levels of lead could be explained because these trial plots only had 50.8 cm of backfilled soil whereas, in Oklahoma, Rule 165:10-7-26 says that petroleum hydrocarbons must be applied to a soil that has at least 102 cm thick and has a depth to groundwater that exceeds 3.05 meters, which would further dilute the concentrations of metals in the soil.

WBM Components

Some components that make up WBM will vary, while others are more consistent. Some of the chemicals and components that can occur in WBM are barite ($BaSO_4$), bentonite clay, calcium lignosulfate, lignite, potassium chloride, sodium dichromate, sodium hydroxide and pieces of rock that come from the bore hole geology (Moseley, 1983). Deville et al. (2011) found that WBM was more effective in drilling when they were customized for each individual shale play and developed based upon the distinct formation chemistry. However, WBM is characterized as typically containing a high amount of water (>70%), TSS, and sodium. Therefore it is the high salinity and sodicity of the WBM that presents the greatest risk and challenge of land application. Due to the high moisture content, WBM is usually sprayed onto soils.

Rules/Regulations

In Oklahoma disposal of WBM is done primarily by land application. Disposal of WBM in Oklahoma is regulated by the OCC and the rules for WBM application are stated in the Oklahoma administrative code and register, Title 165:10-7-19 (One-time land application of water-based fluids from earthen pits and tanks). Current guidelines dictate that WBM can be applied to a single site only once every three years if the soils test requirements are met. The maximum allowable slope that can be applied to is 8 percent by spray irrigation method. Depth to bedrock must be at least 51 cm. Soil EC must be below 4 mS cm^{-1} and ESP must be less than 10%.

Potential Hazards

When applying high concentrations of saline and sodic water via WBM application there is an increased risk of obtaining soils that are saline (EC > 4 mS cm^{-1}), sodic (SAR > 15), and saline-sodic (EC > 4 mS cm^{-1} and SAR > 15). The effects of saline-sodic soils can lead to the potential loss of yield in crops, stunting of plant growth, complete crop failure, and loss of land due to salinization (Rhoades and Loveday, 1990). Not only does sodicity decrease crop yield, it also destroys important soil physical properties. Zvomuya et al. (2009) conducted a field plot study looking at the effects of WBM applications on sandy loam soil hydraulic properties in Medicine Hat, Alberta Canada. The WBM used consisted of bentonite (primarily Na-montmorillonite), other additives which depend on the type of formation present and formation cuttings. Specific densities of the WBM for the years 2003-2005 were 1170, 1065, and 1130 kg m^{-3}. It was applied at 80 m3 ha^{-1}, 40 m3 ha^{-1}, which equals mass rates of 93.6 Mg ha^{-1} and 46.8 mg ha^{-1} in 2004, 85.2 Mg ha^{-1} and 42.6 Mg ha^{-1} in 2005, and 90.4 Mg ha^{-1} and 45.2 Mg ha^{-1} in 2006. There was a 0 m3 ha^{-1} (control) included each year for comparison. Some of the plots received a single application over the three year period while other plots received one application per year. It was found that when applying WBM every year at the rate of 80 m^3 ha^{-1}, soil structure, hydraulic conductivity and the hydraulically active macroporosity were negatively affected, whereas the 40 m^3 ha^{-1} annual application had no significant effect on the soil hydraulic properties when compared to the control (0 m3 ha-1) treatment. The results also showed that there was no significant effect on soil hydraulic properties after the single WBM application of 80 m^3 ha^{-1}, whereas three applications of WBM at 80 m^3 ha^{-1} had noticeable effects on the soil hydraulic properties one year after the last application.

Bates (1988) managed a column study designed to examine the fate of barium, zinc, chromium and chloride from reserve pit fluids and sludges that were applied to potted bermudagrass in a silt loam and a sand. The reserve pit fluids were applied at a ratio of 8 parts soil to 1 part reserve pit fluids. Over the next 84 days, 100 mL of tap water was applied per day to the columns. The total masses of Ba, Cr, Zn, and Cl in the silt loam columns after application were 308, 429, 285, and 115 mg, respectively. The total masses of Ba, Cr, Zn, and Cl in the sand columns after application were 116, 76, 93, and 142 mg, respectively. It was found that out of the total amount of contaminants applied to the columns, only 0.25% of barium, 0.13% of zinc, 0% of chromium, and 78% of chloride was found in the silt loam leachate; whereas, 1% of barium, 0.62% of zinc, 0% of chromium, and 81% of chloride was found in the sand leachate. When looking at metal uptake in bermudagrass, it was found that the Zn and Cr concentrations in the silt loam control were 36.1 and 4.28 mg kg^{-1} whereas the silt loam with reserve pit fluids had Zn and Cr concentrations of 37.9 and 4.96 mg kg^{-1}, respectively. When looking at metal uptake in bermudagrass, it was found that the Zn and Cr concentrations in the sand control were 68.60 and 10.97 mg kg^{-1} whereas the sand with reserve pit fluids had Zn and Cr concentrations of 82.12 and 9.20 mg kg^{-1}, respectively. The results showed that the pollutants added to the soil can be taken up by plants and or transported through the soil column. Conveyance of the contaminants was related to soil texture with the coarser textured soils transporting the contaminants faster than the finer textured soil. Even though metals leached through the soil column, the total amount was very small compared to the total metal loading rate.

NORM concentrations in WBM can pose a threat to health just as OBM can. Under certain conditions WBM can be even worse due to the increased chloride concentrations of the water enhancing the solubility of the NORM element Ra (Zielinski and Otton, 1999).

Application of WBM to Soils

Bauder et al. (2005) conducted a two year study in which WBM was land applied to wheat in loamy to sandy loam soils. The WBM's used over the two year study were primarily dominated by bentonite clays, formation cuttings, and Na compounds; the WBM's specific gravities ranged from 1.03-1.29 g cm^{-3}, pH ranged from 8.40-9.60, EC ranged from 1.14-2.63 dS m^{-1}, and solids content ranged from 7.2-35%. The WBM was applied once a year during the fallow period at multiple rates that ranged from 2.2-94 Mg ha^{-1}. The authors showed that while a WBM application rate of about 100 Mg ha^{-1} was not beneficial to wheat production, it was not detrimental to yield on three of the four test sites. The site that did exhibit a significant increase in yield had the sandiest texture and most likely benefited from the increased water retention capacity due to added bentonite from the WBM. Soil tests indicated the WBM applications did not significantly increase extractable trace element concentrations.

Bauder et al. (1999) established a greenhouse experiment to examine the effects of applying WBM to corn and sudangrass and monitored crop growth and iron and zinc uptake. The WBM consisted primarily of Na-bentonite, barite ($BaSO_4$), Soda ash, $Ca(OH)_2$ xH_2O, sawdust, Drillpac (proprietary material), lignite, and partially hydrolyzed polyacrylamide (anionic polymer).They found that sorghum-sudangrass and corn both had an increase in dry matter yield when applying drilling fluids at rates of 5 to 60 g kg^{-1} soil. At these application rates Fe and Zn concentrations were beneficially taken up by the crop which led to the increase in dry yield. Soil EC and pH did not increase appreciably; however soil SAR values did increase but did not impact the soil negatively when drilling mud was applied at the previously mentioned rates.

Ganjegunte et al. (2005) conducted a three year study to evaluate the effects of coal bed natural gas (CBNG) water application on soil chemical properties at multiple locations in Northwestern Wyoming. The CBNG used in this study consisted primarily of sodium

(Na^+) and bicarbonate (HCO_3^-) ions; the pH ranged from 7.7 to 8.2, EC from 2.0 to 2.9 dS m^{-1}, SAR from 17.2 to 32.8. Typical Northwestern Wyoming CNGB water has a TDS concentration that ranges from 270-2,720 mg L^{-1}. The plots monitored had three years of previous applications of CNGB water to them. Six soil depths (0-5, 5-15, 15-30, 30-60, 60-90, and 90-120 cm) were analyzed for EC and SAR via saturated paste extract. It was found that the EC and SAR of the saturated paste extracts were significantly higher in the irrigated soils when compared to the controls. Their results also showed that the CBNG waters generally used for irrigation were not suitable for direct land application and that they caused a buildup of salts and Na in the irrigated sites compared to the control.

Ganjegunte et al. (2008) monitored six sites in Northwestern Wyoming that had previous land applications of saline-sodic coal bed natural gas (CBNG) co-produced water for up to four years. Application was monitored by multiple CBNG producers and loading rates were undetermined. Their objective was to determine the effects of CBNG water on soil chemical properties (EC, pH, and SAR) at multiple depths (0-5, 5-15, 15-30, 30-60, 60-90, and 90-120 cm) when compared to non-irrigated soils. The pH of the CBNG used in the experiment ranged from 7.1-9.1, EC ranged from 1.6-4.8 dS m-1, and SAR ranged from 17.2-56.1. In 2003 the soil EC at the 0-5, 5-15, 15-30, 30-60, 60-90, and 90-120 cm depths at the Johnson-2 site was 1.0, 0.8, 0.8, 1.4, 3.9, and 5.7 dS m^{-1}, respectively. In 2004 the soil EC was 12.3, 8.8, 3.8, 1.7, 7.6, and 12.1 dS m^{-1}, respectively. The SAR for the same site and depths in 2003 was 1.6, 1.2, 2.0, 3.4, 7.4, and 8.6, respectively. In 2004 the SAR was 28.8, 22.3, 8.5, 5.1, 10.4, and 12.5, respectively. It was found that multiple applications of CBNG water gave rise to significant increases in soil EC to 120 cm on the fine-textured soils and to 60 cm on the course-textured soils. The SAR significantly increased to a depth of 120 cm in the fine-textured soil when compared to the nonirrigated soils. It was determined that the finer textured soils were more susceptible to salinity and sodicity buildup.

Jalali et al. (2007) conducted a column study where they monitored the effects of applying saline-sodic (EC=6.04 dS m^{-1} and ESP=25.3) irrigation waste water to two different calcareous soils; soil 1 had an initial EC of 19.1 dS m^{-1} and ESP of 28.8 while soil 2 had an EC of 5.2 dS m^{-1} and ESP of 9. After 6-7 pore volumes leached through the soil columns, soil 1 had an ESP of 29.7, and soil 2 had an ESP of 21.The results showed that ESP increased for both soils via the increase of exchangeable Na and the decrease of Ca, Mg, and K on the exchange complex. No soil structure sodification was noted as long as the high EC wastewater was continually used for irrigation. This was due to the fact that dispersion of soil occurred if good quality irrigation water was used which decreased the salinity via leaching of salts and left excess amounts of sodium in the profile which led to high SAR's.

Numerous studies have been carried out looking at the effects of land applying saline and saline-sodic water for irrigation. However, there is a scarcity of research looking at the effects WBM application has on wheat production and soil chemical properties. Addressing these impacts in a meaningful way will require continued research efforts to understand the immediate and long term effects of land applying WBM.

BTEX and Oil-Base Mud

The primary fluid used in OBM is diesel fuel (Mosely, 1983). This diesel fuel is what gives rise to the TPH concentrations in the drilling mud. TPH consists of a broad family of several hundred chemical hydrocarbon compounds that are derived from crude oil. TPH is characterized by carbon chain lengths that range from C_6-C_{35} TPH can be further broken down into gasoline range organics (GRO) with carbon chain lengths that range from C_6 to C_{10-12} and Diesel range organics (DRO) with carbon chain lengths that range from C_{8-12} to C_{24-26}. The TPH fraction that deals with the C_6-C_8 carbon chains (BTEX) poses a significant risk to humans and the environment. All BTEX's are known to cause neurological effects; primarily by causing central nervous system depression. The greatest concern of the BTEX's is the compound benzene which has well documented hematological, immunological, and lymphoreticular effects in humans and animals at low levels of inhalation exposure. It is also a known carcinogenic to humans via inhalation or oral exposure (ATSDR, 1999). At 25 ° C, benzene has a log Kow value of 2.13 and its solubility in water is 1,760 mg benzene L^{-1}. This solubility poses a potential threat of transportation of benzene via water; benzene is known to cause leukemia and because of this the current drinking water MCL is 5 µg L^{-1} (Sawyer et al., 2002).

BTEX Degradation, Sorption, and Transportation

Dou et al. (2007) conducted a study examining the effects of anaerobic BTEX degradation in soils bioaugmented with mixed consortia under nitrate reducing conditions. They found that BTEX could be biodegraded anaerobically to undetectable limits within 70 days if initial concentrations of BTEX were 100 mg kg^{-1} soil or below. Degradation was fastest for toluene followed by ethylbenzene, m-xylene, o-xylene, benzene, and p-xylene respectively.

Franzmann et al. (1999) evaluated the effects of microbial benzene degradation at four soil depths (0-0.25, 0.25-0.35, 0.5-0.6, and 2.6-2.8 m) that was overlaying gasoline contaminated groundwater. Their results showed that the fastest benzene degradation to carbon dioxide occurred in the soil samples collected at the 0.25-0.35 m depth at a rate of 83±13 μmol kg^{-1} soil day^{-1} with a half-life of 11±1 days. At this depth, there was a plentiful supply of oxygen and benzene. Microbial degradation at the surface, 0.5-0.6, and 2.6-2.8 m were all lower due to limiting concentrations of benzene for the surface and oxygen for the two lower depths.

Hers et al. (2000) monitored the biodegradation processes of benzene, toluene, and xylene (BTX) through extensive sampling of a BTX contaminated soil at multiple depths using model simulations. Surface soil solution concentrations of BTX ranged from 0.005 mg L^{-1} at the surface to 50 mg L^{-1} at 1.5 m. BTX vapor concentrations, soil moisture, and oxygen content were evaluated. The zero-order BTX degradation rates in pore water ranged from 0.8 to 6 mg L^{-1} h^{-1} and maximum degradation occurred at the 0.9 m depth. Model simulations that incorporated diffusion, sorption, and biodecay revealed that microbial degradation kinetics, oxygen transport and the availability to mineralize hydrocarbons were critical. It was discovered that when BTX concentrations in the soil were high, first-order rate degradation curves over estimated BTX degradation because it assumed that both oxygen and hydrocarbon degrading microbes were available in excess which in this case they were limited.

Zhang and Bouwer (1997) conducted an experiment that monitored the biodegradation of benzene, toluene and naphthalene in soil-water slurry microcosms. The objective of the study was to simulate biodegradation at waste sites where sorption reaches equilibrium before biodegradation becomes a major contributing factor. Biodegradation was analyzed in soil-free solutions and pre-equilibrated soil-water slurry solutions that were inoculated with soil bacteria. All microcosms contained benzene, toluene, and naphthalene at concentrations of 3.9, 4.6, and 1.28 mg L^{-1}, respectively. Results showed that in the soil-free solutions degradation started in the first 24 hours and nearly 100% degradation of benzene, toluene, and naphthalene occurred at a maximum of 90, 14, and 60 hours, respectively. In the soil-water slurry microcosms, degradation of benzene, toluene, and naphthalene did not start to occur until three days have passed and it took two weeks to achieve around 100% degradation. This was due to the hydrocarbon adsorption to the soil which made it harder for the microbes to obtain the hydrocarbon compounds and degrade them.

Fine et al. (1997) conducted a review examining hydrocarbon volatilization and transportation in multiple soils. As soil moisture content decreased, hydrocarbon sorption to soil increased as well as vapor phase transportation. As soil moisture increased, transportation of hydrocarbons in the nonaqueous phase liquid increased.

Voudrias and Li (1993) monitored unsteady state benzene vapor transport in large (10.5 cm x 100 cm) columns that were packed with dry and wet soils to assess the adequacy of the diffusion equation. It was determined that the diffusion equation used for water phase portioning and linear sorption isotherm adequately described benzene vapor transport in a dry soil column. However, it did not describe benzene vapor transport in the wet soil possibly due to microbial degradation.

Nathwani and Phillips (1977) conducted a study looking at the effects of hydrocarbon concentration, soil type, and organic matter content on the adsorption and desorption of certain hydrocarbon components in crude oil in the soil. The soils used in this experiment had textures that ranged from sand to silty clay, organic matter ranged from 1.0 to 16.2%, and clay content ranged from 1.3 to 45.4%. The hydrocarbon compounds used in this experiment were benzene, o-xylene, toluene, and n-hexadecane. For the adsorption-desorption experiments, 10 mL of the selected hydrocarbon compounds were applied to 10 g of soil in a 250 mL flask. Adsorption equilibrium was allowed to take place between the hydrocarbons and the soil. Once equilibrium was reached, 10 mL of distilled water was added to the soil and then allowed to equilibrate and a sample of the aliquot was taken for analysis of hydrocarbon desorption. It was found that the equilibrium distribution of the four selected hydrocarbons between the liquid phase and adsorbed phase can be represented by the Freundlich isotherm as long as the concentration range was between 1-100 mg hydrocarbons L^{-1}. The percent of hydrocarbons desorbed varied inversely with the amount of organic matter. The soil with the highest organic matter (16.2%) had 48.6% desorption of benzene compared to 75.5% desorption of benzene on the soil with 1.0% organic matter.

There have been many studies dealing with biodegradation, adsorption, and transportation of benzene and other low molecular weight and volatile aromatic hydrocarbons in the soil. However, very few studies have dealt with the degradation, adsorption, and transportation of BTEX in soils after land applying OBM. Due to the hazardous risks of BTEX toxicity to humans and to the environment, further studies should be conducted looking at BTEX transportation and degradation in soils after land applying OBM under different scenarios such as: multiple loading rates, moisture regimes, and time.

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CHAPTER II

SURFACE APPLICATION OF OIL-BASE DRILLING MUD MIXED WITH GYPSUM, LIME, AND CALICHE

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ABSTRACT

The current increase in oil and gas drilling activity has resulted in production of large quantities of oil base “mud” (OBM) to be disposed of. Land application of OBM to agricultural land is a common disposal technique that presents some agronomic and environmental challenges since the material is rich in petroleum hydrocarbons. The objective of this study was to determine the effect of mixing OBM with bulking materials on hydrocarbon degradation and forage production after land application of the mixtures. An OBM was collected from Western Oklahoma and characterized for environmentally relevant properties such as total petroleum hydrocarbons (TPH) and trace metals. OBM was bulked with either lime, gypsum, or caliche, at a ratio of 3:1 or 1.5:1 OBM:bulking material. All mixtures were surface applied at equal TPH loading rates (8625 kg ha^{-1}) and soil samples taken at 7, 45, 60, and 170 days after application for evaluating TPH, electrical conductivity (EC), pH, and sodium absorption ratio (SAR). After 7 days >50% of applied TPH degraded, which resulted in soil concentrations less than thresholds

recommended for residential neighborhoods. By day 170, approximately 99% of applied TPH degraded. There was no difference in TPH degradation as a function of type and amount of bulking agents used with the OBM. Application of OBM did not significantly decrease total biomass compared to unamended control. Therefore, use of caliche, lime, or gypsum bulked with OBM at a 1.5:1 ratio (OBM:bulking agent) would suffice for achieving acceptable TPH degradation when surface applied and non-incorporated.

INTRODUCTION

The United States is currently experiencing an oil and gas drilling boom. There are approximately 910,000 and 4,900 onshore and offshore oil and natural gas wells, respectively, that produce nearly 16 million m^3 of oil and 665 billion m^3 of natural gas (American Petroleum Institute, 2013). However, increased drilling activity has also lead to an increase in the production of drilling wastes, specifically drilling fluids and drill cuttings (aka “mud”). In 1995, a study conducted by the American Petroleum Institute estimated that around 150 million barrels of drilling wastes were generated on-shore in the United States alone (American Petroleum Institute, 2000).

Drilling mud is manufactured and utilized by the drilling industry to help cool the drill bit, maintain borehole pressure, and aid in bringing the drill cuttings to the surface where the fluids and cuttings can then be separated (Ukeles and Grinbaum, 2004). Drilling muds are comprised of a base liquid (water or diesel fuel) with other potential additives such as barium sulfate, bentonite, calcium hydroxide, and byproducts such as cotton seed hulls, used for specific drilling conditions (Moseley, 1983). If the base solution used to make the mud is diesel fuel, then the mud is known as “oil base mud” (OBM). Oil-base mud is typically utilized when drilling depths exceed 1500 m and for the horizontal portion. Due to the high cost of production, OBM is re-used by drillers for as long as possible.

At some point when the OBM can no longer be used in drilling, it must be properly disposed of. On average, a typical southeastern Oklahoma natural gas well that ranges from 4200-5200 m deep will produce 340 m^3 of OBM (Barker et al., 1992). Some of the products added to the mud may be deleterious and therefore need to be handled properly (Onwukwe and Nwakaudu, 2012). There are two options for mud disposal: land application and burial. Burial of the waste can occur onsite in “reserve pits” or at more sophisticated commercial facilities. In Oklahoma, land application is the most common method of OBM disposal. The purpose of land application of OBM is to allow soil microorganisms to degrade petroleum hydrocarbons (i.e. “total petroleum hydrocarbons”; TPH). Land application of OBM is regulated by the Oklahoma Corporation Commission (OCC) and application rates are limited based on loading of TPH, chlorides, and solids (Oklahoma administrative code and register, Title 165:10-7-26). In addition to loading limits, there are also several site suitability requirements such as soil texture, depth to groundwater and limiting layers, slope, soil sodium concentrations, and proximity to surface waters. The OCC also requires that OBM be mixed with a bulking material such as lime or gypsum, at a ratio of 3:1 OBM:bulking material.

Despite the fact that thousands of hectares are currently receiving OBM, there has been relatively little research conducted on this method of disposal. Excessive application rates could lead to soil TPH concentrations that are detrimental to crop growth and present environmental issues. In addition, depending on the geologic formation where drilling occurred, the cuttings contained in the mud could be elevated in trace metals (Bates, 1988) and naturally occurring radioactive material (NORM; Zielinski and Otton, 1999). Although not as common for OBM compared to other types of drilling mud, some OBM samples present a risk of causing soil salinization or sodicity. Few studies have examined TPH degradation from applied OBM; Penet et al. (2004) found that microbes degraded the straight chained hydrocarbons faster than the branched chain hydrocarbons. Macyk and Abboud (1994) conducted a field study with five different application rates of OBM; 0, 1, 2, 3, and 4% oil in the soil-waste matrix. Forage production and degradation of the TPH was monitored over a three year period. It was determined that more TPH degraded when applied on a cultivated soil at the 2% or lower oil application rates. There was an increase in TPH degradation when multiple additions of diesel waste were added to the soil compared to the single application. Forage yield was highest on the control (no amendment) followed by the 1% and 2% oil application rates, and practically no yield on the 4% addition.

The impact of amending OBM with a bulking material prior to land application, as required by the OCC, on TPH degradation is unknown. Specifically, there is no information regarding the effect of the type and rate of OBM bulking material on TPH degradation. Utilization of a bulking ratio less than 3:1 OBM:bulking material would save an appreciable amount of money on land application and conserve bulking materials such as limestone and gypsum. Thus, the objectives of this study were to (i) monitor TPH degradation with time after land application of OBM, (ii) determine the impact of bulking material type and mixing rate on TPH degradation, and (iii) evaluate impact of land application of OBM on forage production and soil pH, salinity, and sodicity.

MATERIALS AND METHODS

Site description and source of materials

The OBM land application study was conducted on a pasture located five miles south of Shattuck, OK (USA). The dominant forage plant was old world bluestem (*Bothriochloa* spp.) and the soil was a Mansic clay loam (fine-loamy, mixed, superactive, thermic aridic calciustolls), which is a well-drained soil found on 3-5% slopes. Mean annual precipitation is 53 to 76 cm. The OBM source was from a horizontally drilled well located in the Cleveland formation. Mixing agents utilized in this experiment for the OBM included lime, caliche, and gypsum. Lime and gypsum were transported in semi-trailers from Woodward, OK and the caliche was transported locally within 20 miles of the application site. In addition, local stockpiled beef feedlot manure was used as a nutrient source for certain treatments (see below).

Oil-base mud characterization

The OBM was analyzed for TPH and total solids prior to land application and mixing with lime, gypsum, and caliche. Total petroleum hydrocarbons were extracted with hexane at a 1:10 solids:solvent ratio, plus addition of 0.5 g Na_2SO_4 for 5 minutes on a reciprocating shaker followed by centrifugation for 10 minutes. Five mL of the resulting supernatant was then equilibrated for 2 minutes with 1 g of silica gel in a glass tube for removal of polar organic compounds. The solution was then analyzed for TPH using infrared spectroscopy (ASTM method D 7066) with the InfraCal TOG/TPH analyzer (model HATR-T2, Wilks Enterprise Inc., East Norwalk, CT). Samples were sent to an outside lab for measurement of low molecular weight petroleum compounds (C_6-C_{12}; Texas Commission on Environmental Quality Method 1005). Radiation was tested by an analysis of "naturally occurring radioactive material" (NORM) through an outside laboratory (Radium 226 and 228: SM7500Ra [M] or DOE EML HASL300 4.5.4 [M].

Calcium carbonate equivalent (CCE) was determined on the OBM sample, lime, and caliche using the method described by Stout et al. (1988). Solids content of all materials was determined gravimetrically after drying in an oven at 35ºC. Beef feedlot manure was analyzed for total nitrogen (TN) and carbon (TC) by the combustion method (Leco TruSpec; St. Joseph, MI).

The OBM was analyzed for total P, K, Mg, Ca, Na, Mn, Cu, Fe, Zn, S, Al, Ni, B, As, Cd, Cr, Ba, Pb, and Mo using the EPA 3050 acid digestion method followed by solution analysis with inductively coupled argon plasma analyzer [ICP-AES; Spectro Ciros, Mahwah, NJ]. Total nitrogen (TN) and total carbon (TC) were analyzed by the combustion method previously described. Soil electrical conductivity (EC) and pH were measured using pH and EC probes with a solid:solution ratio of 1:5 and an equilibration time of 45 min. Total chloride was extracted with de-ionized (DI) water using a 1:20 solid:solution ratio for 1 hr followed by colorimetric flow-injection analyzer (Lachat QuickChem 8000, Loveland, CO).

Experiment setup and mud application

The experimental design consisted of a randomized complete block that had twelve treatments replicated three times. Individual plot dimensions were 4.6 x 27.4 m with 30.4 m alleys. Prior to application, composite soil samples were taken from each block at depths of 0-5, 5-10, and 10-15 cm. Each of the three mixing agents (aka "bulking materials"; limestone, gypsum, and caliche) examined in this study were mixed with OBM on a volumetric basis using a backhoe. Specifically, limestone (L) and gypsum (G) were mixed with OBM at a 3:1 (1) and 1.5:1 (2) mixing agent:OBM ratio. Caliche (C) was only utilized at the 3:1 mixing agent:OBM ratio. In addition, a subsample of the G2 sample was additionally mixed with beef feedlot manure (M) to create two additional treatments. This resulted in a 1.5:1 gypsum:OBM material that contained enough beef manure to provide the plant available nitrogen rate (PAN) for the plot (G2M1), and 1.5 times the PAN rate (G2M2). A control plot that received no amendments was also included. Note that a non-mixed OBM sample was not applied since the Oklahoma Corporation Commission requires that all OBM be mixed with a bulking agent prior to land application. Since TPH of the raw OBM was measured prior to mixing, land application rates to the plots could be made to achieve equivalent TPH loading of 8,625 kg TPH ha^{-1} for each treatment. This rate of TPH loading was chosen because it corresponded to the treatment that added the highest legally allowable solids loading rate (222 Mg ha^{-1}), which was the 3:1 lime:OBM treatment (L1).

The OBM mixtures were applied using a tractor and John Deere hydraulic push gate manure spreader. After application, the amendments were not incorporated in order to simulate the worst case scenario for TPH degradation. Composite soil samples were taken from each plot at 7, 45, 60, and 170 days after application at depths of 0-5, 5-10, and 10-15 cm. At day 60, all plots were harvested and plant yield (biomass) was determined.

Soil analysis

Background soils were tested for routine fertility by the Soil, Water, and Forage Analytical Laboratory (SWFAL) at Oklahoma State University. This included KCl extractable N-NO_3^- Gavlak et al., 2003) and Mehlich-3 extractable P and K (Mehlich, 1984), pH, and EC. The soil collected at each sampling interval was tested for EC and pH via the saturated paste method (USDA, 1954); extract was tested for EC and pH using an appropriate meter. In addition, saturated paste extract was analyzed for Na, Ca, Mg, K, SO_4, B, P, Fe, Zn, Cu, Mn, Al, and Ba via ICP-AES. Results were from the saturated paste extract were used to calculate sodium absorption ratio (SAR):

$$\frac{Na}{\sqrt{\frac{Ca+Mg}{2}}} \tag{1}$$

Where Na, Ca, and Mg are in units of meq L^{-1}. All soils were tested for TPH using the same method described for OBM, except that the soil:solvent ratio was 1:10.

Statistics

Soil chemical data and plant yield were analyzed using the SAS (SAS Institute, 2002) statistical software package. An ANOVA model of response variable was constructed using the PROC MIXED routine. The ANOVA model used mud application rate-bulking treatment and replication as effects. The three replications were included in the model as a random factor. Type III least-square means obtained from the PROC MIXED routine were used for mean comparison tests using the PDIFF option (SAS Institute, 2002). Model parameters and treatment differences were considered significant at the $P < 0.05$ level, specifically to make comparisons between treatments within each sampling event (i.e. time). Potential differences between treatments in TPH degradation rates were assessed by conducting the PROC MIXED routine to compare slope and intercepts for the relationship between time and soil TPH concentrations.

RESULTS AND DISCUSSION

Properties of the oil-base mud and background soil

Background soil tests indicated that phosphorus (P) was 60 to 70% sufficient (Zhang and Raun, 2006) for big bluestem with P concentrations ranging from 4 to 5 mg kg^{-1} at the 0-5, 5-10, and 10-15 cm depth. Potassium (K) was 100% sufficient for big bluestem as K concentrations ranged from 186 to 240 mg kg^{-1} at the three soil depths. There was only a small amount of background nitrate at all three depths (0.5 mg kg^{-1}). Initial soil pH was 7.7, which is common for this semi-arid region of Oklahoma. The low soil EC value of 0.5 mS cm^{-1} indicated that the soil was not near the “saline” threshold of 4 mS cm^{-1}.

Table 1 shows the general properties of the un-amended OBM used in this study. Note the high concentration of TPH, which is typically considered the main parameter of environmental concern. Thus, land application rates are often based on TPH loading. The concentration of TPH in the OBM sample is similar to the OBM examined by Barker et al. (1992). The pH and EC is somewhat elevated, although EC is only slightly higher than 4 mS cm^{-1}, which is considered to be a threshold value for salt sensitive plants (Zhang and Raun, 2006). The elevated EC is likely due in part to the presence of chlorides found in the OBM (Table 1). Note that the EC of the OBM is much less compared to the OBM described by Barker et al. (1992) which was 24.1 mS cm^{-1}.

Although the pH was elevated, this material is not expected to have a dramatic impact on soil pH since the CCE was relatively low. While the material did not possess appreciable levels of N or P, it did contain relatively high concentrations of plant nutrients Ca, Mg, and K which are from geologic materials at the drilling site. Barium concentrations were elevated due to the addition of barium sulfate as a "weighting agent" used during the drilling process.

Concentrations of heavy metals (Zn, Cu, Ni, As, Cd, Cr, and Pb) found in the OBM (Table 1) were all well below EPA 503 thresholds for "exceptional quality" biosolids, indicating that there is little risk of metals contamination from land application of this OBM sample (USEPA, 2014A). In fact, total metals concentrations of OBM were in the normal range typically found in soils (McBride, 1994). Based on the application rates of OBM used in this study, which were made according to Oklahoma Corporation Commission guidelines, the resulting metal loading was also well below EPA recommendations for biosolids (USEPA, 2014). The NORM level was only 2.2 pCi g^{-1}, which is well within the range of levels found in un-contaminated topsoils (USEPA 2014B). A NORM level of 5 pCi g^{-1} in the top 15 cm is considered to be a remediation threshold in many states (USGS, 2014)

TPH degradation

At an application rate of 8,625 kg TPH ha^{-1}, the initial concentration of TPH in the top 5 cm of soil would be about 11,500 mg kg^{-1} soil before any degradation occurred. After seven days, at least 50% of the TPH degraded in the 0-5 cm depth where the OBM was applied at the surface. Note that there was very little TPH measured below the 0-5 cm depth compared to the surface (Tables 2 and 3). Figure 1 shows that many of the treatments exhibited > 70% TPH degradation at seven days after OBM application. After 45 and 60 days after application, TPH levels decreased at least 90% (Figure1 and Table

2). Note that biomass samples were harvested on day 60. By day 170, degradation of TPH exceeded 99% for nearly all treatments (Figure 1 and Table 3). Final TPH concentrations measured at day 170 indicated that TPH levels were well below the plant protection limit of 10,000 mg kg^{-1}, and 2,600 mg $kg^{-1}$for residential areas established by the Oklahoma "Guardian" (Billingsley, 2003). In fact, Figure 1 and Table 2 shows that all but two treatments achieved TPH levels < 1000 mg kg^{-1} by day 45 and 60. This data suggests that surface applications of TPH at 8,625 kg ha^{-1} and through use of the mixing agents used in this study, safe soil TPH levels are mostly achieved at 45 days after application.

This rapid TPH degradation is illustrated in Figure 1. Part of the reason for the high level of TPH degradation in only seven days is due in part to a rainfall event that occurred within four days of OBM application. The increased soil moisture likely provided ideal conditions for the microorganisms to degrade the TPH (Das and Chandran, 2011). However, after the rainfall event this region of Oklahoma experienced a drought of historic proportion for nearly two years. The next appreciable rainfall event at the site did not occur until about 150 days after OBM application.

While some of the decrease in soil TPH levels at the 0-5 cm depth may be due to volatilization of low molecular weight hydrocarbon, mainly benzene, toluene, ethylbenzene, and xylene (BTEX;C_6-C_8), such volatilization would be minimal compared to degradation. The GC analysis showed that the low molecular weight hydrocarbons (C_6-C_{12}) comprised only 17% of the TPH; even if this entire fraction volatized within seven days (which is highly unlikely), it cannot account for the > 50% decrease in TPH during this time period.

Statistical analysis of the slope and intercept for the relationship between soil TPH concentrations at the 0-5 cm depth (Figure 1), where almost all of the TPH was located, revealed that degradation rates were not significantly different between treatments. This is indicated by the lack of significant differences for slope and intercept between treatments.

Soil TPH levels at 0-5 cm at 170 days after OBM application were not significantly different from the control plot for almost all of the treatments. However, OBM bulked with caliche and gypsum at a 3:1 OBM:bulking agent ratio, and gypsum (1.5:1) plus manure (PAN) were all significantly higher in TPH at day 170 compared to the control. Among treatments that received bulking agents at the 3:1 OBM:bulking agent ratio, the lime bulking agent resulted in a statistically lower soil TPH concentration at 0-5 cm after 170 days compared to caliche and gypsum (Table 3). The exact reason for this is unknown. Although all of the amendments are rich in Ca, it is unlikely that the microorganisms were initially deficient in Ca, especially since the OBM possessed appreciable Ca (Table 1). The microorganisms were also not limited by acid pH since both the OBM and the background soil possessed a pH > 7.5. One possibility is that the liming agent may have resulted in more suitable physical conditions for microbial growth due to having a much smaller particle size compared to the gypsum and caliche.

A comparison among gypsum and lime between the bulking ratio of 3:1 and 1.5:1 suggests that the increased bulking agent did not significantly improve final TPH concentrations at day 170 (0-5 cm; Table 3). However, increased addition of beef manure to 1.5xPAN levels compared to PAN application resulted in significantly lower TPH concentrations at day 170. Several studies have shown that animal manure application to soils high in TPH can improve TPH degradation due to the additional nutrients and organic matter from manure (Wellman et al., 2001; Barker et al., 1992). Barker et al. (1992) showed that addition of chemical fertilizer or composted beef manure increased TPH degradation compared to OBM applied without nutrients.

At 60 days after OBM application, subsurface TPH concentrations were significantly higher than the control plot which received no OBM (Table 2). However, TPH concentrations were extremely low and did not exceed any environmental or agronomic thresholds. In general, there was a large amount of variability in surface (0-5 cm) TPH concentrations: specifically, at least one of the replications would have extremely low TPH concentrations. Due to this high variability, the statistical model rarely indicated significant differences between the 0-5 cm depth and the two deeper depths within a treatment (Tables 2 and 3). It is not apparent if the increased subsurface TPH levels relative to the control at day 60 were due to leaching from the surface or if the values are a residual of the soil sampling process. In regard to leaching, the low molecular weight hydrocarbons such as benzene are most prone to leaching due to their relatively low octanol:water partition coefficient (Karickhoff et al., 1979). However, by day 170 all treatments were not significantly different from the control in TPH concentrations in subsoil, except for the 3:1 gypsum:OBM treatment (Table 3).

Soil pH, soluble salts, and sodium absorption ratio

Application of OBM had no impact on soil pH in this study. There were no significant differences in pH between any treatments or between the treated plots and control at any time or for any soil depth. Although the raw OBM had an elevated pH, it was not very well buffered as indicated by the CCE value (Table 1). While gypsum typically has little effect on soil pH, lime and caliche are often used to increase pH in acid soils. However, calcium carbonate minerals that are found in caliche and limestone are mostly insoluble in alkaline soils such as that used in this study (pH 7.7). Therefore it is not surprising that although the lime and caliche mixed with the OBM contained appreciable CCE (85 and 36% CCE, respectively), there was no increase in soil pH.

Soil EC is an indicator of soluble salts which can cause plant damage at excessive concentrations through increasing osmotic potential. As previously discussed, a soil EC of 4 mS cm^{-1} is considered a threshold salinity level for salt-sensitive plants, while forages that can tolerate over 7.8 mS cm^{-1} are less sensitive. Since the EC of the raw OBM exceeded the 4 mS cm^{-1} threshold, there was concern for increasing soil salinity beyond 4 mS cm^{-1}. At seven days after application of OBM, soil EC varied from 3 to 6.3 mS cm^{-1} in the 0-5 cm depth. However, due to the high mobility and solubility of the salts, EC levels quickly decreased due to downward leaching. By day 170, all treatments except for one had decreased to an EC below 4 mS cm^{-1}. Note that all treatments increased soil EC to levels greater than the control at all depths, although not always statistically significant (Table 4). Table 4 clearly shows the downward movement of the salts initially applied to the surface through OBM.

Due to the elevated Na content in the raw OBM (Table 1), there was concern regarding the potential increase in soil sodium absorption ratio (SAR) with OBM application. However, due to the relatively high Ca content of the OBM compared to Na and also from application of Ca through bulking agents, soil SAR at seven days after application was less than 2 at the 0-5 cm depth. A SAR level of 13 is considered to be a threshold at which dispersion and degradation of soil physical properties could potentially occur.

However, if an OBM sample contained extremely high levels of Na compared to Ca+Mg, then bulking the OBM with gypsum would be beneficial for preventing excessive soil SAR levels. Gypsum is a more soluble Ca source compared to lime and caliche, except in very acid soils.

Yield

Total plant biomass harvested 60 days after OBM application was not significantly impacted by OBM application or bulking agent (Figure 2). This is likely due to the fact that soil TPH levels quickly decreased below the 10,000 mg kg^{-1} concentration considered harmful to plants. Similarly, Kisic et al. (2009) found that there were no effects on crop density when applying OBM at a rate of 5,000 mg TPH kg^{-1} soil

Also, soil soluble salts (i.e. EC) was mostly below 4 mS cm^{-1} by day 60. According to Mann (2007) old world bluestem (i.e. the dominant forage at the site) is sensitive to soil salt concentrations as yield begins to decrease at soil EC concentrations less than 4 mS cm^{-1}.

CONCLUSIONS

The OBM amended to field plots in this study did not contain concentrations of heavy metals beyond typical soil concentrations. In addition, application of OBM did not result in a metals loading rate that exceeded EPA 503 regulations for biosolids. However, caution should be exercised due to the fact that the source of metals is mainly from geologic material that is drilled into. Therefore, if a region is known to naturally contain elevated concentrations of metals in the subsurface, then there would be some risk of high metal concentrations in the resulting OBM produced in that region. For example, the groundwater of central OK possesses naturally high concentrations of arsenic (Welch et al., 2000). Similarly, although the OBM used in this study had levels of NORM that is typical of non-contaminated topsoil, an OBM produced from drilling in a region with high levels of NORM in the subsurface could likewise produce an OBM sample elevated in NORM.

After seven days, the TPH applied with OBM rapidly decreased to concentrations less than sensitive environmental thresholds for residential areas. By day 170, approximately 99% of applied TPH had degraded. There was no difference in TPH degradation as a function of the type and amount of bulking agents used with the OBM. Therefore, the use of caliche, lime, or gypsum bulked with OBM at a 1.5:1 ratio (OBM:bulking agent) would suffice for achieving acceptable TPH decreases when surface applied and non-incorporated. However, if an OBM with high SAR is to be land applied, it would be more beneficial to use gypsum as a bulking agent due to the high Ca solubility.

The decrease in TPH is mainly attributed to biological degradation since OBM TPH contained only 17% of low molecular weight carbon. However, nothing is known about the potential volatilization of benzene and other low molecular weight TPH from land applied OBM. Similarly, although surface applied OBM was mostly in the 0-5 cm layer with subsurface soil TPH concentrations far below environmental thresholds, there was some significant increases in subsurface TPH compared to the control. Therefore, there is a need for future research on potential volatilization and leaching of low molecular weight TPH compounds (i.e. BTEX) from land applied OBM.

Salts applied through application of OBM leached out quickly into the subsurface. As a result of the fast rate of TPH degradation and relatively low impact on soil salinity, there were no significant differences between OBM amended soils and the control with regard to total plant biomass production. For the soil used in this study, type and amount of bulking agent used with the OBM did not have a significant impact on total plant biomass production.

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TABLES AND FIGURES

Table 1. Characterization of the oil-base mud used in the land application study. TPH; total petroleum hydrocarbons, CCE; calcium carbonate equivalent, EC; electrical conductivity. All concentrations in units of mg kg^{-1} (dry weight basis) unless noted otherwise.

Oil-base mud parameter	
TPH	152458
CCE (g 100 g^{-1})	8.4
pH	10.8
EC (mS cm^{-1})	5.90
Solids (g 100 g^{-1})	83.0
N (g 100 g^{-1})	0.089
C (g 100 g^{-1})	6.0
Na	2397
Ca	20762
Mg	6749
K	5657
S	2756
P	912
Fe	29751
Zn	91
Cu	27
Mn	501
Al	22912
Ni	26
B	69
As	0.03
Cd	0
Cr	37
Ba	1252
Pb	6.6
Cl	102847

Table 2. Average soil total petroleum hydrocarbon (TPH) concentration (mg kg^{-1}) among three different depths at 60 days after surface application of several oil-base mud (OBM) mixtures. All treatments except control initially received 8625 kg TPH ha^{-1}. Different upper case letters indicate significant differences between treatments within depth (i.e. across row) and lower case letters indicate differences between depths within treatments (i.e. within column) at P = 0.05.

	Treatment†								
Depth (cm)	C	C1	G1	G2	G2M1	G2M2	L1	L2	LSD‡
0-5	13 Ca	1729 Aa	1584 ABa	576 ABCa	819 ABCa	894.1 ABCa	863 ABCa	432 BCa	1166.4
5-10	5 Ca	23 ABa	52 Ab	44 Aa	23 ABa	36.2 ABa	49 Aa	28 ABa	32.6
10-15	8 Ca	23 ABa	39 ABb	34 Aba	23 ABa	31.0 ABa	31 ABa	59 Aa	46.9
LSD	9	2700.4	1211	1034.4	1210.0	1684.7	1271.0	689.9	

† C = control (no amendment); C1 = 3:1 caliche:OBM; G1 = 3:1 gypsum:OBM; G2 = 1.5:1 gypsum:OBM; G2M1 = 1.5:1 gypsum:OBM + cattle manure at plant available nitrogen rate; G2M2 = 1.5:1 gypsum:OBM + cattle manure at 1.5 times plant available nitrogen rate; L1 = 3:1 lime:OBM; L2 = 1.5:1 lime:OBM.

‡ Least significant difference between treatments within depth and between depths within treatments at P = 0.05.

Table 3. Average soil total petroleum hydrocarbon (TPH) concentration (mg kg^{-1}) among three different depths at 170 days after surface application of several oil-base mud (OBM) mixtures. All treatments except control initially received 8625 kg TPH ha^{-1}. Different upper case letters indicate significant differences between treatments within depth (i.e. across row) and lower case letters indicate differences between depths within treatments (i.e. within column) at P = 0.05.

	Treatment†								
Depth (cm)	C	C1	G1	G2	G2M1	G2M2	L1	L2	LSD‡
0-5	12.9 Ba	191.2 Aa	356.6 Aa	183.5 Aba	188.6 Aa	72.4 Ba	137.0 Ba	147.3 ABa	212.6
5-10	10.3 Aa	31.0 Aa	59.4 Ab	49.1 Ab	41.3 Aa	64.6 Aa	38.8 Ab	36.2 Aa	68.9
10-15	15.5 Ba	25.8 Ba	137.0 Ab	95.6 ABb	67.2 ABa	64.6 ABa	59.4 ABb	46.5 Ba	83.5
LSD	7.2	347.8	213.6	90.1	174.1	56.5	90.1	283.0	

† C = control (no amendment); C1 = 3:1 caliche:OBM; G1 = 3:1 gypsum:OBM; G2 = 1.5:1 gypsum:OBM; G2M1 = 1.5:1 gypsum:OBM + cattle manure at plant available nitrogen rate; G2M2 = 1.5:1 gypsum:OBM + cattle manure at 1.5 times plant available nitrogen rate; L1 = 3:1 lime:OBM; L2 = 1.5:1 lime:OBM.

‡ Least significant difference between treatments within depth and between depths within treatments at P = 0.05.

Table 4. Average soil electrical conductivity (EC; mS cm^{-1}) among three different depths at 170 days after surface application of several oil-base mud (OBM) mixtures. Different upper case letters indicate significant differences between treatments within depth (i.e. across row) and lower case letters indicate differences between depths within treatments (i.e. within column) at P = 0.05.

	Treatment†																
	C		C1		G1		G2		G2M1		G2M2		L1		L2		LSD‡
Depth (cm)																	
0-5	0.71	Ba	2.84	ABa	3.79	Aa	4.24	Aa	3.99	Aa	3.46	Aa	3.48	Aa	2.55	ABa	2.72
5-10	0.62	Ba	2.46	ABa	4.01	Aa	3.79	Aab	3.52	Aa	4.26	Aab	2.95	Aa	2.48	ABa	1.95
10-15	0.43	Da	0.95	CDa	3.24	Aa	2.86	ABb	2.19	ABCa	2.58	ABb	1.54	BCDb	1.83	ABCDa	1.44
LSD	2.45		2.75		1.75		1.14		2.06		1.50		0.95		1.71		

† C = control (no amendment); C1 = 3:1 caliche:OBM; G1 = 3:1 gypsum:OBM; G2 = 1.5:1 gypsum:OBM; G2M1 = 1.5:1 gypsum:OBM + cattle manure at plant available nitrogen rate; G2M2 = 1.5:1 gypsum:OBM + cattle manure at 1.5 times plant available nitrogen rate; L1 = 3:1 lime:OBM; L2 = 1.5:1 lime:OBM.

‡ Least significant difference between treatments within depth and between depths within treatments at P = 0.05.

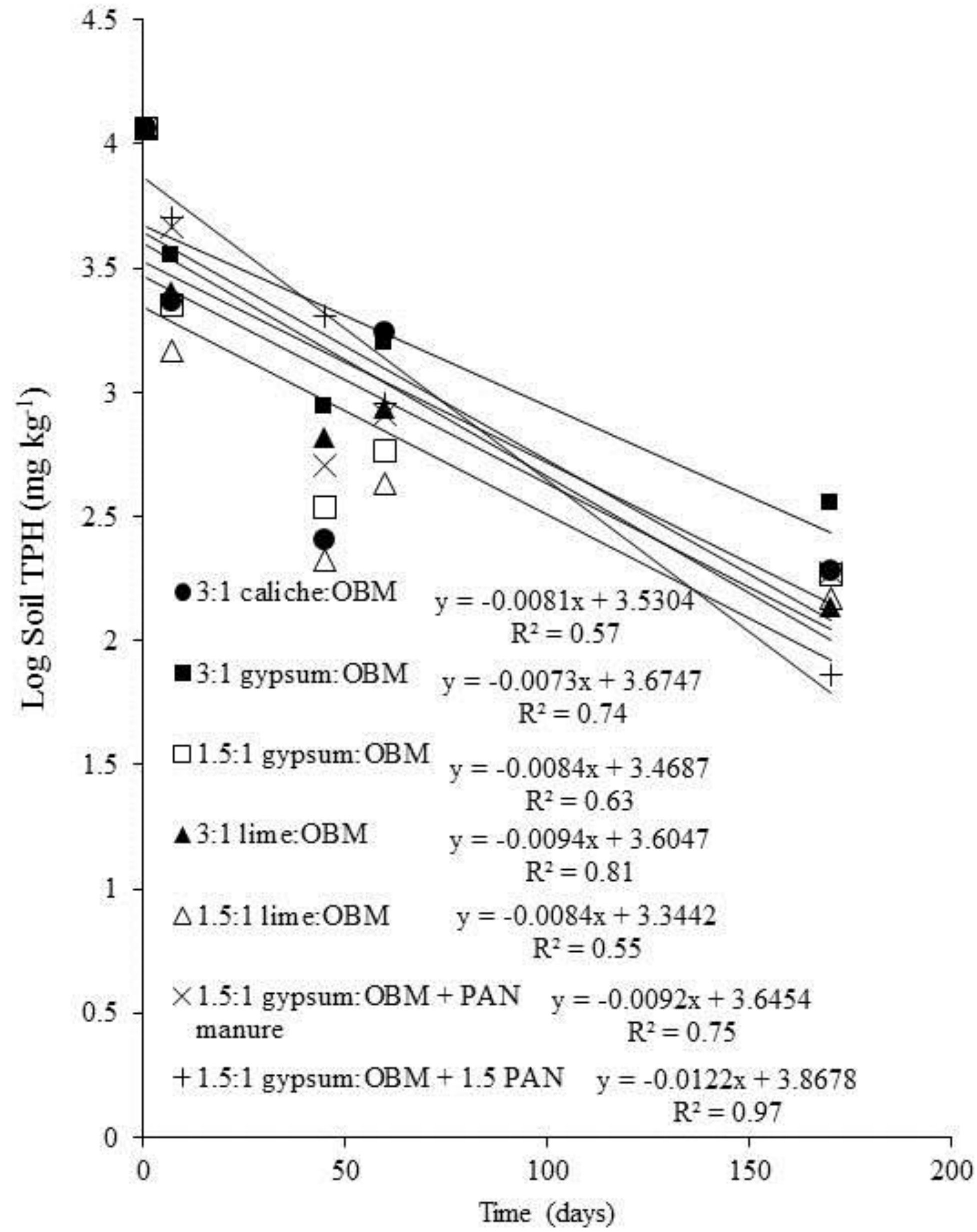

Figure 1. Changes in soil TPH (log transformed) with time at 0-5 cm depth among soils amended with oil-base mud (OBM) mixtures. No significant differences for slopes and intercepts between treatments at P = 0.05. C1 = 3:1 caliche:OBM; G1 = 3:1 gypsum:OBM; G2 = 1.5:1 gypsum:OBM; G2M1 = 1.5:1 gypsum:OBM + cattle manure at plant available nitrogen rate; G2M2 = 1.5:1 gypsum:OBM + cattle manure at 1.5 times plant available nitrogen rate; L1 = 3:1 lime:OBM; L2 = 1.5:1 lime:OBM.

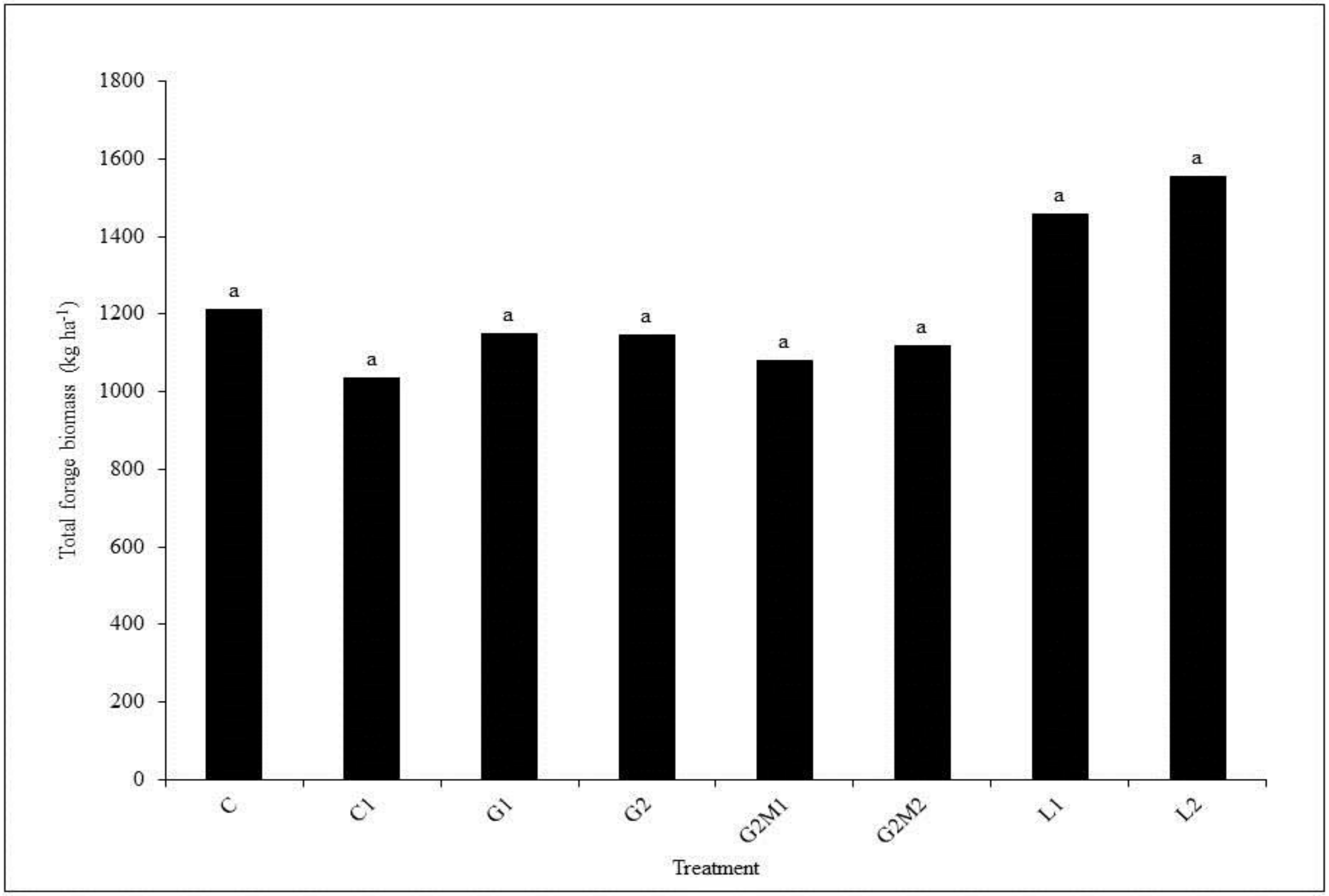

Figure 2. Total forage biomass at 60 days after surface application of several oil-base mud (OBM) mixtures. C = control (no amendment); C1 = 3:1 caliche:OBM; G1 = 3:1 gypsum:OBM; G2 = 1.5:1 gypsum:OBM; G2M1 = 1.5:1 gypsum:OBM + cattle manure at plant available nitrogen rate; G2M2 = 1.5:1 gypsum:OBM + cattle manure at 1.5 times plant available nitrogen rate; L1 = 3:1 lime:OBM; L2 = 1.5:1 lime:OBM. No significant differences between treatments at P = 0.05.

CHAPTER III

SURFACE APPLICATION OF WATER-BASE DRILLING MUD TO WINTER WHEAT

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ABSTRACT

Increased oil and gas drilling has resulted in large quantities of water base "mud" (WBM) that requires disposal. Land application of WBM to agricultural land is a common disposal technique that presents agronomic and environmental challenges since the material is rich in total soluble salts (TSS) that can be hazardous to crops and the soil. The objective of this study was to determine the impact of WBM application rate on salt accumulation and leaching in the soil, and the impact of application timing and rate on wheat production. WBM was characterized for pH, EC, and TSS. A field study was conducted where WBM was applied once, at varying times (Oct, Dec, Jan, Feb, and March) at a 1.0X and 0.66X rate. The 1.0X and 0.66X rate were equivalent to soil TSS concentrations of 6,721 kg ha^{-1} and 4,480 kg ha^{-1}, respectively. Soil samples were taken at 0, 30, and 90 days after application and on August 28th (post-harvest) for evaluating electrical conductivity (EC), pH, and sodium adsorption ratio (SAR). WBM rates had no effect on soil pH. By August, soil EC had decreased below 4 mS cm^{-1} at the 0-15 cm depth. Soil SAR increased at every sampling day. WBM application date and rate had no

significant effect on wheat yield even though large differences were seen in March when compared to control. If WBM is applied at the proper time and adequate rainfall is received, salt accumulation in the soil should not pose a significant threat to wheat production.

INTRODUCTION

The oil and natural gas drilling industry uses water-based drilling mud (WBM) to help lubricate and cool the drilling bit, seal off porous geologic formations, balance subsurface and formation pressures required for the prevention of well blowouts, and carry geologic drill cuttings from the bottom of the well up to the surface (Ukeles and Grinbaum, 2004). Water-based mud is generally used during shallow vertical drilling operations. The salinity and chemical composition of WBM varies greatly depending on well depth, geography, and the geologic formation that is being drilled through. On average, WBM's are characterized by having pH values that range from 7-13, high total soluble salts (TSS), and high sodium concentrations. Typically, WBM consists of colloidal clays (bentonite), potassium chloride, sodium dichromate, sodium hydroxide, lignite, barium sulfate, mica, ground nut shells, polymers, and numerous other additives depending on the exact chemistry needed for the particular well (Moseley, 1983). Typical diameter of drilling pipe is 10cm with the bottom of the well borehole diameter reaching up to 20cm. Drilling operations can last anywhere from a few days to more than a year (Ukeles and Grinbaum, 2004). On average, the amount of WBM needed to drill a well is equal to three times the total volume of the well (U.S. Army Corps of Engineers, 2001). After the well has been completed, spent drilling fluids and geologic formation cuttings are allowed to settle in a holding pit before disposal

Recently, there has been an explosion of oil and natural gas exploration across the United States. Newer technologies such as hydrofracking and horizontal drilling have allowed more access to harder to reach oil and natural gas reserves. A big part of United States oil and natural gas exploration is occurring in the state of Oklahoma. In Oklahoma from the year 2000 through 2011 on average there were 2500 new wells drilled every year. These wells consisted of oil, natural gas, and dry holes. From 2009 to 2011, drilling permits and average monthly rigs in Oklahoma increased from 2,500 to 3,732 and 94 to 180, respectively (Oklahoma Corporation Commission, 2011).

This escalation in oil and natural gas drilling ultimately leads to an increase in WBM waste. These large amounts of WBM waste need to be disposed of properly. Potential hazards to soil and plants can occur from disposal of WBM due to the high total soluble salts and sodium concentrations if application is done improperly. In order to avoid these potential hazards proper WBM disposal techniques must be selected. There are several methods of WBM disposal which include, onsite burial, storage in hazardous waste landfills, underground injection, and land application. Depending upon the circumstances, one or more of these disposal techniques will be used. In Oklahoma, disposal of WBM waste occurs primarily by land application. Land application of WBM in Oklahoma involves the spreading of the wastes at a predetermined rate based upon the conditions of the soil and composition of the WBM. Land application of WBM is monitored by the Oklahoma Corporation Commission (OCC). The rules are specifically stated in the Oklahoma administrative code and register in Title 165:10-7-19.

Numerous studies have been carried out looking at the effects of land applying saline and saline-sodic water for irrigation. However, there is a scarcity of research looking at the effects WBM application has on wheat production and soil chemical properties. Addressing these impacts in a meaningful way will require continued research efforts to understand the immediate and long term effects of land applying WBM. Thus, the objectives of this study were to (i) monitor the impact of WBM application rate on salt accumulation and leaching in the soil over time, and (ii) determine the impact of application timing and rate on wheat production.

MATERIALS AND METHODS

The location of this field trial was in Lahoma, Oklahoma. The study was conducted on the Billings wheat variety which was located on a Grant silt loam (fine-silty, mixed, superactive, thermic Udic Argiustolls). The experimental design for soil analysis consisted of a split-split plot that had eleven treatments replicated three times. The main plot was WBM application date and rate; the two splits were sampling day and soil depth. The experimental design for wheat yield analysis consisted of a randomized complete block with three replications and eleven treatments. Each plot was 3.05 by 6.10 m. Composite soil samples were taken from all three reps before WBM was applied. Prior to planting, plots were fertilized with DAP $(NH_4)_2HPO_4$ at 112 kg ha^{-1} and disked 10.2 cm deep and then cultivated. Billings' wheat was planted two inches deep into moisture on October 18th, 2012 at a planting rate of 78 kg ha^{-1}. The wheat plots received an additional 34 kg N ha^{-1} in February. The application of WBM to the wheat plots started on October 16th, 2012. The mud had a total soluble salts (TSS) concentration of 155,541 mg L^{-1} and was applied at two different rates using a 1/10th horsepower electric water pump. WBM volume was measured with a Seametrics (MJ-Series) water meter. The 1X rate brought the soil TSS concentration to the maximum of 6,721 kg ha^{-1}. The 0.66X rate brought the soil TSS concentration to 4,480 kg ha^{-1}. After the October 16th application, the plots were cultivated again to mix the WBM within the soil prior to planting. Application of the WBM was continued in December, January, February, and March on different plots that had not previously received WBM. Once a plot had received the WBM application, composite plot soil samples were taken at 0 (immediately after application), 30, and 90 days after application, at depths of 0-7.5, and 7.5-15 cm. In addition, soils were sampled several months after wheat harvest on August 27th, 2013 (post-harvest). All soil samples were ground and passed through a 2 mm sieve. 125 grams of each soil sample was used in a modified saturated paste extraction. Electrical conductivity and pH were determined on all extracts. Random soil samples were duplicated and check soils were used to assure accuracy and precision of the results. Extracts were analyzed for Na, Ca, Mg, K, SO_4, B, P, Fe, Zn, Cu, Mn, Al, Mo, As, Cr, Cd, and Pb by ICP-AES. Cl extract concentrations were measured via Lachat-FIA. All wheat plots were harvested for grain yield by a combine on June 20th, 2013.

Statistics

ANOVA methods were utilized in PROC GLM (SAS Institute, 2011) to analyze the effect of WBM application rates and timing on soil chemical properties and wheat production. When the main effects or interactions of WBM application rates and timing were significant, treatment means were separated using pairwise comparisons via Duncan's multiple range test. Statistical decisions were made at $\alpha=0.05$.The data analysis for this paper was computed using SAS software (Copyright, SAS Institute Inc. SAS and all other SAS Institute Inc. product or service names are registered trademarks or trademarks of SAS Institute Inc., Cary, NC, USA).

RESULTS AND DISCUSSION

Background Soil Properties

The soil utilized for this experiment was ideal for determining the effects of WBM application due to the relatively low soil TSS concentration (Table 1) when compared to the OCC rules which are stated in the Oklahoma administrative code and register, Title 165:10-7-19 which allows the soil to receive a maximum amount of WBM that would result in a TSS concentration of 6,700 kg ha^{-1}. The low initial soil TSS concentration allowed for higher loading rates of the WBM to be applied which would simulate a worst case scenario were a soil would go from relatively low concentrations of soluble salts in the soil to the maximum amount of soluble salts allowed. Based upon Oklahoma soil nutrient recommendations that were established by Zhang and Raun (2006), soil test P and K levels for wheat were 92 and 100% sufficient, respectively.

Water-Based Mud Properties

The initial chemical analysis of the liquid and non-dissolved portion of WBM used in the Lahoma study is listed in Table 2. The liquid fraction of the WBM had an EC of 233 mS cm^{-1} and SAR of 344 both of which are considered extremely high in the context of irrigation water. Constant use of irrigation water with an EC of 3.0 mS cm^{-1} can cause severe salinity problems in the soil (Essington, 2004). It is important to keep in mind that the land application of WBM is a one-time event. The dominant cations in the liquid portion of the WBM were Na, Ca, Mg, and K with Na being the greatest. The dominant anions in the liquid portion of the WBM were chloride and sulfate with chloride making the biggest contribution. The dominant cations in the non-dissolved portion of the WBM were Na, Ca, Mg, K, Fe, and Al with Na having the largest influence. The dominant cations and anions in the WBM often depend on the type of geologic material that was drilled through during oil and natural gas exploration.

Soil pH

Table 3 shows that there were no significant main effects or interactions in soil pH due to the application of WBM at the 1.0X and 0.66X rate across the five application dates (Oct, Dec, Jan, Feb, and March). However there was a significant difference in soil pH between the 0-7.5 cm depth (6.90) and the 7.5-15 cm depth (7.21). The difference in pH was likely due to natural variances in soil horizon chemistry.

Soil Electrical Conductivity

The main effects of rate, day, and depth and the interactions between application date*rate, application date*depth, rate*depth, application date*day, depth*day, application date*rate*depth, application date*depth*day, rate*depth*day were significant at ($\alpha = 0.05$) for soil EC and are shown in Table 3 along with a complete list of main effects and interactions.

The two-way interaction of depth by day was significant ($Pr \leq 0.05$) and is shown in further detail in Table 4. There is a significant decrease in soil EC across all sampling days at the 0-7.5 cm depth. Soil EC is significantly higher in the 0-7.5 cm depth at all soil sampling days when compared to the 7.5-15 cm depth except for in August. There is also a significant increase in soil EC in the 7.5-15 cm depth over all sampling days except for August. These results can be explained by examining rainfall amounts (Table 5) that occurred since time of WBM application. The general decrease in soil EC in the 0-7.5 cm depth over time and the increase soil EC in the 7.5-15 cm depth occurred because of the degree of cumulative rainfall with time. There is no significant difference in soil EC between the 0-7.5 cm depth and the 7.5-15 cm depth at the August sampling day because the soil had sufficient time and rainfall (56.54-68.98 cm) for the soil EC to become equilibrated between both depths due to salt leaching. Wheat is moderately tolerant to soil salinity and can grow in soils with an EC around 6 mS cm^{-1} in the top 15 cm of soil (Zhang and Raun, 2006; Maas, 1990). Although all surface soil EC values were above 6 mS cm^{-1} except for the August sampling date, the average of the two depths must be taken into account. When the soil EC is averaged over both depths at each sampling day the EC approaches the suitable level for wheat.

The three-way interaction of application date by depth by day was significant ($Pr \leq 0.05$) and is listed in Table 6. Immediately after application (day 0) there are no significant differences between the five application dates within the 0-7.5 cm depth since no rainfall had yet occurred to redistribute the salts. There are significant differences in soil EC between the two depths at each application date. Given that it is day 0 (after application) and no rainfall has occurred, it is expected that the EC will be greater at the 0-7.5 cm depth than 7.5 to 15 cm. At day 30 there are significant differences in soil EC between the application dates at the 0-7.5 cm and 7.5-15 cm. These results can be explained by using the results in Table 5. By day 30 the December and January application dates only received 0.71 and 1.07 cm of rainfall, respectively. This was not enough rainfall to leach soluble salts out of the top depth and equilibrate the soil EC between the two depths. At day 30 the February and March application dates received a total of 8.53 and 8.05 cm of rainfall, respectively. This was enough rainfall to leach some of the soluble salts from the top depth into the bottom depth and allow soil EC to equilibrate between the two depths. At day 90 the October application date soil EC is significantly different than the other four application dates soil EC at both depths. This can also be explained using rainfall data from (Table 5). At day 90 the October application date received a total rainfall amount of 2.84 cm. This was not enough rainfall to significantly decrease the EC in the top depth and increase the EC in the bottom depth. This is why plots receiving WBM in October possessed a significantly higher EC at 0-7.5 cm than all other application dates at day 90 while soil EC at the 7.5-15 cm depth was still significantly lower than all other application dates. At day 90 the December, January, February, and March application dates had accumulated rainfall amounts of 10.67, 17.30, 20.04, and 24.69 cm, respectively. These application dates had enough rainfall for soluble salts to leach from the top depth into the bottom depth, thereby equilibrating soil EC between the two depths. By the time post-harvest soil samples were taken, soil EC at all application dates were not significantly different between application dates or depths. At post-harvest, the October, December, January, February, and March application dates had accumulated rainfall amounts of 68.98, 67.67, 66.14, 65.07, and 56.54 cm, respectively. By this time there was enough rainfall for soluble salts to leach from the top depth, into the bottom depth, and out of the bottom depth further downward into the soil profile. At post-harvest the soil EC averaged over depth at each application date was not only lower than the 6 mS cm^{-1} EC requirement for wheat but was lower than the 4 mS cm^{-1} EC requirement for salt sensitive plants stated by (Zhang and Raun, 2006; Maas, 1990).

Table 7 compares soil EC between application dates within each application rate and depth at day 90 and the post-harvest sampling day. In addition Table 7 also allows comparison between application rates within each application date and depth at day 90 and post-harvest sampling events. At day 90 at the 0-7.5 cm depth the soil EC from the October application date at the 0.66X rate is higher than all other application dates and is significantly higher than EC at the January application date. Similarly, soil EC at day 90 within the 0-7.5 cm depth was significantly highest for the October application date at the 1.0X rate compared to all other application dates. This can be explained with rainfall data from Table 5. Soil EC at the October application date is higher than all other application dates at both rates because at day 90 the October application date had only received 2.84 cm of rainfall whereas the other four treatments rainfall amounts ranged from 10.67 to 24.69 cm which led to more leaching and lower soil EC. The day 90, 0-7.5 cm depth soil EC was never significantly different between the application rates at any application date. The 90 day, 7.5-15 cm depth soil EC for the 0.66X rate October application was significantly lower than the December and February application dates, but was not significantly different than the remaining two application dates, although it was lower than all application dates. However, the 1.0X rate applied in October was significantly lower than all other application dates. Again, this was due to the lack of rainfall that was received within 90 days of the October application (Table 5). This lack of rainfall for October would lead to less leaching of soluble salts from the top depth into the bottom depth when compared to the other four application dates which had received more rainfall by day 90. Another probable cause of the lower soil EC values for the October application date would be due to the fact that the plots were cultivated after initial WBM application which allowed for the dilution of the soil EC. The 7.5-15 cm depth also showed that there was no significant difference between the 0.66X and 1.0X rate at any application date.

At post-harvest the 0-7.5 cm depth soil EC values amongst the application dates were mostly not significantly different at the 0.66X rate and the 1.0X rate. This was due to the fact all of the application dates had received 56.54-68.98 cm of rainfall by the post-harvest sampling time. Again, there were very few significant differences in soil EC values between the two rates at each application date. For the 7.5-15 cm depth, the soil EC was not significantly different between application dates at either rate which can be explained by examining rainfall accumulation since application. There were significant differences (although small) for soil EC between application rates, where the 1.0X rate for the December, January, and March application dates at the 7.5-15 cm depth was significantly higher than the 0.66X rate.

Soil Sodium Adsorption Ratio

The main effects of application date, rate, day, and depth and the interactions of rate*depth, and application date*depth*day were significant at ($\alpha = 0.05$) for soil SAR and are shown in Table 3 along with the full list of main effects and interactions.

The main effect of day on soil SAR was significant ($Pr \leq 0.05$) and is shown in Table 8. Day 0 had the lowest soil SAR (6.43) and soil SAR values continued to significantly increase with sampling day; by August the SAR had reached 11.12. This can be explained by examining the solids characterization of WBM (Table 2). The solids portion of the WBM had a high concentration of undissolved Na (93963 mg kg^{-1}) which was equivalent to applying 64 and 42.6 kg Na ha^{-1} at the 1.0X and 0.66X rate, respectively. With time, the amount of rainfall at each sampling date also increased which led to the dissolution/desorption of the solid-phase Na and an increase in saturated-paste extractable Na concentrations. Another likely cause of the increase in SAR over time could be due to the extremely high concentrations of Na in the liquid portion of the WBM that when applied exceeded the soils cation exchange capacity (CEC) and leached out the Mg and Ca cations and replaced them with Na. Overtime, plant uptake of water allowed Na that had leached to come back to the surface soil and increase soil SAR values.

The main effect of application date on soil SAR was significant ($Pr \leq 0.05$) and is shown in Table 9. The control (no WBM application) is significantly lower than all other application dates as expected. Statistically, soil SAR for plots receiving WBM in October, December, January, and March were not significantly different. Rainfall data from (Table 5) shows that these four application dates had an average accumulation of rainfall after application that were similar and would allow leaching of soluble salts to occur resulting in similar soil SAR values. The anomaly is the soil SAR value for the February application date (10.62) which is higher than all of the other application dates and is significantly higher than the October and March application date even though the average accumulation of rainfall at day 30 and 90 was higher than October, December, and January application dates. Difficulty with use of the application pump may have allowed a greater amount of non-dissolved solids to be applied at the February date.

The two-way interaction effect of rate by depth on soil SAR was significant ($Pr \leq 0.05$) and is shown in Table 10. At the 0-7.5 cm depth the control, 0.66X rate, and the 1.0X rate were all significantly different from each other. As expected the control had the lowest soil SAR, followed by the 0.66X rate, with the 1.0X rate having the highest soil SAR. The 7.5-15 cm depth followed the same trend. The control, 0.66X rate, and the 1.0X rate all had significantly different soil SAR values between depths. Differences in SAR between depths for the control can be explained by soil horizon variation in soil chemical properties. Soil SAR at both the 0.66X and 1.0X rate were significantly higher in the 0-7.5 cm compared to the 7.5-15 cm depth because an appreciable portion of the WBM SAR was found in the non-dissolved form which is less able to leach initially.

The two-way interaction effect of application date by rate on soil SAR was significant ($Pr \leq 0.05$) and is shown in Table 11. Soil SAR values at the 0.66X rate were significantly lower for October, January, and March application compared to the December and

February application date. Both January and March had higher accumulations of rainfall by day 30 and 90 (Table 5) than did December while the October application had the longest amount of time and the highest overall amount of accumulated rainfall compared to December and February. Keep in mind that the reason for the high February soil SAR value could potentially be because of disproportionally higher application of WBM solids. Similar trends were noted for the 1X application rate.

Wheat Yield

Table 12 shows that there were no significant main effects or interactions in wheat yield ($\alpha = 0.05$) when analyzing the effects of WBM application dates and rates. However, application date was close to being significant ($P = 0.0592$). Although rate was not significant Figure 1 shows that rate still had an effect on wheat yield; the control had the highest yield (2962 kg ha^{-1}) followed by the 0.66X rate (2584 kg ha^{-1}) and the lowest yield was from the 1.0X rate (2335 kg ha^{-1}). Figure 2 shows the non-significant differences in wheat yield (kg ha^{-1}) in response to WBM application dates. In order to explain Figure 2 the rainfall data from Table 5 must be examined and the figure needs to be broken down into four groups: group 1 = control, group 2 = October, December, and January application dates, group 3 = February application date, and group 4 = the March application. The control did not receive a WBM application and therefore had the highest yield at (2962 kg ha^{-1}). Out of the four groups, group 2 had the second lowest yield which can be explained with rainfall data (Table 5). The October, December, and January application yields were similar because the average accumulated rainfall at day 30 and 90 was 1.00 and 10.27cm compared to accumulated rainfall after 30 and 90 days after the

February application which averaged 8.53 and 20.04 cm, respectively. Group 3 (February application) had the second highest yield (2830 kg ha^{-1}) due to the fact that the accumulation of rainfall at 30 and 90 days after application was higher than all other application dates except for the March application date. The majority of the wheat roots are found in the top 15 cm of soil (Subbiah et al., 1968). In general, yield will suffer least when the time of root exposure to a saline environment is minimized. Group 4 (March application) had the lowest yield out of all the application dates. At $\alpha = 0.1$ the Dunnett's multiple comparison test (1955) showed that the March application wheat yield was significantly lower than the control even though the accumulated rainfall for March application at day 30 and 90 was 8.05 and 24.69 cm, respectively. This can be explained by considering the physiological growth stage at the time of application (Large, 1954). In March the wheat was at Feekes stage 5 and 6 and was more mature than the wheat at the earlier applications. As wheat progresses further into maturity a negative effect (salt burn) of WBM application is seen when it is applied directly to the wheat tissue as opposed to the initial impact WBM application can have on wheat emergence when WBM is applied mostly to the soil. Although differences in wheat yield were observed, none, were significant at $\alpha = 0.05$. This can potentially be due to the fact that wheat is moderately tolerant to soil salinity (Zhang and Raun, 2006; Maas, 1990) and by day 90 the average soil EC over the 0-15 cm depth (Table 4) was close to the 6 mS cm^{-1} EC threshold for wheat. Another factor that could describe why WBM application had no significant effect on wheat yield could be that average soil SAR across all sampling days never reached sodic levels (15; Lauchli and Grattan, 1990)

CONCLUSION

The WBM used in this study had no significant effects on soil pH. Initial increases in soil EC from WBM application were dramatic. However, soil EC levels greatly decreased with rainfall. Soil EC values measured at 30 and 90 days after application depended on the application date (i.e. cumulative rainfall after application). At post-harvest, there were no differences in soil EC between application date, rate, or soil depths. This shows the importance of rainfall accumulation totals on the leaching of soluble salts in the soil profile. Depending on application date and the time of sampling, the 1.0X WBM application rate resulted in greater soil EC values than 0.66X application rate. Water-based mud application rate and date did have significant effects on soil SAR when comparing SAR values at depth and over time. Soil SAR values significantly increased with time after application via dissolution and release of non-dissolved sodium with rainfall; higher sodium loading at the 1.0X application rate led to soil SAR values that were consistently higher than the 0.66X rate. At the post-harvest soil sampling date the average EC and SAR at the 0-15 cm depth were 3.33 mS cm^{-1} and 11.12, respectively and were below the saline (4 mS cm^{-1}) and sodic soil SAR thresholds (15). By day 90, an average of 15 cm of cumulative rainfall was required to decrease the 0-7.5 cm depth EC for all application dates by 5 mS cm^{-1}. No significant effects on wheat yield were observed due to WBM application rate or application date. Although, an agronomic effect on wheat yield was seen at the March application date due to the advanced maturity of the wheat being more susceptible to salt burn. For the soil used in this study at Lahoma, Oklahoma the range of environmental consequences and changes in soil chemical properties and crop yield depend primarily on the chemical characteristics of the WBM (pH, EC, SAR, and Na loading rate), crops grown, and rainfall. Damage to wheat can be minimized by applying WBM at a time when appreciable rainfall is expected, thereby quickly moving soluble salts out of the root zone. Further studies should be conducted to quantify the long term effects of WBM applications to the soil by monitoring salt leaching deeper within the soil profile and determine the amount of time required for complete dissolution of the non-dissolved sodium and an eventual decrease in soil SAR values.

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TABLES AND FIGURES

Table 1. Background chemical properties at the 0-15 cm depth for the soils that received water base mud (WBM) in the Lahoma study. Except when noted otherwise, all values determined on a saturated paste extract.

pH	6.9
Electrical Conductivity (mS cm^{-1})	0.57
Total Soluble Salts (mg L^{-1})	483.0
KCl extractable N-NO_3^- (kg ha^{-1})	12.2
Mehlich-3 Phosphorus (kg ha^{-1})	50.3
Mehlich-3 Potassium (kg ha^{-1})	446.5
Na (mg L^{-1})	5.2
Ca (mg L^{-1})	88.6
Mg (mg L^{-1})	26.0
K (mg L^{-1})	4.0
Cl^- (mg L^{-1})	16.8
SO_4^{-2} (mg L^{-1})	20.2
B (mg L^{-1})	0.0
HCO_3^- (mg L^{-1})	319.1
CO_3^{-2} (mg L^{-1})	2.8

Table 2. Characterization of the liquid and non-dissolved solids (NDS) portion of water based mud (WBM) surface applied to wheat at the Lahoma study.

WBM Liquid Analysis		WBM Non-dissolved Solid Analysis	
EC (mS cm^{-1})	233	% NDS	1.8
TSS (mg L^{-1})	155,541	Na (mg kg^{-1})	93963.0
pH	7.1	Ca (mg kg^{-1})	15385.3
SAR	344	Mg (mg kg^{-1})	29321.6
Total alk. (mg L^{-1})	85	K (mg kg^{-1})	9565.8
Na (mg L^{-1})	59082.6	S-SO$_4^{-2}$ (mg kg^{-1})	2887.0
Ca (mg L^{-1})	2053.1	B (mg kg^{-1})	103.9
Mg (mg L^{-1})	113.1	P (mg kg^{-1})	350.3
K (mg L^{-1})	174.4	Fe (mg kg^{-1})	26057.3
S-SO$_4^{-2}$ (mg L^{-1})	4908.9	Zn (mg kg^{-1})	84.0
B (mg L^{-1})	0.6	Cu (mg kg^{-1})	22.3
P (mg L^{-1})	0.1	Mn (mg kg^{-1})	248.8
Fe (mg L^{-1})	0	Al (mg kg^{-1})	30779.7
Zn (mg L^{-1})	0	As (mg kg^{-1})	0.0
Cu (mg L^{-1})	0	Cr (mg kg^{-1})	33.5
Mn (mg L^{-1})	0.1	Ba (mg kg^{-1})	182.4
Al (mg L^{-1})	0	Pb (mg kg^{-1})	0.0
As (mg L^{-1})	0	Co (mg kg^{-1})	0.0
Cr (mg L^{-1})	0	Cd (mg kg^{-1})	0.0
Ba (mg L^{-1})	0.1	Ni (mg kg^{-1})	22.4
Pb (mg L^{-1})	0		
Cl (mg L^{-1})	89123.0		
NO$_3$-N	0.3		

Table 3. Analysis of variance (ANOVA) model results for the impact of water base mud (WBM) application date, rate, and sampling time and depth on soil electrical conductivity (EC), pH, and sodium absorption ratio (SAR) for the Lahoma study. P = 0.05.

	EC		SAR		pH	
Variable	DF	Pr > F	DF	Pr > F	DF	Pr > F
appdate	4	0.1476	4	0.0113	4	0.2958
rate	1	<.0001	1	<.0001	1	0.911
rep	2	0.0162	2	0.0838	2	0.0944
day	3	<.0001	3	<.0001	2	0.7794
depth	1	<.0001	1	<.0001	1	<0.0001
appdate*rate	4	0.2434	4	0.2872	4	0.488
appdate*depth	4	<.0001	4	0.3321	4	0.1069
rate*depth	1	<.0001	1	0.0065	1	0.1616
appdate*day	11	0.0002	11	0.0796	7	0.1945
rate*day	3	0.1147	3	0.5749	2	0.0732
depth*day	3	<.0001	3	0.863	2	0.0162
appdate*rate*depth	4	0.0221	4	0.6836	4	0.6359
appdate*rate*day	11	0.569	11	0.9324	7	0.9213
appdate*depth*day	11	<.0001	11	0.0225	7	0.0119
rate*depth*day	3	0.0018	3	0.8657	2	0.9384
appdate*rate*depth*day	11	0.0773	11	0.7616	7	0.562

Table 4. Mean soil EC (mS cm $^{-1}$) averaged across application date and rate at each soil sampling day and depth. Water-base mud was applied to winter wheat in Oct, Dec, Jan, Feb, and March at a 1.0X and 0.66X rate that was equivalent to 6,721 and 4,480 kg TSS ha^{-1}soil, respectively. Soils were sampled at 0 (right after application), 30, 90 days after application, and on August 28th, 2013 (Post-harvest). Uppercase letters represent mean separation between all days within depth. Lowercase letters represent mean separation between depths within day. P = 0.05.

Depth (cm)	Day 0	Day 30	Day 90	Post-harvest
0-7.5	12.11Aa	11.48Aa	7.64Ba	3.45Ca
7.5-15	1.29Cb	2.99Bb	4.56Ab	3.20Ba

Table 5. Cumulative rainfall totals (cm) for each WBM application date from the time of application to 30 and 90 days after application, to harvest, and on August 28th, 2013 (Post-harvest) when soil samples were taken for the Lahoma study.

Year	App date	Day 30	Day 90	Harvest	Post-harvest
2012	16-Oct	1.24	2.84	37.13	68.98
2012	6-Dec	0.71	10.67	35.81	67.67
2013	14-Jan	1.07	17.3	34.29	66.14
2013	15-Feb	8.53	20.04	33.22	65.07
2013	20-Mar	8.05	24.69	24.69	56.54

Table 6. Mean soil EC (mS cm^{-1}) averaged across application rate and compared between application dates and depths for each sampling day. Water-base mud was applied to winter wheat in Oct, Dec, Jan, Feb, and March at a 1.0X and 0.66X rate that was equivalent to 6,721 and 4,480 kg TSS ha^{-1}soil, respectively. Soil sampling occurred at 0, 30, and 90 days after application, and at August 28th, 2013 (Post-harvest). NA = not available. Day 0 indicates values immediately after water base mud application. Control plots received no application. Uppercase letters represent mean separation between application dates at each depth within each day. Lowercase letters represent mean separation between depths within application date and each day. P = 0.05.

			Day 0			
Depth (cm)	Control	Oct	Dec	Jan	Feb	Mar
0-7.5	0.90Ba	11.17Aa	14.18Aa	12.87Aa	15.35Aa	12.58Aa
7.5-15	0.57Ca	1.28ABCb	1.97Ab	1.15BCb	1.43ABb	1.00BCb
			Day 30			
Depth (cm)	Control	Oct	Dec	Jan	Feb	Mar
0-7.5	NA	NA	17.67Aa	15.65Aa	6.98Ba	5.62Ba
7.5-15	NA	NA	1.07Bb	1.37Bb	5.40Aa	4.13Aa
			Day 90			
Depth (cm)	Control	Oct	Dec	Jan	Feb	Mar
0-7.5	1.03Ca	14.75Aa	7.37Ba	5.85Ba	6.90Ba	6.63Ba
7.5-15	0.57Bb	2.28Bb	5.77Aa	6.07Aa	5.97Aa	4.72Aa
			Post-harvest			
Depth (cm)	Control	Oct	Dec	Jan	Feb	Mar
0-7.5	0.63Ba	3.13Aa	4.10Aa	4.37Aa	4.02Aa	3.04Aa
7.5-15	0.59Ba	3.71Aa	3.71Aa	3.54Aa	3.59Aa	3.04Aa

Table 7. Mean soil EC (mS cm^{-1}) at 90 days after application, and Post-harvest for application of water-base drilling mud on winter wheat with applications in October, December, January, February, and March, averaged across the 1.0X and 0.66X rate that was equivalent to 6,721 and 4,480 kg TSS ha^{-1}soil, respectively. Uppercase letters represent mean separation between application dates within rate, depth and sampling day. Lowercase letters represent mean separation between rates within each application date, depth and sampling day. Statistical decisions were made at P = 0.1.

Day 90: 0-7.5 cm

Rate	Oct	Dec	Jan	Feb	Mar
0.66X	11.10Aa	6.83ABa	4.23Bb	5.97ABa	5.13ABb
1.0X	18.40Aa	7.90Ba	7.47Ba	7.83Ba	8.13Ba

Day 90: 7.5-15 cm

Rate	Oct	Dec	Jan	Feb	Mar
0.66X	1.67Ba	5.47Aa	4.27ABb	5.90Aa	3.47ABa
1.0X	2.90Ba	6.07Aa	7.87Aa	6.03Aa	5.97Aa

Post-harvest: 0-7.5 cm

Rate	Oct	Dec	Jan	Feb	Mar
0.66X	2.26Ba	2.92ABb	2.27Bb	3.96Aa	2.73ABa
1.0X	4.01BCa	5.28ABa	6.48Aa	4.09BCa	3.35Ca

Post-harvest: 7.5-15 cm

Rate	Oct	Dec	Jan	Feb	Mar
0.66X	2.85Aa	2.99Ab	2.17Ab	3.00Aa	2.31Ab
1.0X	4.00Aa	4.44Aa	4.91Aa	4.18Aa	3.77Aa

Table 8. Mean soil sodium absorption ratio (SAR) averaged across application rate, date, and depth comparing each soil sampling time. Water-base mud was applied to winter wheat in Oct, Dec, Jan, Feb, and March at a 1.0X and 0.66X rate that was equivalent to 6,721 and 4,480 kg TSS ha^{-1}soil, respectively. Soil sampling occurred at 0 (immediately after), 30, and 90 days after application, and on August 28th, 2013 (Post-harvest) at depths of 0-7.5 and 7.5-15 cm. Uppercase letters represent mean separation of soil SAR at each individual soil sampling day. Statistical decisions were made at P = 0.05

Soil SAR at each Sampling Time			
Day 0	Day 30	Day 90	Post-harvest
6.43D	7.88C	8.73B	11.12A

Table 9. Mean soil sodium absorption ratio (SAR) by application date. Water-base mud was applied to winter wheat in Oct, Dec, Jan, Feb, and March at a 1.0X and 0.66X rate that was equivalent to 6,721 and 4,480 kg TSS ha^{-1}soil, respectively. Soil sampling occurred at 0 (immediately after), 30, and 90 days after application, and on August 28th, 2013 (Post-harvest) at depths of 0-7.5 and 7.5-15 cm. SAR values are averaged across application rate, depth, and soil sampling time comparing each application date. Uppercase letters represent mean separation of soil SAR at each individual application date. Statistical decisions were made at P = 0.05

Mean Soil SAR at each Application Date					
Con	Oct	Dec	Jan	Feb	March
1.11C	8.04B	9.78AB	9.75AB	10.62A	7.42B

Table 10. Mean soil sodium absorption ratio (SAR) values averaged across application date and sampling time at each application rate and depth. Water-base mud was applied to winter wheat in Oct, Dec, Jan, Feb, and March at a 1.0X and 0.66X rate that was equivalent to 6,721 and 4,480 kg TSS ha^{-1}soil, respectively. Soil sampling occurred at 0 (immediately after), 30, and 90 days after application, and on August 28th, 2013 (Post-harvest) at depths of 0-7.5 and 7.5-15 cm. Uppercase letters represent mean separation of all application rates within depth. Lowercase letters represent mean separation of all depths within application rate. Statistical decisions were made at P = 0.05.

Depth (cm)	Con (0X)	0.66X Rate	1X Rate
0-7.5	0.94Cb	11.64Ba	16.50Aa
7.5-15	1.27Ba	3.64Ab	4.93Ab

Table 11. Mean soil sodium absorption ratio (SAR) values averaged across sampling time and depth at each application date and rate. Water-base mud was applied to winter wheat in Oct, Dec, Jan, Feb, and March at a 1.0X and 0.66X rate that was equivalent to 6,721 and 4,480 kg TSS ha^{-1}soil, respectively. Soil sampling occurred at 0 (immediately after), 30, and 90 days after application, and on August 28th, 2013 (Post-harvest) at depths of 0-7.5 and 7.5-15 cm. Uppercase letters represent mean separation of all application dates within application rate. Lowercase letters represent mean separation of all application rates within application dates. Statistical decisions were made at P = 0.05.

Rate	Oct	Dec	Jan	Feb	March
0.66X	6.64Bb	8.74Ab	6.89Bb	9.17Ab	6.51Bb
1.0X	9.43DCa	10.82BCa	12.60Aa	12.08ABa	8.33Da

Table 12. Analysis of variance (ANOVA) model results for the impact of water base mud (WBM) application date, and rate on winter wheat yield (kg ha^{-1}) for the Lahoma study. P = 0.05.

Variable	DF	Pr > F
rep	2	0.1748
rate	1	0.2211
appdate	4	0.0592
rate*appdate	4	0.6693

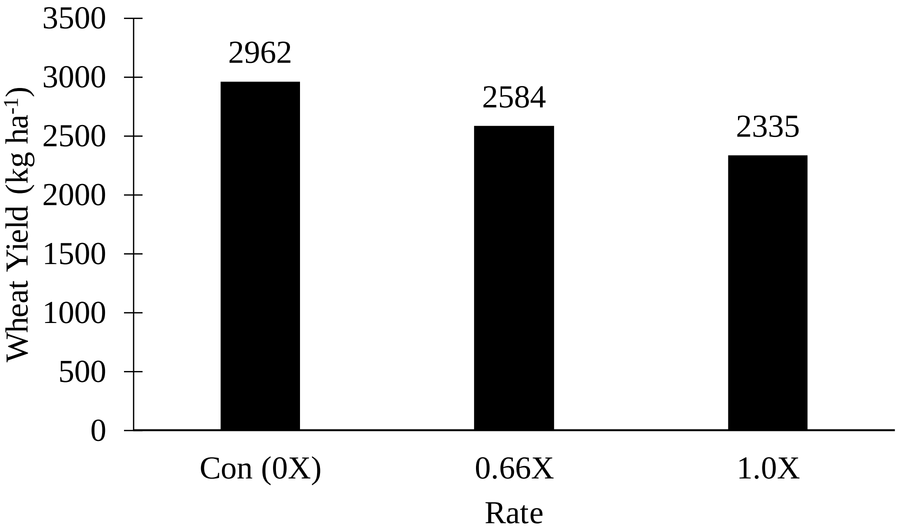

Figure 1. Mean winter wheat yield (kg ha^{-1}) averaged across application date and compared at each application rate. Water-base mud was applied to winter wheat in Oct, Dec, Jan, Feb, and March at a 1.0X and 0.66X rate that was equivalent to 6,721 and 4,480 kg TSS ha^{-1}soil, respectively.

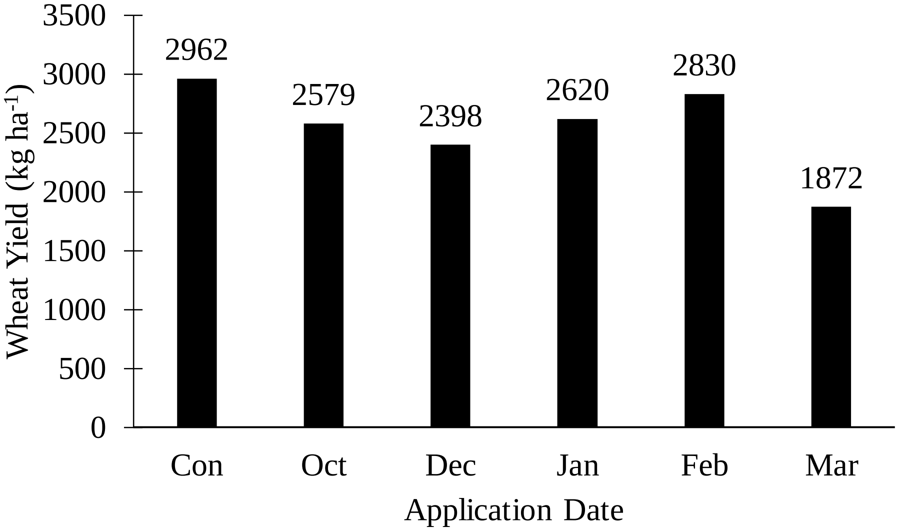

Figure 2. Mean winter wheat yield (kg ha^{-1}) averaged across application rate and compared at each application date. Water-base mud was applied to winter wheat in Oct, Dec, Jan, Feb, and March at a 1.0X and 0.66X rate that was equivalent to 6,721 and 4,480 kg TSS ha^{-1}soil, respectively.

CHAPTER IV

TOTAL PETROLEUM HYDROCARBON DEGRADATION AND BTEX LEACHING IN SOILS AS A FUNCTION OF OIL-BASE DRILLING MUD APPLICATION RATE, RAINFALL REGIME, AND TIME

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ABSTRACT

Increases in oil and gas drilling has resulted in large quantities of oil base “mud” (OBM) to be disposed of. Land application of OBM to agricultural land is a common disposal technique that presents agronomic and environmental challenges since the material is rich in total petroleum hydrocarbons (TPH). Leaching of lower molecular weight hydrocarbons, mainly benzene, toluene, ethylbenzene, and xylene (BTEX), is a concern due to their relatively low octanol:water partition coefficients. The objective of this study was to determine the effect of rainfall regime and TPH loading rate on TPH degradation and BTEX leaching after OBM application. An OBM was characterized for TPH, BTEX, and trace metals. A soil column study was conducted where OBM was applied at five loading rates (0, 22,000, 45,000, 67,000, and 90,000 kg TPH ha^{-1}) and was subjected to four moisture regimes. OBM samples were taken at day 0, 7, 30, 60, and 91 to monitor TPH degradation. Leachate samples were taken at day 0, 14, 28, 35, 49, 56, 63, 77, and 84 to monitor EC, pH, metal concentrations, and BTEX concentrations. After 60 days a

maximum TPH degradation of 35% was measured. Leachate BTEX concentrations increased as TPH application rate increased and were mostly undetectable by day 28. Leachate EC increased over time and with increasing TPH rates. TPH rate had no effect on leachate pH. OBM loading rates had the greatest effect on TPH degradation and BTEX leaching. There is little risk of BTEX leaching from land applied OBM.

INTRODUCTION

The United States is currently experiencing an oil and gas drilling boom. There are approximately 910,000 and 4,900 onshore and offshore oil and natural gas wells, respectively, that produce nearly 16 million m^3 of oil and 665 billion m^3 of natural gas (American Petroleum Institute, 2013). However, increased drilling activity has also lead to an increase in the production of drilling wastes, specifically drilling fluids and drill cuttings (aka “mud”). In 1995, a study conducted by the American Petroleum Institute estimated that around 150 million barrels of drilling wastes were generated on-shore in the United States alone (American Petroleum Institute, 2000).

Drilling mud is manufactured and utilized by the drilling industry to help cool the drill bit, maintain borehole pressure, and aid in bringing the drill cuttings to the surface where the fluids and cuttings can then be separated (Ukeles and Grinbaum, 2004). Drilling muds are comprised of a base liquid (water or diesel fuel) with other potential additives such as barium sulfate, bentonite, calcium hydroxide, and byproducts such as cotton seed hulls, used for specific drilling conditions (Moseley, 1983). If the base solution used to make the mud is diesel fuel, then the mud is known as “oil base mud” (OBM). Oil-base mud is typically utilized when drilling depths exceed 1500 m and for the horizontal portion. Due to the high cost of production, OBM is re-used by drillers for as long as possible.

At some point when the OBM can no longer be used in drilling, it must be properly disposed of. On average, a typical southeastern Oklahoma natural gas well that ranges from 4200-5200 m deep will produce 340 m^3 of OBM (Barker et al., 1992). Some of the products added to the mud may be deleterious and therefore need to be handled properly (Onwukwe and Nwakaudu, 2012). There are two options for mud disposal: land application and burial. Burial of the waste can occur onsite in “reserve pits” or at more sophisticated commercial facilities. In Oklahoma, land application is the most common method of OBM disposal. The purpose of land application of OBM is to allow soil microorganisms to degrade petroleum hydrocarbons (i.e. “total petroleum hydrocarbons”; TPH). Land application of OBM is regulated by the Oklahoma Corporation Commission (OCC) and application rates are limited based on loading of TPH, chlorides, and solids (Oklahoma administrative code and register, Title 165:10-7-26). Although TPH is taken into account when applying OBM there is still a potential for the over-application low molecular weight hydrocarbons benzene, toluene, ethylbenzene, and xylene (BTEX) to the soil. Benzene is a known human carcinogen and all compounds in BTEX are known to cause neurological effects (ATSDR, 1999). BTEX’s are prone to leaching due to their relatively low octanol:water partition coefficients (Sawyer et al., 2002) and therefore pose a threat to drinking water. In addition to loading limits, there are also several site suitability requirements such as soil texture, depth to groundwater and limiting layers, slope, soil sodium concentrations, and proximity to surface waters. The OCC also requires that OBM be mixed with a bulking material such as lime or gypsum, at a ratio of 3:1 OBM:bulking material.

Despite the fact that thousands of hectares are currently receiving OBM, there has been relatively little research conducted on the degradation of TPH and the leaching of BTEX after land application of OBM. Excessive application rates could lead to soil TPH concentrations that would be detrimental to soil and water quality leading to environmental issues. Penet et al. (2004) conducted a study that examined biodegradation of hydrocarbons in the soil and found that microbes degraded the straight chained hydrocarbons faster than the branched chained hydrocarbons. Dou et al. (2007) conducted a study focused on anaerobic BTEX degradation under nitrate reducing conditions. Results indicated that BTEX could be biodegraded to undetectable concentrations in 70 days if initial concentrations of BTEX were 100 mg kg^{-1} soil or below.

Very few studies have dealt with TPH degradation and BTEX leaching in soils after land application of OBM. Due to the hazardous risks of TPH, specifically BTEX toxicity to humans and to the environment. There is a need to examine TPH degradation and BTEX leaching in soils after land application of OBM under different scenarios such as: multiple loading rates and moisture regimes. Thus, the objective of this study was to determine the impact of rainfall regime and TPH loading rates from OBM application on TPH degradation and BTEX leaching.

MATERIALS AND METHODS

A soil column study was conducted in Stillwater, Oklahoma in a temperature controlled greenhouse. There were a total of 240 aluminum soil columns that were 30.5 cm tall and 7.6 cm in diameter. Columns were filled 15.2 cm with a sandy loam soil from Perkins, Oklahoma. The soil series used in this experiment came from the Dougherty loamy fine sand (Loamy, mixed, active, thermic Arenic Haplustalfs). Glass wool and aluminum screen with a 7.6 cm hose clamp was placed on the bottom of all columns in order to prevent soil from leaching out. The experimental design was a randomized complete block with factorial structure. There were three replications of each treatment.

The OBM sample was characterized for pH, electrical conductivity (EC), total soluble salts (TSS), total solids content, total and water extractable metals and total chloride. OBM pH and EC were measured using pH and EC probes with a solid:solution ratio of 1:5 and an equilibration time of 45 min. The OBM was analyzed for total P, K, Mg, Ca, Na, Mn, Cu, Fe, Zn, S, Al, Ni, B, As, Cd, Cr, Ba, Pb, and Mo using the EPA 3050 acid digestion method followed by solution analysis with inductively coupled argon plasma analyzer [ICP-AES; Spectro Ciros, Mahwah, NJ]. Water extractable metals and total chloride were extracted with de-ionized (DI) water using a 1:10 solid:solution ratio for 1 hour followed by ICP-AES analysis on the metals and colorimetric flow-injection analysis (Lachat QuickChem 8000, Loveland, CO) for chloride.

Prior to the application of OBM, BTEX and TPH concentrations were analyzed. Total petroleum hydrocarbons were extracted with hexane at a 1:10 solids:solvent ratio, plus addition of 0.5 g Na_2SO_4 for 5 minutes on a reciprocating shaker followed by centrifugation for 10 minutes. Five mL of the resulting supernatant was then equilibrated for 2 minutes with 1 g of silica gel in a glass tube for removal of polar organic compounds. The solution was then analyzed for TPH using infrared spectroscopy (ASTM method D 7066) with the InfraCal TOG/TPH analyzer (model HATR-T2, Wilks Enterprise Inc., East Norwalk, CT). Random samples were duplicated and check samples were utilized in order to assure precise and accurate results. Initial benzene, toluene, ethylbenzene, o-xylene, m, p-xylene and TPH concentrations were 2.65, 23, 35, 64, 94, and 161,558 mg kg^{-1}. Treatments included five TPH (i.e. OBM) loading rates and four rainfall regimes.

Soil columns were harvested for OBM analysis of TPH at four different times. OBM was applied onto an aluminum screen which rested on top of the soil that allowed soil to OBM contact yet prevented mixing and dilution of the applied OBM TPH with the soil. OBM loading rates were 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. Each column had one leaching event per month which consisted of 1.5 pore volumes of tap water. Moisture regime indicates the number non-leaching wetting events that occurred per month. Moisture regime levels 4, 3, 2, and 1 had 3, 2, 1, and 0 non-leaching wetting events per month, which consisted of 0.5 pore volumes of tap water. Leachate was analyzed for benzene, toluene, ethylbenzene, and xylene using the EPA 8021B method followed by solution analysis with Gas Chromatography with a photoionization detector (GC-PID). In addition, leachate was also analyzed for Na, Ca, Mg, K, SO_4, B, P, Fe, Zn, Cu, Mn, Al, Mo, As, Cd, Co, Cr, Pb, and Ba via ICP-AES. OBM was harvested 7, 30, 60, and 90 days after application and analyzed for TPH concentrations (mg TPH kg mud^{-1}) with the Wilks TOG/TPH IR Analyzer. Mud BTEX concentrations were only measured 7 days after application.

Statistics

Analyses of Variance (ANOVA) methods were utilized in PROC GLM (SAS Institute, 2011; Cary, NC, USA) to analyze the effects of OBM loading rates and moisture regimes on TPH degradation and BTEX leaching. When the main effects or interactions of OBM loading rates and moisture regimes were significant, treatment means were separated using pairwise comparisons via Duncan's multiple range test. Statistical decisions were made at $\alpha=0.05$.The data analysis for this paper was computed using SAS software.

RESULTS AND DISCUSSION

Background Soil Properties

The soil utilized in the BTEX leachate study was a sandy loam texture (Table 1) which made it an ideal soil to measure BTEX leaching in the soil profile that originated from land applied OBM. The OCC states in the Oklahoma administrative code and register, Title 165:10-7-26 that OBM must be incorporated into the soil after application; incorporation of the OBM leads to increased mixing (dilution) of the OBM into the soil and faster hydrocarbon degradation. Due to the large hydraulic conductivity of the sandy loam soil and the fact that the OBM was not incorporated made this study a worst case scenario for land application of OBM with respect to BTEX leaching and hydrocarbon degradation. The column soil had $N\text{-}NO_3^-$, P, and K concentrations of 8, 6, and 147 kg ha^{-1}, respectively. Soil pH was 6.8 and was in the optimal range for microbial degradation and limiting metal migration in the soil (Sims et al, 1989).

Oil-Based Mud Properties

The initial chemical analysis of the raw OBM and water extractable portion are listed in Table 2. The raw OBM had an initial TPH concentration of 161,558 mg TPH kg^{-1} and consisted of 74% solids. The raw OBM had a benzene concentration of 2.65 mg kg^{-1} which was higher than the inhalation limit of 0.8 mg kg^{-1} established by the U.S. EPA

(USEPA, 1996) and also for risk to groundwater by leaching (0.03 mg kg^{-1}; USEPA, 1996). The water soluble benzene concentration (0.015 mg L^{-1}) was higher than the groundwater limit of 0.005 mg L^{-1} set by the Oklahoma Guardian (Billingsley, 2003). Calcium was the dominant cation in both the raw solid and water extractable portion of the OBM. Chloride and sulfate were the two most abundant anions in the water extractable portion of the OBM. All heavy metal concentrations (Zn, Cu, Ni, As, Cd, Cr, and Pb) measured in the raw OBM were below EPA 503 thresholds for "exceptional quality" biosolids, indicating that there is only slight risk of metals contamination from land application of this OBM sample (U.S. EPA, 2014A). In fact, heavy metal concentrations in the OBM were in the normal range typically found in soils (U.S. EPA, 1992).

OBM TPH

The main effects of TPH rate, moisture regime, sampling day (i.e. time), and the two-way interaction of rate*day were significant at $\alpha = 0.05$ for TPH concentration (mg kg^{-1}OBM). An ANOVA table with the complete list of main effects and interactions for TPH concentration (mg kg^{-1}OBM) are listed in Table 3.

The main effect of TPH application rate (kg TPH ha^{-1}) on overall TPH concentration (mg TPH kg^{-1} OBM) was significant ($Pr \leq 0.05$) and is shown in more detail in Table 4 (averaged across all sampling times and moisture regimes). TPH application rate 1 (22,000 kg TPH ha^{-1}) had a significantly lower TPH concentration than all other rates and was closely followed by application rate 2 (45,000 kg TPH ha^{-1}) which was also significantly different than all other application rates. Application rate 3 (67,000 kg TPH ha^{-1}) and rate 4 (90,000 kg TPH ha^{-1}) had the highest TPH concentrations but were not significantly different than each other. The decreased TPH degradation displayed by application rates 3 and 4 were likely due to the decreased OBM to soil surface contact area, resulted in higher TPH concentrations.

The main effect of sampling day (time) on overall TPH concentration was significant ($Pr \leq 0.05$) and is shown in further detail in Table 5 (averaged across all application rates and moisture regimes). As time increases, a significant decrease in TPH concentration was observed until day 60. Day 60 and 91 had a significantly lower TPH concentration than all previous sampling days, however there was no significant difference between day 60 and 91. Figure 1 and Table 6 illustrates the insignificant degradation between day 60 and 91 for each application rate. There was a large decrease in TPH concentration for all TPH rates up until day 60. This plateau effect in degradation is likely due to the consumption of microbial nutrients (N) that inhibited further biodegradation of hydrocarbons. There are no significant differences in TPH concentrations between TPH application rates at day 0 or 7. Significant difference between TPH rates occur at day 30 and continue through day 91. TPH application rate 1 had the lowest TPH concentration followed by rate 2 and rate 3, while rate 4 is not significantly higher than rate 3. The TPH application rates 1 and 2 possess a higher proportion of OBM in contact with the soil surface, which may have improved degradation. Not only do TPH rates 3 and 4 have lower OBM to soil contact ratios which limited biodegradation of TPH, but the excessive loading rates could have impeded oxygen flow into the soil which may have further restricted microbial degradation of hydrocarbons. TPH concentrations for all application rates at day 90 were higher than the Oklahoma Guardian thresholds established for non-sensitive soils (46,000 mg kg^{-1}; Billingsley, 2003).

OBM BTEX

The main effect of sampling day on OBM BTEX concentration was significant at $\alpha = 0.05$. The main effect of TPH rate and the two-way interaction of rate*day was also significant at $\alpha = 0.05$ and is shown in Table 7 which provides a complete list of ANOVA results for the main effects and interactions.

Table 8 shows a significant decreases in BTEX concentration (mg kg^{-1}) for all BTEX constituents between day 0 and 7. These losses in BTEX concentrations over time can be attributed to volatilization, biodegradation, sorption to soil, and loss through leachate due to relatively high water solubility's that range from 1760-174 mg L^{-1} (Sawyer et al., 2003). At day 7, the benzene concentration was 0.06 mg kg^{-1} and was the only compound of BTEX that exceeded regulations established by the U.S. EPA for risk to groundwater leaching (U.S. EPA, 1996). Concentrations of toluene, ethylbenzene, o-xylene, and m, p-xylene were well below the EPA threshold limits for inhalation and groundwater risks.

The main effect of TPH application rate (kg TPH ha^{-1}) was significant ($Pr \leq 0.05$) for OBM BTEX concentration (mg kg^{-1}) for every OBM BTEX constituent except for benzene (Table 9), when averaging over the two sampling days. TPH application rate 1 had significantly lower concentrations of ethylbenzene, toluene, o-xylene, and m, p-xylene than all other TPH application rates. The TPH application rate 2 had the next lowest concentration values which were significantly different than all other TPH application rates. TPH application rates 3 and 4 had the highest concentrations of BTEX and were both significantly higher than TPH rates 1 and 2, although not significantly different from each other. Similar trends were noted regarding TPH concentrations (Table 4). Higher BTEX concentrations (i.e. lower degradation) at the highest TPH application (3 and 4) likely occurred for the same reasons as previously discussed for TPH degradation.

BTEX Leachate Concentrations

The main effects of TPH rate, moisture regime, and leaching event and the interactions between each are shown in Table 10.The main effect of leaching event was significant ($Pr \leq 0.05$) for BTEX leachate concentrations ($\mu g\ L^{-1}$) and is shown in added detail in Table 11, averaged over TPH application rate and moisture regime. Significantly higher concentrations of BTEX were measured for leaching event 1 when compared to leaching events 2 and 3. For every BTEX constituent except for o-xylene, leaching event 1 was the only leaching event in which detectable levels of BTEX were measured in leachate. All BTEX concentrations at leaching event 1 were low ($< 5\ ng\ L^{-1}$) and below the threshold limits for drinking water established by EPA 816F regulations. It is noteworthy to mention that each leaching event is an average of three leachate sampling days and a higher leaching event also indicates a greater amount of time that has occurred since application of OBM. No BTEX was detected in leachate from leaching events 2 and 3 because it was either mostly volatized, degraded, or sorbed to soil surfaces.

The main effect of moisture regime was significant ($Pr \leq 0.05$) for BTEX leachate concentrations ($\mu g\ L^{-1}$) and is presented in further detail in Table 12. Moisture regime 1

(0 wetting events per month) showed significantly higher BTEX concentrations than all other moisture regimes that received non-leaching wetting events. The concentrations of each BTEX constituent at moisture regime 2, 3, and 4 were statistically the same. However, the highest concentrations of BTEX observed in the leachate for moisture regime 1 is likely due to the fact that values from moisture regime one was averaged over the first sampling day of each month (day 0, 35, and 63). Specifically, moisture regime 1 was sampled (i.e. leached) on day 0, 35, and 63 and only had BTEX concentrations above 0 on day 0, which were the highest for the entire study. The highest BTEX leachate concentrations from day 0 thus caused moisture regime 1 to be significantly higher than the other moisture regimes. Again, this shows the importance of time on BTEX degradation, volatilization, and sorption to the soil.

The three-way interaction of TPH rate by moisture regime by leaching event was significant ($Pr \leq 0.05$) for benzene and toluene leachate concentrations ($\mu g\ L^{-1}$) and is shown in Table 13. Moisture regime 1 is the only regime shown in Table 13 because this was the only moisture regime that had significant amounts of BTEX in the leachate (Table 12). A general trend of increasing concentrations of benzene and toluene in leachate was observed as the rate of TPH application increased with regard to leaching event 1. Leaching events 2 and 3 have no detectable concentrations of benzene or toluene in the leachate. By the time leaching events 2 and 3 occurred, all amounts of benzene and toluene were lost via microbial degradation, volatilization, and sorption to the soil, or through leachate.

BTEX Loading

The main effects of TPH application rate, moisture regime, and leaching event on BTEX leachate loads and the interactions between variables are shown in Table 14.

The three-way interaction of TPH rate by moisture regime by leaching event was significant ($Pr \leq 0.05$) for all constituents of BTEX except for o-xylene and is shown in further detail in Table 15. Moisture regime 1 is the only moisture regime listed due to the fact that this was the only moisture regime that contained detectable concentrations in leachate (Table 12). A general trend of increasing BTEX loads is seen as the TPH rate increased with regard to leaching event 1. Leaching events 2 and 3 have no detectable concentrations of BTEX in the leachate. By the time leaching event 2 and 3 occurred, all BTEX was degraded, volatized, sorbed to the soil, or lost via leaching.

Leachate EC

The main effects of TPH application rate, moisture regime, and leaching event on leachate EC, and interactions between variables are shown in Table 16. The two-way interaction of TPH rate by leaching event was significant ($Pr \leq 0.05$) for leachate EC (mS cm^{-1}) and is shown in greater detail in Table 17. A significant increase in leachate EC is observed with increasing TPH application rates for each leaching event. As TPH rate increased, the total amount of salts applied from the OBM (Table 2) increased which led to higher leachate EC values as the salts dissolved with the leachate. A general trend of increasing leachate EC with leaching events was observed for each TPH application rate except for the control which received no amendment. The leachate EC can serve as an indicator of the mobility of soluble species in the solution and can be used to show the leaching front for soluble species. Due to the fact that leachate EC continues to increase at each leaching event while BTEX concentrations do not (Table 11), this confirms that the BTEX has either degraded or sorbed to the soil or was lost via volatilization.

Leachate pH

The main effects of moisture regime and leaching events and the interactions of TPH rate*leaching event and moisture regimes*leaching events were significant at $\alpha = 0.05$ for the pH of the leachate. (Table 16) provides a complete list of ANOVA results for all main effects and interactions for leachate pH.

The main effect of leaching event was significant ($Pr \leq 0.05$) for BTEX leachate pH and is shown in more detail in Figure 3. There were significant increases in BTEX leachate pH with each additional leaching event. However, TPH application rate had no effect on leachate pH because the pH of the control leachate also had significant increases in pH at each leaching event (Figure 4). The increase in BTEX leachate pH across the leaching events was likely due to the alkaline pH (8.23) water that was used to leach the soil columns. As time progressed throughout leaching events the leachate pH approached the higher pH values of the water that that was used to leach the soil columns.

CONCLUSION

The application of OBM in this study occurred mostly at rates in great excess of current OCC regulations, and were not incorporated in order to examine the worst case scenario regarding environmental impact. The OBM used in this soil column leachate study did not possess heavy metal concentrations beyond normal soil concentrations Benzene concentrations in the raw OBM (2.65 mg kg^{-1}) were higher than the EPA threshold limits established for inhalation (0.8 mg kg^{-1}) and leaching to groundwater (0.03 mg kg^{-1}). Regardless, by day 7 the BTEX concentration in the mud had decreased by 88% and benzene only leached out during the first leaching event which produced benzene concentrations less than drinking water standards. This was surprising due to the high benzene content in the OBM which greatly exceeded EPA risk levels for groundwater leaching, the short column length, and high hydraulic conductivity of soil. An explanation for this is found in closer examination of the assumptions made in creating the EPA threshold for leaching to groundwater i.e. no degradation of benzene and the entire soil profile contains benzene from the surface to the groundwater interface. As expected, increased OBM application rates resulted in higher leachate benzene concentrations. All leachate BTEX concentrations were below drinking water thresholds. No trace metals were detected in leachate. Part of the reason for non-detectable BTEX concentrations in leachate after the initial leaching event on day 0 is due to 88% OBM BTEX degradation by day 7.

The main effect of TPH application rate had the greatest effect on TPH degradation, BTEX concentrations in the OBM, leachate BTEX concentrations and loads, and leachate EC. As the rate of TPH increased, a decrease in hydrocarbon degradation was seen due to the higher OBM to soil ratio that limited oxygen inflow and microbial degradation. A plateau effect on biodegradation of TPH was seen at day 60 and continued throughout day 91. At this point, the microbes had likely consumed all of the nutrients (N) and could no longer biodegrade the TPH. Therefore, applying a source of fertilizer and increasing the surface area to volume ratio of the OBM via disking or using a bulking agent is important when considering microbial degradation of TPH. During the study, the maximum TPH degradation that occurred was 35%, which occurred from the lowest TPH application rate.

Leachate EC increased as TPH rate increased due to higher loads of soluble salts. Leachate EC also increased at each leaching event as opposed to the decreasing BTEX concentrations with additional leaching event which confirmed that the BTEX had either volatilized, sorbed to the soil, or degraded. The main effect of TPH rate had no effect on leachate pH.

Future studies need to be conducted to quantity the amount of BTEX volatilization. Volatilization of hydrocarbons does occur, especially in the lower molecular weight hydrocarbons such as in BTEX. However, in this study volatilization was not measured.

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TABLES AND FIGURES

Table 1. Background chemical analysis of the soils used in the BTEX leaching column study.

Soil Texture	Sandy Loam
pH	6.8
Electrical Conductivity (mS cm^{-1})	0.69
Total Soluble Salts (mg L^{-1})	454.1
$N\text{-}NO_3^-$ (kg ha^{-1})	8
Soil Test Phosphorus (kg ha^{-1})	6
Soil Test Potassium (kg ha^{-1})	147
Ca (kg ha^{-1})	1789
Mg (kg ha^{-1})	297
$S\text{-}SO_4^{-2}$ (kg ha^{-1})	9

Table 2. Characterization of the raw (solids plus liquid) and the water extractable portion of the oil-base mud (OBM) used in the BTEX leaching column study. All water extraction results were obtained by using a 1:10 solids to DI water ratio unless otherwise noted.

Raw OBM Concentration		Oil-Based Mud Water Extraction	
TPH (mg kg^{-1})	161,558	EC (mS cm^{-1}) (1:3)	6.91
Benzene (mg kg^{-1})	2.65	pH (1:3)	11.3
Toluene (mg kg^{-1})	23.0	Total Soluble Salts (mg L^{-1})	4561
Ethylbenzene (mg kg^{-1})	35.0	Benzene (mg kg^{-1})	0.15
o-Xylene (mg kg^{-1})	64.0	Toluene (mg kg^{-1})	1.12
m, p-Xylene (mg kg^{-1})	94.0	Ethylbenzene (mg kg^{-1})	0.49
% Solids Content	74.0	o-Xylene (mg kg^{-1})	0.79
Na (mg kg^{-1})	2395.4	m, p-Xylene (mg kg^{-1})	1.32
Ca (mg kg^{-1})	55518.1	Na (mg kg^{-1})	1616.9
Mg (mg kg^{-1})	5867.1	Ca (mg kg^{-1})	6240.0
K (mg kg^{-1})	1962.9	Mg (mg kg^{-1})	0.0
SO_4^{-2}-S (mg kg^{-1})	14925.8	K (mg kg^{-1})	259.0
P (mg kg^{-1})	312.6	SO_4^{-2}-S (mg kg^{-1})	3145.5
Fe (mg kg^{-1})	17681.3	P (mg kg^{-1})	1.3
Zn (mg kg^{-1})	61.1	Fe (mg kg^{-1})	0.5
Cu (mg kg^{-1})	71.1	Zn (mg kg^{-1})	0.0
Mn (mg kg^{-1})	414.1	Cu (mg kg^{-1})	0.2
Al (mg kg^{-1})	8232.3	Mn (mg kg^{-1})	0.0
Ni (mg kg^{-1})	17.7	As (mg kg^{-1})	0.0
B (mg kg^{-1})	46.6	Cd (mg kg^{-1})	0.0
As (mg kg^{-1})	0.1	Cr (mg kg^{-1})	0.0
Cd (mg kg^{-1})	0.0	Ba (mg kg^{-1})	1.68
Cr (mg kg^{-1})	20.7	Pb (mg kg^{-1})	0.07
Ba (mg kg^{-1})	971.9	Co (mg kg^{-1})	0.0
Pb (mg kg^{-1})	21.9	Cl (mg kg^{-1})	8549.34
Co (mg kg^{-1})	0.0		

Table 3. ANOVA results for total petroleum hydrocarbons (TPH; mg kg^{-1} OBM) concentrations in the surface applied OBM. Results are significant when ($Pr \leq 0.05$).

Variable	DF	Pr > F
rep	2	0.1175
rate	3	<0.001
moisture	3	0.0301
day	4	<0.0001
rate*moisture	9	0.0686
rate*day	12	<0.0001
rate*moisture*day	36	0.1721

Table 4. Mean total petroleum hydrocarbon (TPH) concentrations (mg kg^{-1} OBM) in the surface applied OBM averaged across moisture regime and sampling day for each TPH application rate. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes. OBM was sampled at 0, 7, 30, 60, and 90 days after application. Uppercase letters represent mean separation of TPH concentration (mg TPH kg^{-1} mud) between TPH rates. Statistical decisions were mad at P = 0.05.

TPH application rate (kg TPH ha^{-1})	OBM TPH (mg TPH kg^{-1} OBM)
22,000	117,403C
45,000	126,037B
67,000	134,100A
90,000	137,788A

Table 5. Mean total petroleum hydrocarbon (TPH) concentration (mg TPH kg^{-1} OBM) at each sample day averaged over moisture regime and TPH application rate. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes. OBM was sampled at 0, 7, 30, 60, and 91 days after application. Uppercase letters represent mean separation of TPH degradation (mg TPH kg^{-1} OBM) between all sample days. Statistical decisions were made at P = 0.05.

Sample Day	OBM TPH (mg TPH kg^{-1} OBM)
0	161,558A
7	139,705B
30	129,652C
60	105,641D
91	107,605D

Table 6. Mean total petroleum hydrocarbon (TPH) concentration (mg TPH kg^{-1} OBM) averaged across moisture regime and compared between sample day and TPH application rate. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes. OBM was sampled at 0, 7, 30, 60, and 91 days after application. Uppercase letters represent mean separation of TPH concentration between sampling days at each TPH application rate. Lowercase letters represent mean separation of TPH concentration between TPH application rates at each sampling day. Statistical decisions were made at P = 0.05.

	TPH (mg TPH kg^{-1} OBM)				
TPH rate (kg TPH ha^{-1})	Day 0	Day 7	Day 30	Day 60	Day 91
22,000	161,558Aa	136,549Ba	112,346Cc	88,738Dc	87,827Dc
45,000	161,558Aa	136,047Ba	126,705Cb	102,749Db	103,127Db
67,000	161,558Aa	140,816Ba	140,394Ba	111,807Ca	115,926Ca
90,000	161558Aa	145,409Ba	139,163Ba	119,272Ca	123,540Ca

Table 7. ANOVA results for oil-base mud (OBM) benzene, ethylbenzene, toluene, o-xylene, and m, p-xylene (BTEX) concentrations in mg kg^{-1} OBM for the BTEX leaching column study. Results are significant when ($Pr \leq 0.05$).

		Benzene	Ethylbenzene	Toluene	o-Xylene	m, p-Xylene
Variation	DF	Pr > F				
rep	2	0.2719	0.1352	0.1841	0.1450	0.1331
rate	3	0.1591	<0.0001	0.0005	<0.0001	<0.0001
moisture	3	0.7561	0.3655	0.3879	0.3127	0.3086
day	1	<.0001	<0.0001	<0.0001	<0.0001	<0.0001
rate*moisture	9	0.5836	0.0969	0.2358	0.0792	0.0795
rate*day	3	0.2112	<0.0001	0.0004	<0.0001	<0.0001
moisture*day	3	0.6667	0.1548	0.1952	0.1005	0.1151
rate*moisture*day	9	0.3669	0.1124	0.2246	0.0787	0.0920

Table 8. Mean benzene, ethylbenzene, toluene, o-xylene, and m, p-xylene (BTEX) concentrations (mg kg^{-1} OBM) averaged across moisture regime and total petroleum hydrocarbon (TPH) application rate and compared between each sampling day. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes. OBM BTEX was sampled at 0, and 7 days after application. Lowercase letters represent mean separation for BTEX degradation (mg kg^{-1} OBM) between each sampling day. Statistical decisions were made at P = 0.05.

	BTEX (mg kg^{-1} OBM)				
Day	Benzene	Ethylbenzene	Toluene	o-Xylene	m, p-Xylene
0	2.65a	34.99a	23.28a	63.53a	94.08a
7	0.06b	4.40b	2.58b	7.80b	12.07b

Table 9. Mean benzene, ethylbenzene, toluene, o-xylene, and m, p-xylene (BTEX) concentrations (mg kg^{-1} OBM) at each total petroleum hydrocarbon (TPH) application rate for the BTEX leaching column study. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes. OBM BTEX was sampled at 0, and 7 days after application. Lowercase letters represent mean separation for BTEX concentration (mg kg^{-1} OBM) between each TPH rate. Statistical decisions were made at P = 0.05.

	BTEX (mg kg^{-1} OBM)				
TPH rate (kg TPH ha^{-1})	Benzene	Ethylbenzene	Toluene	o-Xylene	m, p-Xylene
22,000	1.33a	17.96c	11.97b	32.75c	48.43c
45,000	1.33a	19.20b	12.48b	35.09b	51.97b
67,000	1.37a	20.67a	13.48a	37.25a	55.60a
90,000	1.39a	20.96a	13.79a	37.56a	56.29a

Table 10. ANOVA results for benzene, ethylbenzene, toluene, o-xylene, and m, p-xylene (BTEX) leachate concentrations in (μg L^{-1}) for the BTEX leaching column study. Results significant when ($Pr \leq 0.05$). LE = “leaching event”.

		Benzene	Ethylbenzene	Toluene	o-Xylene	m, p-Xylene
Source	DF			Pr > F		
rep	2	0.085	0.1017	0.0924	0.144	0.0717
rate	4	0.031	0.0994	0.1102	0.0059	0.0747
moisture	3	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
LE	2	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
rate*moisture	12	0.005	0.1310	0.0752	0.2328	0.1163
rate*LE	8	0.0116	0.0702	0.0839	0.0034	0.0496
moisture*LE	6	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
rate*moisture*LE	24	0.0005	0.1104	0.0491	0.2416	0.1032

Table 11. Mean benzene, ethylbenzene, toluene, o-xylene, and m, p-xylene (BTEX) leachate concentrations (µg L^{-1}) averaged over total petroleum hydrocarbon (TPH) application rate and moisture regimes and compared between leaching events. for the BTEX leaching column study. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes which had one leaching event per month. Lowercase letters represent mean separation between leaching events for each BTEX constituent concentration (µg L^{-1}). Statistical decisions were made at P = 0.05. LE = “leaching event”.

LE	Benzene (µg L^{-1})	Ethylbenzene (µg L^{-1})	Toluene (µg L^{-1})	o-Xylene (µg L^{-1})	m, p-Xylene (µg L^{-1})
1	0.50a	2.12a	3.77a	3.27a	4.89a
2	0.00b	0.00b	0.00b	0.15b	0.00b
3	0.00b	0.00b	0.00b	0.00b	0.00b

Table 12. Mean ethylbenzene and toluene leachate concentrations (µg L^{-1}) averaged over total petroleum hydrocarbon (TPH) application rate and leaching events and compared between moisture regimes. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes which had one leaching event per month. Uppercase letters represent mean separation for ethylbenzene and toluene between moisture regimes. Statistical decisions were made at P = 0.05.

	Moisture 1	Moisture 2	Moisture 3	Moisture 4
Benzene (µg L^{-1})	0.67A	0.00B	0.00B	0.00B
Ethylbenzene (µg L^{-1})	2.65A	0.09B	0.04B	0.05B
Toluene (µg L^{-1})	4.91A	0.06B	0.02B	0.03B
o-Xylene (µg L^{-1})	3.51A	0.43B	0.34B	0.28B
m, p-Xylene (µg L^{-1})	6.06A	0.12B	0.15B	0.18B

Table 13. Mean benzene and toluene leachate concentrations (µg L^{-1}) at moisture regime one comparing total petroleum hydrocarbon (TPH) application rates and leaching events for the BTEX leaching column study. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes which had one leaching event per month. Uppercase letters represent mean separation of benzene and toluene leachate concentrations between TPH application rates at each leaching event within moisture regime one. Lowercase letters represent mean separation of benzene and toluene leachate concentrations between leaching events at each TPH application rate within moisture regime one. Statistical decisions were made at P = 0.05.

TPH Rate (kg TPH ha^{-1})	0	22,000	45,000	67,000	90,000
Leaching Event			Benzene (µg L^{-1})		
1	0.00Ca	0.44BCa	2.48ABCa	2.92ABa	4.20Aa
2	0.00Aa	0.00Aa	0.00Ab	0.00Aa	0.00Ab
3	0.00Aa	0.00Aa	0.00Ab	0.00Aa	0.00Ab
Leaching Event			Toluene (µg L^{-1})		
1	0.00Ba	6.37ABa	15.56ABa	24.72Aa	27.00Aa
2	0.00Aa	0.00Aa	0.00Ab	0.00Aa	0.00Ab
3	0.00Aa	0.00Aa	0.00Ab	0.00Aa	0.00Ab

Table 14. ANOVA results for benzene, ethylbenzene, toluene, o-xylene, and m, p-xylene (BTEX) leachate loads (µg) for the BTEX leaching study. Results were significant when (Pr ≤ 0.05). LE = "leaching event".

		Benzene	Ethylbenzene	Toluene	o-xylene	m, p-xylene
Variation	DF			Pr > F		
rep	2	0.0981	0.1569	0.1057	0.3109	0.0896
rate	4	0.0051	0.0239	0.0340	<0.0001	0.0094
moisture	3	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
LE	2	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
rate*moisture	12	0.0002	0.0595	0.0176	0.2637	0.0535
rate*LE	8	0.0005	0.0061	0.0127	<0.0001	0.0016
moisture*LE	6	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
rate*moisture*LE	24	<0.0001	0.0274	0.0044	0.1646	0.0290

Table 15. Mean benzene, ethylbenzene, toluene, o-xylene, and m, p-xylene (BTEX) leachate loads at moisture regime one, comparing TPH application rates and leaching events. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes which had one leaching event per month. "†"- o-xylene was not significant at $P = 0.05$. Uppercase letters represent mean separation of BTEX loads (µg) between TPH application rates at each leaching event, for moisture regime one. Lowercase letters represent mean separation of BTEX loads (µg) between leaching events at each TPH rate, for moisture regime one. Statistical decisions were made at $P = 0.05$.

Moisture Regime One

TPH Rate (kg TPH ha^{-1})	0	22,000	45,000	67,000	90,000
Leaching Event			Benzene (µg)		
1	0.00Ca	0.21BCa	1.13ABCa	1.22ABa	2.10Aa
2	0.00Aa	0.00Aa	0.00Ab	0.00Aa	0.00Ab
3	0.00Aa	0.00Aa	0.00Ab	0.00Aa	0.00Ab
Leaching Event			Ethylbenzene (µg)		
1	0.00Ca	1.75BCa	3.91ABCa	5.30ABa	7.15Aa
2	0.00Aa	0.00Aa	0.00Ab	0.00Aa	0.00Ab
3	0.00Aa	0.00Aa	0.00Ab	0.00Aa	0.00Ab
Leaching Event			Toluene (µg)		
1	0.00Ca	2.93BCa	7.00ABCa	10.22ABa	13.38Aa
2	0.00Aa	0.00Aa	0.00Ab	0.00Aa	0.00Aa
3	0.00Aa	0.00Aa	0.00Ab	0.00Aa	0.00Aa
Leaching Event			†o-Xylene (µg)		
1	0.00	2.66	5.60	6.49	8.74
2	0.00	0.00	0.00	0.00	0.57
3	0.00	0.00	0.00	0.00	0.00
Leaching Event			m, p-Xylene (µg)		
1	0.00Ca	4.30BCa	9.34ABCa	11.72ABa	16.06Aa
2	0.00Aa	0.00Aa	0.00Ab	0.00Aa	0.00Ab
3	0.00Aa	0.00Aa	0.00Ab	0.00Aa	0.00Ab

Table 16. ANOVA results for leachate electrical conductivity (EC; mS cm^{-1}) and pH for the BTEX leaching column study. Results were significant when ($Pr \leq 0.05$). LE = “leaching event”.

		EC (mS cm^{-1})	pH
Source	DF	Pr>F	
rep	2	<0.0001	0.0782
rate	4	<0.0001	0.5053
moisture	3	<0.0001	0.0005
LE	2	<0.0001	<0.0001
rate*moisture	12	0.2153	0.2817
rate*LE	8	<0.0001	0.0044
moisture*LE	6	<0.0001	<0.0001
rate*moisture*LE	23	0.1714	0.2605

Table 17. Mean leachate electrical conductivity (EC; mS cm^{-1}) averaged across moisture regime comparing total petroleum hydrocarbon (TPH) application rates and leaching events (LE). OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes which had one leaching event per month._ Uppercase letters represent mean separation of leachate EC (mS cm^{-1}) between leaching events at each TPH application rate. Lowercase letters represent mean separation between TPH application rates at each leaching event. Statistical decisions were made at P = 0.05.

TPH Rate (kg TPH ha^{-1})	LE 1	LE 2	LE 3
0	1.00Ac	1.09Ab	0.71Bc
22,000	1.08Abc	1.24Bab	1.34Bb
45,000	1.15Ab	1.42Aa	1.72Ba
67,000	1.18Ab	1.38Ba	1.56Bab
90,000	1.33Aa	1.41Aa	1.79Ba

Table 18. Mean leachate pH values averaged over total petroleum hydrocarbon (TPH) rate, comparing moisture regimes and leaching events. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes which had one leaching event per month. Uppercase letters represent mean separation of leachate pH between moisture regimes at each leaching event. Lowercase letters represent mean separation of leachate pH between leaching events at each moisture regime. Statistical decisions were made at P = 0.05.

BTEX leachate pH				
Leaching Event	Moisture 1	Moisture 2	Moisture 3	Moisture 4
1	7.63Ab	7.09Bc	6.95Bb	7.01Bb
2	7.31Cb	8.06Aa	7.53BCa	7.73ABa
3	8.21Aa	7.62Bb	7.61Ba	7.78Ba

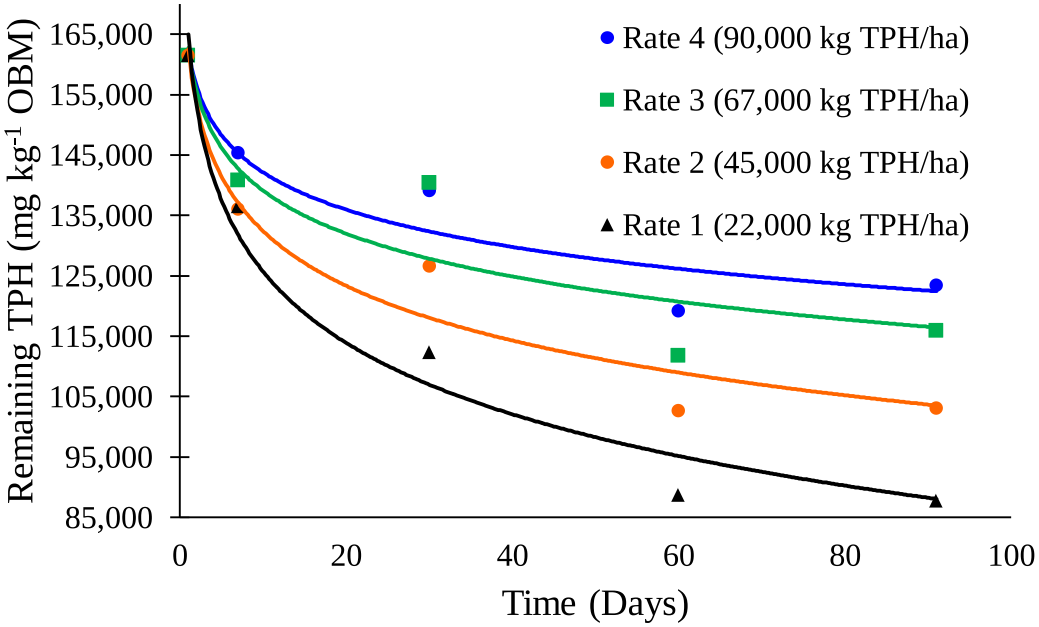

Figure 1. Mean remaining total petroleum hydrocarbon (TPH) concentrations in the OBM in mg kg^{-1} OBM. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes. OBM was sampled at 0, 7, 30, 60, and 91 days after application. TPH values are for each sampling day and TPH application rate averaged over moisture regime.

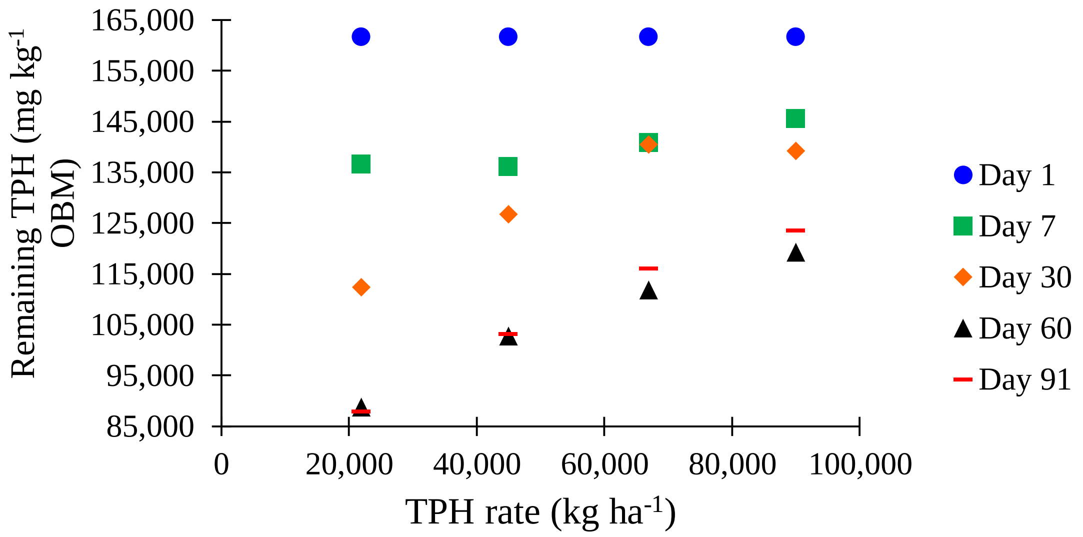

Figure 2. Mean remaining total petroleum hydrocarbon (TPH) concentrations in oil-base mud (mg kg^{-1} OBM) for the BTEX leaching study. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes. OBM was sampled at 0, 7, 30, 60, and 91 days after application. Remaining TPH concentrations are shown for each TPH application rate, and sampling time, and are averaged over moisture regime.

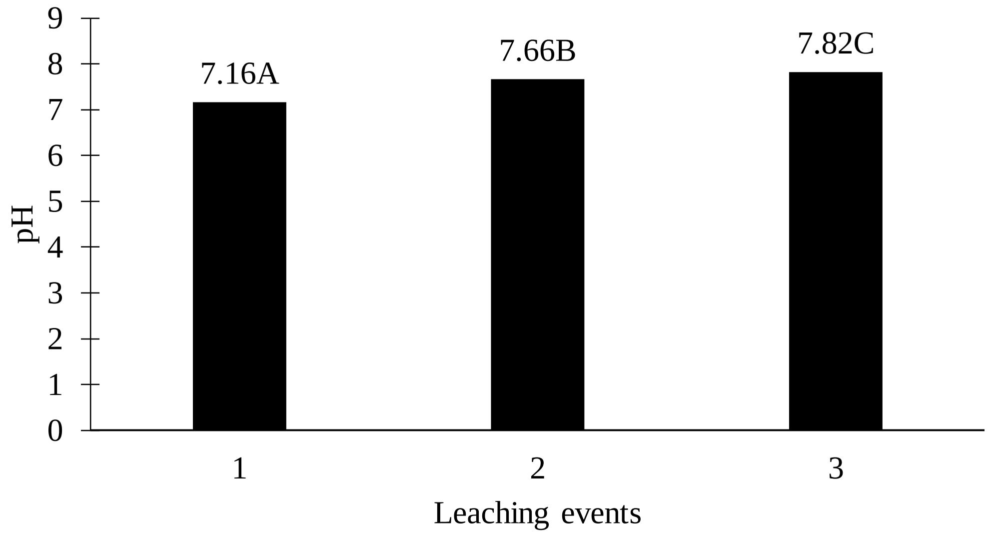

Figure 3. Mean leachate pH comparing each leaching event for the BTEX leaching column study. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes which had one leaching event per month. Leachate pH values are averaged over total petroleum hydrocarbon (TPH) rate and moisture regimes. Uppercase letters represent mean separation of leachate pH between leaching events. Statistical decisions were made at P = 0.05.

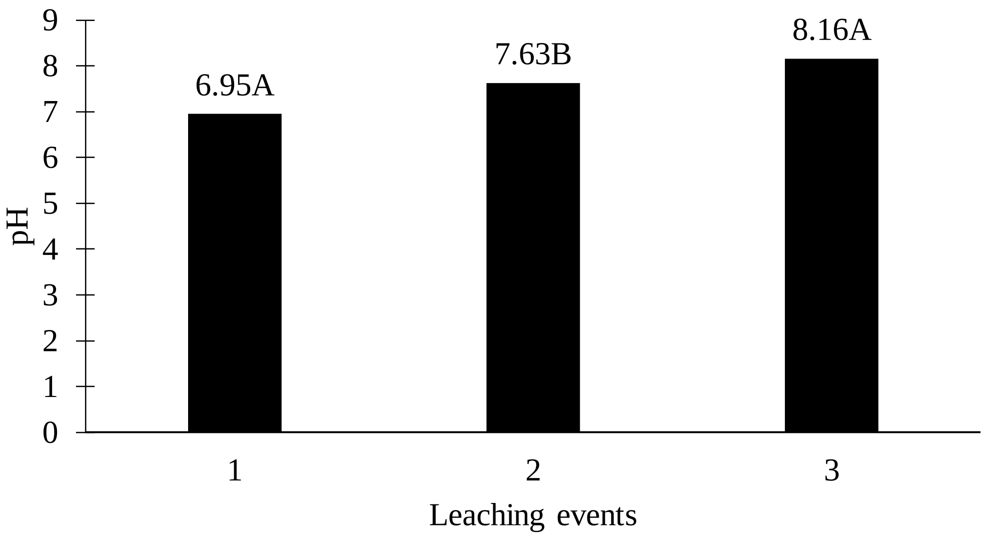

Figure 4. Mean leachate pH of the control (no OBM) comparing each leaching event for the BTEX leachate column study. OBM loading rates were applied at 90,000, 67,000, 45,000, 22,000, and 0 (control) kg TPH ha^{-1}. The treatments were subjected to four different moisture regimes which had one leaching event per month. Leachate pH was averaged over moisture regimes. Uppercase letters represent mean separation of leachate pH between leaching events. Statistical decisions were made at P = 0.05.

CHAPTER V

SUMMARY

Oil-base Mud Studies

With the explosion of oil and natural gas drilling across the United States, there has come to be an abundance of OBM and WBM waste. One way of eliminating large amounts of OBM and WBM is by land application, which is the main method of disposal in Oklahoma. However, when applying OBM and WBM, potential agronomic and environmental challenges can occur due to high TPH levels in the OBM and high total soluble salts in the WBM. Due to these factors, proper application of drilling mud is critical in order to protect the environment. The OBM samples used in both studies had similar TPH, pH, EC, and solids content. Concentrations of heavy metals (Zn, Cu, Ni, As, Cd, Cr, and Pb) for both OBM's were all well below EPA 503 thresholds for "exceptional quality" biosolids, indicating that there was little risk of metals contamination from land application of the OBM. The main environmental concern arising from OBM application is due to the high concentrations of TPH, although SAR, EC, heavy metal concentrations, and NORM can potentially be a problem depending on the geology and the area where drilling occurred. By law, OBM must be mixed with a "bulking material" such as lime or gypsum at a 3:1 (bulking agent:OBM) ratio when land applied in Oklahoma. The first OBM study compared a 3:1 and 1.5:1 mixing agent:OBM ratio on TPH degradation and its effect on plant biomass production. Results showed that TPH degradation was not influenced by the type of mixing material (caliche, lime or gypsum) or the ratio of mixing agent:OBM. Therefore, the use of caliche, lime, or gypsum mixed with OBM at a 1.5:1 mixing agent:OBM would be more economically sound than a 3:1 ratio, and still achieve acceptable TPH decreases when surface applied and non-incorporated for that particular soil and location under the same conditions. By day 170 adequate rainfall had fallen and approximately 99% of applied TPH had degraded and achieved safe levels according to the OK Guardian document. The decrease in TPH is mainly attributed to biological degradation since OBM TPH contained only 17% of low molecular weight hydrocarbons. However, nothing is known about volatilization of the lower molecular hydrocarbons (i.e. BTEX). Most of the TPH remained in the top 5 cm, although there were some significant increases in TPH in the subsurface soil when compared to the control. Salts applied through application of OBM leached out quickly into the subsurface. Plant biomass production was not significantly impacted by OBM application due to the fast rate of TPH degradation and its relatively low impact on soil salinity.

Due to the findings in the first OBM study, a second study (90 days long) was conducted to monitor TPH degradation and the concentration of BTEX in leachate produced from surface applied and non-mixed OBM as impacted by TPH application rates and moisture regimes. This study utilized TPH application rates in excess of current OCC regulations, and surface applications in order to simulate the worst case scenario. A maximum TPH degradation of 35% was observed at day 60. After day 60, TPH degradation was limited likely due to the lack of available microbial nutrients required for respiration. BTEX leachate concentrations were significantly higher as TPH application rate increased; BTEX leaching mostly occurred during the first leaching event. At the highest leachate BTEX concentrations, none exceed thresholds for EPA drinking water quality standards. Leachate EC significantly increased as TPH rate increased. Leachate pH was not impacted by TPH rate. The amount of BTEX lost by volatilization is still unknown and further research needs to be conducted. Based on the results of this study for this particular soil used and under the same conditions, there is little to no risk of land applied OBM causing BTEX leaching to groundwater, even with over-applications of OBM.

Water-base mud study

The main concern when applying WBM to agricultural land is due to high total soluble salts. Proper application of WBM is critical in order to properly protect the soil and plants from salinization and soil sodicity. In this study, WBM was applied at two different rates and at five different application dates onto wheat. The WBM used in this study had no significant effects on soil pH. Initial increases in soil EC from WBM application were dramatic. However, soil EC levels greatly decreased with rainfall. Soil SAR values significantly increased with time after application via dissolution and release of non-dissolved sodium with rainfall and potentially from wicking of previously leached salts upward via plant water uptake. WBM rates and application dates did not significantly affect wheat yield when compared to the control. However, a non-statistically significant agronomic effect on wheat yield was observed when application of WBM occurred in March when the wheat was more mature and had greater susceptibility to the high salt content. The degree of potential environmental consequences and changes in soil chemical properties and crop yield for this location and soil depend primarily on the chemical characteristics of the WBM (pH, EC, SAR), application rate, crops grown, and rainfall. Damage to wheat can be minimized by applying WBM at a time when appreciable rainfall is expected, thereby quickly moving soluble salts out of the root zone. In general, application rates less than the maximum allowable salt loading limit will decrease risk of causing soil salinization, sodicity, and loss of crop yield. Further studies should be conducted that monitor salt leaching deeper within the soil profile and determine the amount of time required for complete dissolution of the non-dissolved sodium and an eventual decrease in soil SAR levels.

APPENDIX A

Soil data collected at the Shattuck, OK field trial where oil-base mud (OBM) was mixed with a bulking agent (caliche, lime or gypsum) at a 3:1 and 1.5:1 bulking agent: OBM ratio and land applied to native range at 8,625 kg TPH ha^{-1}. Soil samples were analyzed for pH, EC, metals, and TPH at 7, 60, and 170 days after application. Forage was harvested 60 days after OBM application.

Table A.1: Saturated paste soil EC and pH for the OBM study in Shattuck, OK.

			Day 7		Day 60		Day 170	
Rep	Samples	Depth (in)	EC (µS/cm)	pH	EC (µS/cm)	pH	EC (µS/cm)	pH
1	C	0-2	428	7.70	404	9.54	1034	7.51
2	C	0-2			405	8.56	655	7.85
3	C	0-2			641	8.51	432	7.76
1	C	2-4			326	8.51	964	7.51
2	C	2-4			453	8.6	543	7.85
3	C	2-4			642	8.4	352	7.70
1	C	4-6			564	8.26	605	7.56
2	C	4-6			416	8.58	326	8.05
3	C	4-6			503	8.55	364	7.62
1	C1	0-2	3390	7.68	1862	8.17	2130	7.43
2	C1	0-2	5440	7.87	6330	8.11	5460	7.73
3	C1	0-2	5210	7.70	7070	8.16	937	7.44
1	C1	2-4			1034	8.32	3100	7.09
2	C1	2-4			1714	8.44	3900	7.56
3	C1	2-4			2430	8.22	366	7.53
1	C1	4-6			776	8.35	1543	7.22
2	C1	4-6			843	8.44	960	7.71
3	C1	4-6			1078	8.4	332	7.81
1	G1	0-2	4480	7.58	3970	8.06	3550	7.21
2	G1	0-2	8120	7.78	5130	8.19	3080	7.75
3	G1	0-2			5850	8.19	4742	7.54
1	G1	2-4			2900	8.14	3640	7.04
2	G1	2-4			3280	8.21	4830	7.55
3	G1	2-4			4650	8.14	3570	7.33
1	G1	4-6			2190	8.18	3060	7.14
2	G1	4-6			2780	8.28	3630	7.47
3	G1	4-6			2760	8.33	3020	7.50
1	G2	0-2	3860	7.44	3700	8.06	3200	7.25
2	G2	0-2	4000	7.57	5390	8.12	4180	7.62
3	G2	0-2	5580	7.68	4610	8.15	5339	7.50
1	G2	2-4			2290	8.07	3790	7.44
2	G2	2-4			2650	8.35	3550	7.53
3	G2	2-4			3790	8.23	4025	7.45
1	G2	4-6			1963	8.22	2200	7.59
2	G2	4-6			1998	8.44	3000	7.70
3	G2	4-6			2420	8.33	3367	7.47
1	G2M1	0-2	447	8.19	358	8.5	421	7.79
2	G2M1	0-2	5900	7.63	5610	8.19	4530	7.60

Table A1: Continued

			Day 7		Day 60		Day 170	
Rep	Samples	Depth (in)	EC (µS/cm)	pH	EC (µS/cm)	pH	EC (µS/cm)	pH
3	G2M1	0-2			5290	8.17	7005	7.42
1	G2M1	2-4			366	8.43	1002	7.32
2	G2M1	2-4			3540	8.23	4560	7.43
3	G2M1	2-4			5130	8.14	5010	7.43
1	G2M1	4-6			308	8.43	300	7.61
2	G2M1	4-6			2080	8.22	2730	7.48
3	G2M1	4-6			2650	8.26	3553	7.44
1	G2M2	0-2	3990	7.36	3200	8.06	2830	7.17
2	G2M2	0-2	5650	7.54	4570	8.12	2188	7.73
3	G2M2	0-2	8020	7.80	5540	8.15	5356	7.47
1	G2M2	2-4			1667	8.2	3640	7.09
2	G2M2	2-4			3390	8.17	4256	7.24
3	G2M2	2-4			5300	8.16	4885	7.41
1	G2M2	4-6			1164	8.25	2140	7.28
2	G2M2	4-6			1951	8.44	2150	7.28
3	G2M2	4-6			3230	8.35	3439	7.39
1	L1	0-2	3210	7.85	3070	8.13	2890	7.35
2	L1	0-2	4980	7.90	3620	8.09	3368	7.70
3	L1	0-2	5850	7.91	6310	8.06	4181	7.54
1	L1	2-4			1869	8.17	2880	7.33
2	L1	2-4			1849	8.31	2849	7.36
3	L1	2-4			2070	8.4	3115	7.44
1	L1	4-6			982	8.29	1828	7.24
2	L1	4-6			1264	8.45	1316	7.27
3	L1	4-6			1351	8.48	1485	7.63
1	L2	0-2	2190	7.76	1665	8.26	1766	7.47
2	L2	0-2	4680	7.88	3330	8.18	3243	7.56
3	L2	0-2	3370	7.84	2700	8.31	2644	7.50
1	L2	2-4			1334	8.3	2500	7.20
2	L2	2-4			2780	8.12	2043	7.58
3	L2	2-4			2740	8.21	2889	7.47
1	L2	4-6			739	8.39	1285	7.33
2	L2	4-6			1554	8.46	3002	7.45
3	L2	4-6			1137	8.41	1189	7.69

Table A.2: Soil TPH (mg/kg) at the OBM trial in Shattuck, OK.

			Day 0	Day 7	Day 60	Day 170
Rep	Treatment	Depth (in)	mg TPH/kg	mg TPH/kg	mg TPH/kg	mg TPH/kg
1	C	0-2	23	69.8	31.0	23.3
2	C	0-2	109		0.0	0.0
3	C	0-2	388	23.3	7.8	15.5
1	C	2-4		23.3	15.5	23.3
2	C	2-4			0.0	0.0
3	C	2-4		7.8	0.0	7.8
1	C	4-6		23.3	23.3	23.3
2	C	4-6			0.0	7.8
3	C	4-6		23.3	0.0	15.5
1	C1	0-2	1713		69.8	54.3
2	C1	0-2	2527	178.3	1085.3	511.6
3	C1	0-2	2729	325.6	4031.0	7.8
1	C1	2-4			38.8	46.5
2	C1	2-4		302.3	0.0	38.8
3	C1	2-4		85.3	31.0	7.8
1	C1	4-6			31.0	23.3
2	C1	4-6		736.4	31.0	46.5
3	C1	4-6		139.5	7.8	7.8
1	C2	0-2	2767	38.8	728.7	255.8
2	C2	0-2	3953		480.6	534.9
3	C2	0-2	155		0.0	7.8
1	C2	2-4		31.0	46.5	38.8
2	C2	2-4			23.3	38.8
3	C2	2-4			0.0	15.5
1	C2	4-6		23.3	46.5	54.3
2	C2	4-6			54.3	85.3
3	C2	4-6			0.0	7.8
1	G1	0-2	2109	93.0	519.4	162.8
2	G1	0-2	6589		1938.0	511.6
3	G1	0-2	1938	1643.4	2294.6	395.3
1	G1	2-4		31.0	23.3	31.0
2	G1	2-4			38.8	62.0
3	G1	2-4		100.8	93.0	85.3
1	G1	4-6		46.5	38.8	124.0
2	G1	4-6			38.8	147.3
3	G1	4-6		124.0	38.8	139.5
1	G2	0-2	2039	155.0	209.3	77.5

Table A2: Continued

Rep	Treatment	Depth (in)	Day 0 mg TPH/kg	Day 7 mg TPH/kg	Day 60 mg TPH/kg	Day 170 mg TPH/kg
2	G2	0-2	1783	333.3	38.8	155.0
3	G2	0-2	2860	550.4	1480.6	317.8
1	G2	2-4		46.5	23.3	15.5
2	G2	2-4		116.3	38.8	23.3
3	G2	2-4		116.3	69.8	108.5
1	G2	4-6		240.3	31.0	38.8
2	G2	4-6		108.5	62.0	38.8
3	G2	4-6		77.5	7.8	209.3
1	G2M1	0-2	23	85.3	0.0	15.5
2	G2M1	0-2	2178	387.6	604.7	302.3
3	G2M1	0-2	7054	1038.8	1852.7	248.1
1	G2M1	2-4		31.0	0.0	15.5
2	G2M1	2-4		170.5	23.3	23.3
3	G2M1	2-4		31.0	46.5	85.3
1	G2M1	4-6		116.3	0.0	23.3
2	G2M1	4-6		93.0	31.0	62.0
3	G2M1	4-6		93.0	38.8	116.3
1	G2M2	0-2	2070		69.8	54.3
2	G2M2	0-2	2791		209.3	0.0
3	G2M2	0-2	10155	2023.3	2403.1	162.8
1	G2M2	2-4			15.5	0.0
2	G2M2	2-4			23.3	0.0
3	G2M2	2-4		131.8	69.8	193.8
1	G2M2	4-6			23.3	0.0
2	G2M2	4-6			23.3	0.0
3	G2M2	4-6		186.0	46.5	193.8
1	L1	0-2	845		426.4	85.3
2	L1	0-2	1767	93.0	178.3	193.8
3	L1	0-2	4961	1217.1	1984.5	131.8
1	L1	2-4			38.8	7.8
2	L1	2-4		38.8	31.0	38.8
3	L1	2-4		100.8	77.5	69.8
1	L1	4-6			23.3	0.0
2	L1	4-6		31.0	46.5	62.0
3	L1	4-6		131.8	23.3	116.3
1	L2	0-2	915	31.0	85.3	0.0
2	L2	0-2	2016		263.6	379.8
3	L2	0-2	1488	387.6	945.7	62.0

Table A2: Continued

Rep	Treatment	Depth (in)	Day 0 mg TPH/kg	Day 7 mg TPH/kg	Day 60 mg TPH/kg	Day 170 mg TPH/kg
1	L2	2-4		23.3	23.3	0.0
2	L2	2-4			38.8	62.0
3	L2	2-4		31.0	23.3	46.5
1	L2	4-6		15.5	46.5	0.0
2	L2	4-6			131.8	7.8
3	L2	4-6		100.8	0.0	131.8

Table A.3: Saturated paste metal concentrations from the OBM study in Shattuck, OK.

			Day 7 (Metal concentrations; mg/L)													
Rep	Samples	Depth (in)	Na	Ca	Mg	K	SO4	B	P	Fe	Zn	Cu	Mn	Al	Ba	Cl
1	C	0-2	14.7	62.0	9.2	18.4	60.0	0.1	0.6	0.0	0.0	0.2	0.1	0.1	0.3	38.4
2	C	0-2														
3	C	0-2														
1	C	2-4														
2	C	2-4														
3	C	2-4														
1	C	4-6														
2	C	4-6														
3	C	4-6														
1	C1	0-2	100.5	650.0	58.9	29.4	813.9	0.1	0.4	0.0	0.0	0.0	0.0	0.0	0.3	641.5
2	C1	0-2	293.1	1102.1	68.6	54.0	740.8	0.1	0.5	0.0	0.0	0.0	0.5	0.0	0.5	1560.2
3	C1	0-2	179.7	891.0	95.4	37.7	933.3	0.1	0.4	0.0	0.0	0.0	0.1	0.0	0.4	1080.9
1	C1	2-4														
2	C1	2-4														
3	C1	2-4														
1	C1	4-6														
2	C1	4-6														
3	C1	4-6														
1	G1	0-2	119.2	913.2	84.8	25.7	1297.5	0.1	0.4	0.0	0.0	0.0	0.3	0.0	0.3	816.1
2	G1	0-2	403.1	1663.2	64.3	57.9	901.6	0.1	0.3	0.0	0.0	0.0	0.3	0.0	0.4	2333.9
3	G1	0-2														
1	G1	2-4														
2	G1	2-4														
3	G1	2-4														
1	G1	4-6														

Table A3: Continued

			Day 7 (Metal concentrations; mg/L)													
Rep	Samples	Depth (in)	Na	Ca	Mg	K	SO4	B	P	Fe	Zn	Cu	Mn	Al	Ba	Cl
2	G1	4-6														
3	G1	4-6														
1	G2	0-2	98.8	715.6	77.2	28.3	1007.1	0.0	0.4	0.0	0.0	0.0	0.1	0.0	0.3	656.7
2	G2	0-2	120.5	777.6	47.4	40.1	947.9	0.1	0.5	0.0	0.0	0.0	0.3	0.0	0.4	709.9
3	G2	0-2	186.5	981.4	95.4	42.4	1126.4	0.1	0.2	0.0	0.0	0.0	0.1	0.0	0.3	1166.6
1	G2	2-4														
2	G2	2-4														
3	G2	2-4														
1	G2	4-6														
2	G2	4-6														
3	G2	4-6														
1	G2M1	0-2	27.7	54.8	6.9	10.4	91.3	0.1	0.8	0.0	0.0	0.1	0.0	0.1	0.2	33.3
2	G2M1	0-2	224.6	1081.2	77.8	62.0	943.2	0.1	0.4	0.0	0.0	0.0	0.1	0.0	0.3	1356.5
3	G2M1	0-2														
1	G2M1	2-4														
2	G2M1	2-4														
3	G2M1	2-4														
1	G2M1	4-6														
2	G2M1	4-6														
3	G2M1	4-6														
1	G2M2	0-2	101.3	743.7	70.1	36.8	1107.0	0.1	0.5	0.0	0.0	0.0	0.0	0.0	0.3	661.7
2	G2M2	0-2	232.0	1157.3	68.6	65.8	1132.6	0.1	0.3	0.0	0.0	0.0	0.4	1.0	0.4	1376.3
3	G2M2	0-2	363.7	1335.8	80.5	71.6	1014.5	0.1	0.5	0.0	0.0	0.0	0.3	0.0	0.4	1703.9
1	G2M2	2-4														
2	G2M2	2-4														
3	G2M2	2-4														

Table A3: Continued

			Day 7 (Metal concentrations; mg/L)													
Rep	Samples	Depth (in)	Na	Ca	Mg	K	SO4	B	P	Fe	Zn	Cu	Mn	Al	Ba	Cl
1	G2M2	4-6														
2	G2M2	4-6														
3	G2M2	4-6														
1	L1	0-2	75.0	692.0	70.3	33.3	999.0	0.1	0.7	0.0	0.0	0.0	0.1	0.0	0.3	553.4
2	L1	0-2	176.6	933.1	62.4	40.4	866.8	0.1	0.4	0.0	0.0	0.0	0.1	0.0	0.4	944.6
3	L1	0-2	202.0	1050.9	86.8	55.4	762.5	0.1	0.8	0.0	0.0	0.0	0.4	0.0	0.5	1334.3
1	L1	2-4														
2	L1	2-4														
3	L1	2-4														
1	L1	4-6														
2	L1	4-6														
3	L1	4-6														
1	L2	0-2	57.8	352.8	38.2	21.1	355.9	0.1	0.3	0.0	0.0	0.1	0.1	0.0	0.2	441.8
2	L2	0-2	204.0	1010.0	42.9	40.6	1034.6	0.1	0.4	0.0	0.0	0.0	0.0	0.0	0.4	1090.6
3	L2	0-2	129.9	565.2	50.8	34.9	520.5	0.1	0.5	0.0	0.0	0.0	0.1	0.0	0.4	720.5
1	L2	2-4														
2	L2	2-4														
3	L2	2-4														
1	L2	4-6														
2	L2	4-6														
3	L2	4-6														

Table A3: Continued

Day 60 (metal concentrations; mg/L)

Rep	Samples	Depth (in)	Na	Ca	Mg	K	SO4	B	P	Fe	Zn	Cu	Mn	Al	Ba	Cl
1	C	0-2	40.0	70.2	8.7	12.2	75.7	0.1	0.5	0.0	0.0	0.0	0.0	0.0	0.3	53.5
2	C	0-2	9.0	52.8	5.4	12.0	46.9	0.0	0.5	0.0	0.0	0.0	0.0	0.0	0.3	15.5
3	C	0-2	18.1	135.1	11.0	21.7	152.4	0.0	1.3	0.0	0.0	0.0	0.0	0.0	0.4	72.4
1	C	2-4	14.9	46.4	7.3	7.9	23.2	0.1	0.8	0.0	0.0	0.0	0.0	0.1	0.2	31.3
2	C	2-4	6.1	41.2	5.5	9.6	28.4	0.0	0.5	0.0	0.0	0.0	0.0	0.0	0.3	15.1
3	C	2-4	5.7	109.1	7.9	15.3	127.7	0.0	0.9	0.0	0.0	0.0	0.0	0.0	0.5	25.0
1	C	4-6	12.7	110.1	15.9	9.5	193.8	0.1	0.6	0.0	0.0	0.0	0.1	0.0	0.5	22.2
2	C	4-6	9.2	44.7	6.9	9.5	30.1	0.0	0.6	0.0	0.0	0.0	0.0	0.0	0.4	14.6
3	C	4-6	4.4	60.3	6.5	11.3	26.8	0.0	0.8	0.0	0.0	0.0	0.0	0.0	0.4	10.0
1	C1	0-2	60.2	331.4	35.4	22.4	535.5	0.0	0.2	0.0	0.0	0.0	0.2	0.0	0.4	292.3
2	C1	0-2	343.1	1200.5	70.2	48.9	738.8	0.0	0.4	0.0	0.0	0.0	0.6	0.0	0.5	1723.8
3	C1	0-2	349.3	1248.3	102.9	45.2	1113.6	0.1	0.5	0.0	0.0	0.0	0.8	0.0	0.5	1569.1
1	C1	2-4	14.6	181.7	21.5	15.4	150.0	0.0	0.3	0.0	0.0	0.0	0.1	0.0	0.7	211.9
2	C1	2-4	44.0	247.6	20.8	21.2	92.4	0.0	0.3	0.0	0.0	0.0	0.0	0.0	0.8	369.2
3	C1	2-4	70.3	373.6	46.5	23.4	181.7	0.0	0.3	0.0	0.0	0.0	0.1	0.0	0.8	567.8
1	C1	4-6	10.9	158.4	19.7	11.0	293.1	0.0	0.4	0.0	0.0	0.0	0.1	0.0	0.6	45.8
2	C1	4-6	15.4	131.2	13.0	12.7	69.1	0.0	0.5	0.0	0.0	0.0	0.0	0.0	0.5	143.9
3	C1	4-6	25.0	155.1	25.0	14.6	85.7	0.0	0.5	0.0	0.0	0.0	0.0	0.0	0.5	248.1
1	G1	0-2	123.6	829.8	84.5	33.8	1336.9	0.0	0.4	0.0	0.0	0.0	0.7	0.0	0.4	593.4
2	G1	0-2	226.5	1121.5	48.3	52.4	1150.0	0.1	0.4	0.0	0.0	0.0	0.4	0.0	0.5	1011.7
3	G1	0-2	225.5	1167.3	54.9	83.6	1309.5	0.1	0.7	0.0	0.0	0.0	0.5	0.0	0.6	1047.7
1	G1	2-4	34.0	492.5	66.5	20.0	700.4	0.0	0.3	0.0	0.0	0.0	0.5	0.0	0.2	427.5
2	G1	2-4	60.1	795.1	36.7	27.7	1100.6	0.0	0.3	0.0	0.0	0.0	0.1	0.0	0.3	512.4
3	G1	2-4	95.4	1030.3	52.9	43.3	1082.3	0.0	0.8	0.0	0.0	0.0	0.3	0.0	0.4	811.8
1	G1	4-6	15.3	572.9	67.7	17.9	1289.5	0.1	0.2	0.0	0.0	0.0	0.4	0.0	0.2	197.0

Table A3:Continued

Day 60 (metal concentrations; mg/L)																	
Rep	Samples	Depth (in)	Na	Ca	Mg	K	SO4	B	P	Fe	Zn	Cu	Mn	Al	Ba	Cl	
2	G1	4-6	26.6	745.7	40.2	22.9	1337.1	0.0	0.3	0.0	0.0	0.0	0.0	0.0	0.3	334.0	
3	G1	4-6	20.2	661.5	37.3	28.4	964.9	0.0	0.5	0.0	0.0	0.0	0.2	0.0	0.3	384.2	
1	G2	0-2	122.9	798.1	79.4	32.0	1342.9	0.0	0.3	0.0	0.0	0.0	0.7	0.0	0.4	562.6	
2	G2	0-2	223.3	987.6	62.1	37.6	1403.2	0.0	0.8	0.0	0.0	0.0	0.8	0.0	0.3	816.8	
3	G2	0-2	191.7	944.4	90.7	45.1	1279.2	0.0	0.4	0.0	0.0	0.0	0.5	0.0	0.5	866.7	
1	G2	2-4	27.7	477.3	53.6	19.8	664.2	0.0	0.3	0.0	0.0	0.0	0.6	0.0	0.3	401.4	
2	G2	2-4	51.1	613.6	35.2	22.1	745.5	0.0	0.2	0.0	0.0	0.0	0.1	0.0	0.4	472.9	
3	G2	2-4	77.9	765.3	90.2	29.8	1179.5	0.0	0.5	0.0	0.0	0.1	0.3	0.0	0.2	670.2	
1	G2	4-6	15.0	499.9	57.5	18.4	1148.7	0.1	0.4	0.0	0.0	0.0	0.5	0.0	0.2	132.7	
2	G2	4-6	18.1	492.1	35.3	17.8	892.8	0.0	0.5	0.0	0.0	0.0	0.1	0.0	0.3	227.1	
3	G2	4-6	40.2	513.6	65.1	21.1	1031.8	0.0	0.5	0.0	0.0	0.0	0.1	0.0	0.2	293.9	
1	G2M1	0-2	9.5	73.2	8.8	12.0	60.7	0.0	0.7	0.0	0.0	0.0	0.0	0.0	0.4	33.2	
2	G2M1	0-2	263.9	1149.5	77.1	65.6	1282.9	0.0	0.4	0.0	0.0	0.0	0.9	0.0	0.4	1122.7	
3	G2M1	0-2	233.6	1082.4	60.5	57.1	1237.1	0.1	0.5	0.0	0.0	0.0	0.5	0.0	0.6	1004.6	
1	G2M1	2-4	7.9	67.8	8.6	8.1	40.6	0.0	0.4	0.0	0.0	0.0	0.0	0.0	0.3	16.1	
2	G2M1	2-4	75.1	763.1	53.8	30.4	938.4	0.0	0.3	0.0	0.0	0.0	0.5	0.0	0.3	627.3	
3	G2M1	2-4	119.4	1114.8	71.0	34.9	982.1	0.0	0.4	0.0	0.0	0.0	0.5	0.0	0.3	1053.7	
1	G2M1	4-6	4.9	41.8	6.9	5.4	19.5	0.0	0.3	0.0	0.0	0.0	0.0	0.0	0.3	10.2	
2	G2M1	4-6	23.4	531.7	42.2	20.5	1054.4	0.0	0.5	0.0	0.0	0.0	0.2	0.0	0.2	234.0	
3	G2M1	4-6	23.5	625.5	48.0	23.9	890.0	0.0	0.5	0.0	0.0	0.0	0.3	0.0	0.2	386.2	
1	G2M2	0-2	95.4	728.3	62.8	40.2	1360.4	0.0	0.5	0.0	0.0	0.0	0.6	0.0	0.4	432.3	
2	G2M2	0-2	212.2	857.3	59.4	42.6	1241.8	0.0	0.5	0.0	0.0	0.0	0.4	0.0	0.4	2.3	
3	G2M2	0-2	254.0	1022.2	67.5	67.0	1221.6	0.1	0.3	0.0	0.0	0.0	0.6	0.0	0.4	1056.7	
1	G2M2	2-4	20.7	378.7	33.9	18.0	534.5	0.0	0.4	0.0	0.0	0.0	0.2	0.0	0.5	275.3	
2	G2M2	2-4	60.7	780.4	48.8	25.8	846.5	0.0	0.3	0.0	0.0	0.0	0.3	0.0	0.3	609.9	

Table A3: Continued

Day 60 (metal concentrations; mg/L)

Rep	Samples	Depth (in)	Na	Ca	Mg	K	SO4	B	P	Fe	Zn	Cu	Mn	Al	Ba	Cl
3	G2M2	2-4	152.0	1163.5	77.3	34.9	1116.7	0.0	0.3	0.0	0.0	0.0	0.5	0.0	0.3	1050.0
1	G2M2	4-6	7.9	276.2	26.9	13.5	556.8	0.0	0.5	0.0	0.0	0.0	0.1	0.0	0.3	68.2
2	G2M2	4-6	15.1	544.8	44.0	19.4	1080.8	0.0	0.6	0.0	0.0	0.0	0.2	0.0	0.2	187.5
3	G2M2	4-6	38.4	679.6	55.3	23.2	1071.5	0.0	0.4	0.0	0.0	0.0	0.2	0.0	0.3	593.2
1	L1	0-2	101.3	678.8	69.8	30.7	1124.6	0.0	0.2	0.0	0.0	0.0	0.3	0.0	0.4	560.6
2	L1	0-2	161.8	730.2	56.2	38.8	938.2	0.0	0.2	0.0	0.0	0.0	0.5	0.0	0.4	643.0
3	L1	0-2	254.3	1202.9	105.9	62.8	1014.3	0.0	0.4	0.0	0.0	0.0	0.9	0.0	0.6	1660.3
1	L1	2-4	30.4	316.8	39.9	18.6	266.8	0.0	0.2	0.0	0.0	0.0	0.3	0.0	0.6	397.4
2	L1	2-4	36.8	320.3	31.1	21.4	212.7	0.0	0.2	0.0	0.0	0.0	0.2	0.0	0.6	396.8
3	L1	2-4	49.5	304.5	39.3	29.8	163.9	0.0	0.5	0.0	0.0	0.0	0.2	0.0	0.7	481.6
1	L1	4-6	12.7	159.9	23.1	11.7	157.0	0.0	0.2	0.0	0.0	0.0	0.0	0.0	0.7	166.1
2	L1	4-6	27.1	218.0	25.7	17.4	192.2	0.0	0.5	0.0	0.0	0.0	0.1	0.0	0.6	244.8
3	L1	4-6	25.8	183.5	30.7	23.0	143.7	0.1	0.6	0.0	0.0	0.1	0.1	0.0	0.6	284.1
1	L2	0-2	69.1	307.9	32.4	22.0	322.3	0.1	0.2	0.0	0.0	0.0	0.1	0.0	0.6	386.4
2	L2	0-2	169.2	606.3	28.7	30.6	807.5	0.0	0.2	0.0	0.0	0.0	0.0	0.0	0.5	602.1
3	L2	0-2	114.6	511.4	44.6	34.4	835.1	0.0	0.4	0.0	0.0	0.0	0.2	0.0	0.4	468.6
1	L2	2-4	20.9	215.6	27.4	16.4	104.4	0.1	0.3	0.0	0.0	0.0	0.1	0.0	0.8	389.1
2	L2	2-4	64.4	559.8	29.9	21.7	420.4	0.0	0.2	0.0	0.0	0.0	0.1	0.0	0.6	622.1
3	L2	2-4	55.4	419.3	46.9	23.2	259.0	0.0	0.4	0.0	0.0	0.0	0.2	0.0	0.5	620.7
1	L2	4-6	15.2	123.2	20.7	11.0	115.6	0.1	0.3	0.0	0.0	0.0	0.0	0.0	0.6	121.7
2	L2	4-6	22.1	294.7	22.6	17.2	283.4	0.0	0.5	0.0	0.0	0.0	0.0	0.0	0.6	277.7
3	L2	4-6	19.5	153.3	23.6	13.6	74.0	0.0	0.8	0.0	0.0	0.0	0.1	0.0	0.6	219.6

Table A3: Continued

Day 170 (metal concentrations; mg/L)																	
Rep	Samples	Depth (in)	Na	Ca	Mg	K	SO4	B	P	Fe	Zn	Cu	Mn	Al	Ba	Cl
1	C	0-2	20.6	166.8	19.1	11.8	152.8	0.0	0.5	0.0	0.0	0.0	0.1	0.0	0.4	184.1
2	C	0-2	10.7	119.5	8.9	14.8	78.2	0.0	1.4	0.0	0.0	0.0	0.1	0.0	0.3	68.9
3	C	0-2	6.5	88.8	6.6	13.3	76.0	0.0	1.4	0.0	0.0	0.0	0.1	0.0	0.3	38.3
1	C	2-4	14.0	158.3	18.4	10.0	104.6	0.0	0.4	0.0	0.0	0.0	0.1	0.0	0.4	175.5
2	C	2-4	7.1	92.5	8.0	12.6	70.0	0.0	1.2	0.0	0.0	0.0	0.0	0.0	0.3	62.0
3	C	2-4	5.2	64.1	5.6	7.6	36.6	0.0	1.6	0.0	0.0	0.0	0.0	0.0	0.2	26.0
1	C	4-6	9.1	89.0	12.4	8.0	42.5	0.1	0.4	0.0	0.0	0.0	0.1	0.0	0.3	101.4
2	C	4-6	6.8	56.5	5.8	7.4	25.2	0.0	1.4	0.0	0.0	0.0	0.0	0.0	0.2	20.9
3	C	4-6	5.3	63.3	6.8	6.9	54.1	0.0	2.2	0.0	0.0	0.0	0.0	0.0	0.3	14.6
1	C1	0-2	91.7	323.3	34.7	24.4	605.0	0.0	0.2	0.0	0.0	0.0	0.1	0.0	0.2	264.1
2	C1	0-2	268.5	854.8	60.4	43.8	658.0	0.0	1.1	0.0	0.0	0.0	0.7	0.0	0.3	1105.0
3	C1	0-2	34.1	87.8	16.2	10.3	162.5	0.0	0.6	0.0	0.0	0.0	0.0	0.0	0.2	41.9
1	C1	2-4	76.7	539.3	53.8	19.4	484.2	0.0	0.3	0.0	0.0	0.0	0.3	0.0	0.3	625.7
2	C1	2-4	65.7	482.9	37.3	24.3	142.3	0.0	1.1	0.0	0.0	0.0	0.2	0.0	0.7	710.6
3	C1	2-4	18.6	20.7	4.2	2.7	24.7	0.0	0.7	0.0	0.0	0.0	0.0	0.0	0.1	17.2
1	C1	4-6	20.4	240.2	29.3	13.0	100.3	0.0	0.4	0.0	0.0	0.0	0.1	0.0	0.6	341.7
2	C1	4-6	12.3	130.2	13.6	12.8	59.0	0.0	1.0	0.0	0.0	0.0	0.0	0.0	0.3	158.8
3	C1	4-6	46.1	26.5	6.2	4.3	40.0	0.0	0.9	0.0	0.0	0.0	0.0	0.0	0.1	14.9
1	G1	0-2	122.3	596.1	80.8	27.7	1036.5	0.0	0.3	0.0	0.0	0.0	0.4	0.0	0.2	532.8
2	G1	0-2	116.0	629.2	27.8	38.8	1089.5	0.0	0.7	0.0	0.0	0.0	0.2	0.0	0.1	409.0
3	G1	0-2	142.6	846.9	46.0	53.0	1170.0	0.0	1.0	0.0	0.0	0.0	0.3	0.0	0.3	653.8
1	G1	2-4	98.7	665.7	84.5	20.7	1034.7	0.0	0.3	0.0	0.0	0.0	0.4	0.0	0.2	558.3
2	G1	2-4	134.4	936.7	48.7	25.0	790.1	0.0	0.8	0.0	0.0	0.0	0.2	0.0	0.4	1059.0
3	G1	2-4	48.1	854.1	49.5	33.0	1105.5	0.0	1.0	0.0	0.0	0.0	0.1	0.0	0.4	605.8
1	G1	4-6	53.0	418.1	86.4	18.1	683.8	0.0	0.4	0.0	0.0	0.0	0.5	0.0	0.1	458.9
2	G1	4-6	39.1	805.5	48.3	20.3	928.9	0.0	0.8	0.0	0.0	0.0	0.2	0.0	0.2	652.1

Table A3: Continued

Day 170 (metal concentrations; mg/L)																	
Rep	Samples	Depth (in)	Na	Ca	Mg	K	SO4	B	P	Fe	Zn	Cu	Mn	Al	Ba	Cl
3	G1	4-6	14.5	637.6	47.4	20.8	1323.6	0.0	1.4	0.0	0.0	0.0	0.1	0.0	0.3	188.6
1	G2	0-2	115.9	527.8	68.0	24.9	1058.7	0.0	0.3	0.0	0.0	0.0	0.4	0.0	0.2	437.3
2	G2	0-2	138.2	688.7	55.3	34.6	934.3	0.0	1.1	0.0	0.0	0.0	0.4	0.0	0.2	629.8
3	G2	0-2	219.7	984.6	100.6	39.8	1166.4	0.0	1.0	0.0	0.0	0.0	1.0	0.0	0.3	1006.2
1	G2	2-4	89.6	664.3	76.2	21.3	726.5	0.0	0.4	0.0	0.0	0.0	0.4	0.0	0.3	744.1
2	G2	2-4	68.9	678.2	40.2	22.7	627.2	0.0	0.9	0.0	0.0	0.1	0.2	0.0	0.4	665.3
3	G2	2-4	105.3	802.2	105.7	26.0	1099.6	0.0	1.1	0.0	0.0	0.0	0.2	0.0	0.4	717.8
1	G2	4-6	30.1	350.6	50.1	15.7	502.9	0.0	0.4	0.0	0.0	0.0	0.3	0.0	0.2	345.0
2	G2	4-6	19.3	556.8	36.7	18.6	731.1	0.0	0.9	0.0	0.0	0.0	0.1	0.0	0.3	419.7
3	G2	4-6	62.0	633.8	93.9	19.4	1420.8	0.0	0.9	0.0	0.0	0.0	0.1	0.0	0.3	314.0
1	G2M1	0-2	7.5	54.3	7.9	9.2	50.9	0.0	0.3	0.0	0.0	0.0	0.0	0.0	0.2	23.8
2	G2M1	0-2	170.2	805.3	66.1	47.1	1028.3	0.0	1.1	0.0	0.0	0.0	1.0	0.0	0.2	797.7
3	G2M1	0-2	247.8	1057.9	73.9	43.7	1075.8	0.0	1.4	0.0	0.0	0.0	0.9	0.0	0.2	1114.5
1	G2M1	2-4	8.8	56.5	7.2	170.3	34.0	0.1	0.5	0.0	0.1	0.1	0.1	0.2	0.2	185.6
2	G2M1	2-4	85.2	798.3	57.2	28.6	764.0	0.0	1.0	0.0	0.0	0.0	0.4	0.0	0.3	848.3
3	G2M1	2-4	78.0	836.1	62.9	24.2	997.7	0.0	0.9	0.0	0.0	0.0	0.2	0.0	0.4	701.3
1	G2M1	4-6	6.0	45.8	6.9	7.0	21.7	0.0	0.4	0.0	0.0	0.0	0.0	0.0	0.2	18.1
2	G2M1	4-6	22.7	647.6	51.5	22.8	1122.9	0.0	1.1	0.0	0.0	0.0	0.2	0.0	0.2	357.6
3	G2M1	4-6	20.0	475.4	63.6	18.6	836.7	0.0	1.2	0.0	0.0	0.0	0.1	0.0	0.2	286.6
1	G2M2	0-2	93.6	560.9	58.3	29.9	1206.8	0.0	0.4	0.0	0.0	0.0	0.3	0.0	0.2	318.8
2	G2M2	0-2	156.6	698.8	52.9	41.6	1098.0	0.0	0.6	0.0	0.0	0.0	0.5	0.0	0.1	598.6
3	G2M2	0-2	174.0	834.3	65.6	50.2	1167.0	0.0	0.9	0.0	0.0	0.0	0.6	0.0	0.2	727.5
1	G2M2	2-4	81.9	696.0	66.8	21.5	847.7	0.0	0.5	0.0	0.0	0.0	0.4	0.0	0.3	658.9
2	G2M2	2-4	105.1	803.2	52.1	23.4	984.4	0.0	0.6	0.0	0.0	0.0	0.5	0.0	0.3	736.2
3	G2M2	2-4	80.9	910.1	71.2	23.7	1171.8	0.0	0.9	0.0	0.0	0.0	0.3	0.0	0.4	701.7
1	G2M2	4-6	24.0	434.5	46.1	16.3	596.2	0.0	0.5	0.0	0.0	0.0	0.2	0.0	0.2	372.4

Table A3: Continued

Day 170 (metal concentrations; mg/L)																	
Rep	Samples	Depth (in)	Na	Ca	Mg	K	SO4	B	P	Fe	Zn	Cu	Mn	Al	Ba	Cl
2	G2M2	4-6	35.4	827.5	55.3	21.6	1196.5	0.0	0.6	0.0	0.0	0.0	0.3	0.0	0.3	523.7
3	G2M2	4-6	24.3	702.4	66.9	19.0	1444.0	0.0	1.3	0.0	0.0	0.0	0.1	0.0	0.3	279.4
1	L1	0-2	113.3	581.5	66.0	28.3	910.7	0.0	0.4	0.0	0.0	0.0	0.6	0.0	0.2	474.7
2	L1	0-2	167.7	717.5	57.4	35.0	840.0	0.0	0.6	0.0	0.0	0.0	0.7	0.0	0.2	707.8
3	L1	0-2	128.8	673.4	66.8	42.9	767.5	0.0	1.1	0.0	0.0	0.0	0.6	0.0	0.2	658.4
1	L1	2-4	95.6	546.6	64.6	21.2	546.0	0.0	0.4	0.0	0.0	0.0	0.4	0.0	0.3	674.9
2	L1	2-4	80.0	577.0	44.1	21.7	368.9	0.0	0.6	0.0	0.0	0.0	0.4	0.0	0.5	671.4
3	L1	2-4	57.3	442.0	52.1	25.8	308.0	0.0	1.1	0.0	0.0	0.0	0.2	0.0	0.5	541.4
1	L1	4-6	35.2	300.7	39.6	15.9	133.5	0.0	0.3	0.0	0.0	0.0	0.2	0.0	0.6	452.0
2	L1	4-6	23.4	323.7	29.5	16.9	151.3	0.0	0.7	0.0	0.0	0.0	0.1	0.0	0.6	396.1
3	L1	4-6	19.9	199.7	29.8	14.6	205.9	0.0	1.0	0.0	0.0	0.0	0.0	0.0	0.4	208.2
1	L2	0-2	57.9	289.4	31.4	18.1	366.1	0.0	0.4	0.0	0.0	0.0	0.1	0.0	0.3	306.8
2	L2	0-2	142.1	657.1	32.1	30.3	830.1	0.0	1.3	0.0	0.0	0.0	0.2	0.0	0.3	565.9
3	L2	0-2	89.3	398.4	40.0	23.2	574.4	0.0	0.9	0.0	0.0	0.0	0.1	0.0	0.2	348.5
1	L2	2-4	42.6	407.8	45.1	15.8	288.0	0.0	0.6	0.0	0.0	0.0	0.1	0.0	0.5	513.2
2	L2	2-4	25.1	345.8	22.4	16.5	263.0	0.0	1.3	0.0	0.0	0.0	0.1	0.0	0.6	334.6
3	L2	2-4	50.9	370.1	48.5	15.8	261.8	0.0	1.0	0.0	0.0	0.0	0.1	0.0	0.6	482.7
1	L2	4-6	17.4	192.9	25.3	13.1	104.9	0.0	0.5	0.0	0.0	0.0	0.1	0.0	0.5	274.5
2	L2	4-6	89.9	664.1	34.5	22.9	465.0	0.0	1.2	0.0	0.0	0.0	0.1	0.0	0.5	714.7
3	L2	4-6	38.4	138.0	24.9	9.8	161.6	0.0	1.0	0.0	0.0	0.0	0.0	0.0	0.4	164.6

Table A4: Forage (biomass) yield 60 days after OBM application at Shattuck, OK.

Rep	Treatments	Subsample	Kg/ha
1	C	1	1234
1	C	2	1103
1	C	3	2099
2	C	1	656
2	C	2	537
2	C	3	1011
3	C	1	935
3	C	2	1859
3	C	3	1334
1	C1	1	1053
1	C1	2	835
1	C1	3	1181
2	C1	1	585
2	C1	2	1075
2	C1	3	820
3	C1	1	780
3	C1	2	1967
3	C1	3	897
1	G1	1	1022
1	G1	2	1082
1	G1	3	775
2	G1	1	1530
2	G1	2	930
2	G1	3	1289
3	G1	1	1785
3	G1	2	479
3	G1	3	779
1	G2	1	966
1	G2	2	948
1	G2	3	1521
2	G2	1	684
2	G2	2	1109
2	G2	3	1149
3	G2	1	1354
3	G2	2	1202
3	G2	3	1260
1	G2M1	1	860
1	G2M1	2	1688

Table A4: Continued

Rep	Treatments	Subsample	Kg/ha
1	G2M1	3	867
2	G2M1	1	1686
2	G2M1	2	894
2	G2M1	3	1541
3	G2M1	1	728
3	G2M1	2	1154
3	G2M1	3	397
1	G2M2	1	1241
1	G2M2	2	900
1	G2M2	3	1159
2	G2M2	1	1631
2	G2M2	2	1169
2	G2M2	3	1190
3	G2M2	1	857
3	G2M2	2	1117
3	G2M2	3	687
1	L1	1	953
1	L1	2	1783
1	L1	3	997
2	L1	1	1012
2	L1	2	952
2	L1	3	1587
3	L1	1	708
3	L1	2	3565
3	L1	3	1393
1	L2	1	1214
1	L2	2	1623
1	L2	3	1801
2	L2	1	707
2	L2	2	732
2	L2	3	1537
3	L2	1	979
3	L2	2	3865
3	L2	3	1364

APPENDIX B

Soil data collected at the Lahoma, OK field trial where water-base mud (WBM) was applied to winter wheat at five different application dates (Oct, Dec, Jan, Feb, or March) at two rates which were equivalent to 6,721 and 4,480 kg TSS ha^{-1} soil, respectively. Soil samples were analyzed for pH, EC, and metals at 0 (immediately after), 30, and 90 days after WBM application, and on August 28th, 2013 (Post-harvest).Wheat was harvested on June 20th, 2013.

Table B1: Saturated paste soil EC and pH for the WBM trial in Lahoma, OK.

						Day 0		Day 30		Day 90		Post-harvest	
Rep	Sample	App. Date	Rate	Trt	Depth (in)	pH	EC (mS/cm)	pH	EC (mS/cm)	pH	EC (mS/cm)	pH	EC (mS/cm)
1	111	Cont	0X	1	0-3	7.5	0.66	.	.	7.3	0.99	7.9	0.61
2	207	Cont	0X	1	0-3	7.3	1.13	.	.	7.5	1.06	8.0	0.65
3	310	Cont	0X	1	0-3	7.4	0.92	.	.	7.3	1.03	8.1	0.64
1	111	Cont	0X	1	3-6	7.7	0.47	.	.	7.6	0.65	8.1	0.62
2	207	Cont	0X	1	3-6	7.5	0.63	.	.	7.9	0.45	8.2	0.54
3	310	Cont	0X	1	3-6	7.7	0.59	.	.	7.5	0.52	8.1	0.61
1	102	16-Oct	1X	2	0-3	6.9	9.24	.	.	5.9	18.11	7.4	2.72
2	208	16-Oct	1X	2	0-3	6.0	18.53	.	.	6.9	12.19	7.9	4.33
3	305	16-Oct	1X	2	0-3	6.6	16.12	.	.	6.3	24.93	7.5	4.97
1	102	16-Oct	1X	2	3-6	8.0	1.48	.	.	7.0	2.78	7.8	2.82
2	208	16-Oct	1X	2	3-6	6.8	1.72	.	.	7.4	1.68	7.9	4.84
3	305	16-Oct	1X	2	3-6	7.0	1.53	.	.	6.6	4.23	7.3	4.34
1	110	16-Oct	0.66X	3	0-3	7.3	5.77	.	.	6.8	16.13	8.1	2.60
2	209	16-Oct	0.66X	3	0-3	7.0	9.65	.	.	6.9	7.17	8.0	2.10
3	306	16-Oct	0.66X	3	0-3	7.0	7.67	.	.	6.9	9.98	7.7	2.09
1	110	16-Oct	0.66X	3	3-6	7.6	0.90	.	.	7.5	2.96	8.0	3.92
2	209	16-Oct	0.66X	3	3-6	7.7	1.03	.	.	7.3	0.94	7.9	2.41
3	306	16-Oct	0.66X	3	3-6	7.4	1.07	.	.	7.5	1.06	7.9	2.21
1	103	6-Dec	1X	4	0-3	6.6	19.98	6.4	17.70	7.3	8.18	7.9	3.66
2	201	6-Dec	1X	4	0-3	8.6	10.30	6.1	21.09	6.3	9.81	6.7	5.77
3	309	6-Dec	1X	4	0-3	6.7	15.98	7.1	21.48	7.3	5.71	8.0	6.40
1	103	6-Dec	1X	4	3-6	7.2	3.93	7.5	1.37	7.0	4.89	8.0	3.48
2	201	6-Dec	1X	4	3-6	6.4	3.31	6.7	0.75	6.3	5.87	6.9	4.84
3	309	6-Dec	1X	4	3-6	7.8	1.31	7.8	1.06	7.2	7.43	8.0	5.01
1	109	6-Dec	0.66X	5	0-3	7.0	11.25	7.0	14.71	7.3	8.18	8.2	2.44
2	206	6-Dec	0.66X	5	0-3	7.0	17.75	7.1	16.51	7.2	6.48	8.2	3.61
3	302	6-Dec	0.66X	5	0-3	6.0	9.69	5.9	14.49	6.4	5.78	6.6	2.72

Table B1: Continued

						Day 0		Day 30		Day 90		Post-harvest	
Rep	Sample	App. Date	Rate	Trt	Depth (in)	pH	EC (mS/cm)	pH	EC (mS/cm)	pH	EC (mS/cm)	pH	EC (mS/cm)
1	109	6-Dec	0.66X	5	3-6	7.5	1.80	7.6	0.75	7.0	4.89	8.1	2.47
2	206	6-Dec	0.66X	5	3-6	7.7	0.85	7.7	1.69	7.2	6.34	7.8	3.53
3	302	6-Dec	0.66X	5	3-6	6.1	0.71	6.4	0.66	6.2	5.22	6.6	2.96
1	104	14-Jan	1X	6	0-3	6.8	15.99	6.9	18.51	7.4	3.76	8.0	6.17
2	203	14-Jan	1X	6	0-3	6.5	17.88	6.0	26.77	6.7	5.89	6.8	6.02
3	304	14-Jan	1X	6	0-3	6.3	18.31	6.1	18.55	6.0	12.74	7.1	7.24
1	104	14-Jan	1X	6	3-6	7.9	0.93	7.7	1.09	7.1	5.28	8.1	3.43
2	203	14-Jan	1X	6	3-6	7.6	1.28	6.6	2.33	6.5	6.65	7.2	4.69
3	304	14-Jan	1X	6	3-6	7.2	1.95	6.7	1.66	6.8	11.72	6.6	6.62
1	106	14-Jan	0.66X	7	0-3	7.1	9.35	7.5	8.00	7.6	2.30	8.2	0.93
2	205	14-Jan	0.66X	7	0-3	7.1	5.69	7.1	6.57	6.8	2.95	8.1	1.56
3	301	14-Jan	0.66X	7	0-3	5.7	9.99	5.9	15.43	6.4	7.46	6.6	4.30
1	106	14-Jan	0.66X	7	3-6	7.6	0.80	7.8	0.84	7.3	3.05	8.3	0.90
2	205	14-Jan	0.66X	7	3-6	7.8	0.97	7.7	0.64	7.2	2.24	8.1	1.79
3	301	14-Jan	0.66X	7	3-6	6.2	0.95	5.9	1.66	5.9	7.58	6.0	3.83
1	108	15-Feb	1X	8	0-3	6.9	16.01	7.6	7.64	7.1	8.48	8.1	4.09
2	202	15-Feb	1X	8	0-3	6.2	14.43	6.6	4.60	6.7	3.58	7.2	2.56
3	308	15-Feb	1X	8	0-3	7.0	24.66	7.4	9.18	7.2	11.38	8.0	5.62
1	108	15-Feb	1X	8	3-6	7.6	1.14	7.4	5.93	7.2	5.42	8.1	4.17
2	202	15-Feb	1X	8	3-6	7.5	2.05	6.5	2.59	7.3	3.43	7.2	2.46
3	308	15-Feb	1X	8	3-6	7.6	1.41	7.0	9.34	7.3	9.26	8.0	5.91
1	101	15-Feb	0.66X	9	0-3	6.1	10.53	7.0	6.02	6.5	3.05	7.0	3.23
2	210	15-Feb	0.66X	9	0-3	7.1	11.76	7.7	6.01	7.6	9.60	8.0	4.69
3	303	15-Feb	0.66X	9	0-3	5.9	14.67	6.1	8.52	6.4	5.21	6.3	3.95
1	101	15-Feb	0.66X	9	3-6	6.2	1.51	7.1	3.78	6.4	3.18	7.4	1.69
2	210	15-Feb	0.66X	9	3-6	7.6	1.14	7.4	5.60	7.2	8.41	7.9	4.08
3	303	15-Feb	0.66X	9	3-6	6.6	1.50	6.3	5.19	6.5	6.11	6.8	3.24

Table B1: Continued

						Day 0		Day 30		Day 90		Post-harvest	
Rep	Sample	App. Date	Rate	Trt	Depth (in)	pH	EC (mS/cm)	pH	EC (mS/cm)	pH	EC (mS/cm)	pH	EC (mS/cm)
1	105	20-Mar	1X	10	0-3	6.8	18.67	7.3	4.38	7.8	5.20	8.2	3.30
2	211	20-Mar	1X	10	0-3	6.9	14.04	7.1	3.48	7.7	4.44	8.0	3.09
3	307	20-Mar	1X	10	0-3	6.8	17.11	7.0	9.32	7.3	14.76	7.9	3.65
1	105	20-Mar	1X	10	3-6	7.6	1.53	7.3	3.30	7.6	4.65	8.1	2.76
2	211	20-Mar	1X	10	3-6	7.7	0.89	7.2	3.53	7.4	4.24	7.9	3.56
3	307	20-Mar	1X	10	3-6	7.6	0.97	7.3	6.93	7.5	9.04	7.9	4.98
1	107	20-Mar	0.66X	11	0-3	7.4	7.95	7.2	4.44	7.6	1.68	8.1	1.29
2	204	20-Mar	0.66X	11	0-3	6.7	10.91	7.3	5.29	7.1	2.99	7.6	2.50
3	311	20-Mar	0.66X	11	0-3	7.0	6.93	7.3	6.78	7.4	10.74	7.9	4.40
1	107	20-Mar	0.66X	11	3-6	7.8	0.93	7.3	2.40	7.5	2.09	8.1	1.46
2	204	20-Mar	0.66X	11	3-6	7.4	0.85	7.2	5.29	7.4	3.72	7.7	2.48
3	311	20-Mar	0.66X	11	3-6	7.2	0.83	7.4	3.37	7.4	4.57	8.0	3.01

Table B2: Saturated paste metal concentrations from the WBM study in Lahoma, OK.

				Day 0 (Metal Concentrations; mg/L)											
Rep	App. Date	Trt	Depth (in)	Na	Ca	Mg	K	S-SO_4	B	P	Fe	Zn	Cu	Mn	Al
1	Cont	1	0-3	18.9	47.8	22.7	5.8	24.5	0.0	0.4	0.0	0.0	0.0	0.0	0.0
2	Cont	1	0-3	47.6	98.1	37.3	7.8	158.7	0.0	0.5	0.0	0.0	0.0	0.0	0.0
3	Cont	1	0-3	26.9	76.8	31.0	6.9	33.9	0.0	0.7	0.0	0.0	0.0	0.0	0.0
1	16-Oct	2	0-3	673.5	406.1	206.2	15.8	79.1	0.0	0.4	0.0	0.2	0.1	0.1	0.0
2	16-Oct	2	0-3	1627.7	612.1	288.9	31.5	216.9	0.1	0.4	0.0	0.0	0.0	1.5	0.0
3	16-Oct	2	0-3	1538.1	698.9	283.6	21.6	92.3	0.0	0.5	0.0	0.0	0.1	0.2	0.0
1	16-Oct	3	0-3	390.6	282.7	147.8	11.9	52.7	0.0	0.4	0.0	0.0	0.1	0.0	0.0
2	16-Oct	3	0-3	766.9	453.4	174.4	13.5	70.3	0.0	0.4	0.0	0.1	0.7	0.1	0.0
3	16-Oct	3	0-3	478.4	275.4	94.5	12.4	28.1	0.0	0.3	0.0	0.0	0.0	0.0	0.0
1	6-Dec	4	0-3	2609.0	1110.0	381.2	39.6	255.9	0.0	0.4	0.0	0.1	0.1	2.7	0.0
2	6-Dec	4	0-3	1342.3	557.0	320.3	34.6	144.5	0.1	0.7	0.0	0.2	0.1	6.4	0.0
3	6-Dec	4	0-3	2767.2	1329.8	446.5	32.6	275.7	0.0	0.5	0.0	0.0	0.0	0.6	0.0
1	6-Dec	5	0-3	1448.1	843.0	326.8	26.1	184.9	0.0	0.5	0.0	0.0	0.0	0.5	0.0
2	6-Dec	5	0-3	2239.2	1101.6	380.7	29.0	263.9	0.0	0.4	0.0	0.0	0.0	0.8	0.0
3	6-Dec	5	0-3	1352.6	547.9	264.9	42.0	154.0	0.0	0.8	0.0	0.2	0.1	6.9	0.0
1	14-Jan	6	0-3	1792.8	927.8	289.6	31.4	230.9	0.0	0.4	0.0	0.0	0.0	0.7	0.0
2	14-Jan	6	0-3	2701.7	795.5	474.5	35.5	267.5	0.1	0.5	0.0	0.0	0.0	4.6	0.0
3	14-Jan	6	0-3	2116.1	837.4	373.5	28.0	238.8	0.0	0.5	0.0	0.0	0.0	2.5	0.0
1	14-Jan	7	0-3	846.7	678.8	169.3	18.0	142.1	0.0	0.4	0.0	0.0	0.0	0.2	0.0
2	14-Jan	7	0-3	573.9	468.7	167.1	17.5	103.3	0.0	0.3	0.0	0.0	0.0	0.2	0.0
3	14-Jan	7	0-3	1147.3	466.5	218.8	39.1	121.3	0.1	0.7	0.0	0.2	0.1	5.5	0.0
1	15-Feb	8	0-3	2036.5	1148.1	373.7	31.4	247.4	0.0	0.4	0.0	0.0	0.0	0.3	0.0
2	15-Feb	8	0-3	2087.2	568.4	345.8	39.8	190.3	0.1	0.3	0.0	0.0	0.0	3.6	0.0
3	15-Feb	8	0-3	3022.3	1252.3	401.1	29.7	269.0	0.0	0.4	0.0	0.0	0.0	0.2	0.0
1	15-Feb	9	0-3	1128.0	488.1	264.2	45.3	104.8	0.1	0.3	0.0	0.2	0.1	5.1	0.0
2	15-Feb	9	0-3	1267.5	664.2	247.6	22.9	157.1	0.0	0.3	0.0	0.0	0.1	0.1	0.0

3	15-Feb	9	0-3	1993.5	628.8	297.5	35.8	196.3	0.0	0.4	0.0	0.0	0.0	2.4	0.0

Table B2: Continued

				Day 0 (Metal Concentrations; mg/L)											
Rep	App. Date	Trt	Depth (in)	Na	Ca	Mg	K	S-SO_4	B	P	Fe	Zn	Cu	Mn	Al
1	20-Mar	10	0-3	2035.5	1172.3	310.2	38.2	257.0	0.0	0.5	0.0	0.0	0.0	0.2	0.0
2	20-Mar	10	0-3	1319.4	726.5	225.1	19.9	143.9	0.0	0.3	0.0	0.0	0.0	0.2	0.0
3	20-Mar	10	0-3	1733.1	1004.3	295.1	27.1	181.5	0.0	0.5	0.0	0.0	0.0	0.3	0.0
1	20-Mar	11	0-3	635.5	529.7	179.0	18.9	105.7	0.0	0.4	0.0	0.0	0.1	0.2	0.0
2	20-Mar	11	0-3	1066.6	451.8	250.0	23.1	122.3	0.1	0.4	0.0	0.0	0.0	0.8	0.0
3	20-Mar	11	0-3	549.9	380.1	153.0	15.6	84.9	0.0	0.4	0.0	0.0	0.0	0.1	0.0
1	Cont	1	3-6	21.1	23.0	12.9	2.3	17.3	0.0	0.3	0.0	0.0	0.0	0.0	0.0
2	Cont	1	3-6	36.2	29.3	13.7	2.8	17.7	0.0	0.3	0.0	0.0	0.0	0.0	0.0
3	Cont	1	3-6	23.3	50.3	24.5	3.6	30.1	0.0	0.5	0.0	0.0	0.0	0.0	0.0
1	16-Oct	2	3-6	136.2	57.7	39.6	4.6	30.2	0.1	0.4	0.0	0.0	0.8	0.0	0.0
2	16-Oct	2	3-6	81.6	73.1	34.8	5.4	21.7	0.0	0.3	0.0	0.0	0.0	0.0	0.0
3	16-Oct	2	3-6	68.0	92.7	44.2	4.8	35.5	0.0	0.5	0.0	0.0	0.0	0.0	0.0
1	16-Oct	3	3-6	64.7	41.0	26.7	4.2	23.6	0.0	0.4	0.0	0.0	0.0	0.0	0.0
2	16-Oct	3	3-6	53.1	49.7	23.8	3.6	21.8	0.0	0.4	0.0	0.0	0.1	0.0	0.0
3	16-Oct	3	3-6	38.5	61.8	25.8	4.4	17.9	0.0	0.4	0.0	0.0	0.0	0.0	0.0
1	6-Dec	4	3-6	248.7	222.9	83.4	10.8	51.4	0.0	0.3	0.0	0.0	0.0	0.1	0.0
2	6-Dec	4	3-6	351.1	136.9	92.6	9.7	54.4	0.1	0.5	0.0	0.0	0.0	0.6	0.0
3	6-Dec	4	3-6	52.6	92.2	36.4	5.8	28.8	0.0	0.2	0.0	0.0	0.0	0.0	0.0
1	6-Dec	5	3-6	128.3	138.0	62.6	7.5	42.3	0.0	0.4	0.0	0.0	0.0	0.1	0.0
2	6-Dec	5	3-6	39.5	79.6	34.3	5.2	31.2	0.0	0.4	0.0	0.0	0.0	0.0	0.0
3	6-Dec	5	3-6	27.8	60.8	32.1	8.1	26.3	0.0	0.7	0.0	0.0	0.0	0.3	0.0
1	14-Jan	6	3-6	52.9	86.9	31.8	5.2	35.4	0.0	0.4	0.0	0.0	0.0	0.0	0.0
2	14-Jan	6	3-6	185.5	56.7	36.2	4.8	37.4	0.1	0.5	0.0	0.0	0.0	0.1	0.0
3	14-Jan	6	3-6	137.0	140.5	63.5	5.8	40.2	0.0	0.5	0.0	0.0	0.0	0.1	0.0
1	14-Jan	7	3-6	29.8	75.6	26.2	4.5	34.4	0.0	0.4	0.0	0.0	0.0	0.0	0.0
2	14-Jan	7	3-6	57.2	76.7	34.3	5.2	26.6	0.0	0.3	0.0	0.0	0.0	0.0	0.0

3	14-Jan	7	3-6	134.0	102.1	46.9	8.3	29.4	0.1	0.5	0.0	0.0	0.0	0.4	0.0

Table B2: Continued

				Day 0 (Metal Concentrations; mg/L)											
Rep	App. Date	Trt	Depth (in)	Na	Ca	Mg	K	S-SO_4	B	P	Fe	Zn	Cu	Mn	Al
1	15-Feb	8	3-6	104.2	67.7	36.4	4.9	36.5	0.1	0.3	0.0	0.0	0.0	0.0	0.0
2	15-Feb	8	3-6	335.1	129.5	52.7	5.8	534.0	0.2	0.4	0.1	0.0	0.0	0.1	0.1
3	15-Feb	8	3-6	81.0	115.1	44.9	5.3	35.6	0.0	0.3	0.0	0.0	0.0	0.0	0.0
1	15-Feb	9	3-6	130.6	78.0	48.2	6.7	37.1	0.1	0.3	0.0	0.0	0.0	0.1	0.0
2	15-Feb	9	3-6	63.1	80.1	39.1	4.9	28.9	0.0	0.2	0.0	0.0	0.0	0.0	0.0
3	15-Feb	9	3-6	92.3	97.5	48.8	6.6	31.6	0.1	0.4	0.0	0.0	0.0	0.1	0.0
1	20-Mar	10	3-6	94.4	112.6	35.3	6.7	44.2	0.0	0.6	0.0	0.0	0.1	0.1	0.0
2	20-Mar	10	3-6	37.7	64.2	24.3	5.3	20.9	0.0	0.4	0.0	0.0	0.0	0.0	0.0
3	20-Mar	10	3-6	47.9	72.9	25.2	5.3	23.2	0.0	0.4	0.0	0.0	0.0	0.0	0.0
1	20-Mar	11	3-6	67.7	66.4	30.9	4.4	46.7	0.0	0.4	0.0	0.0	0.0	0.0	0.0
2	20-Mar	11	3-6	121.6	24.7	15.7	3.5	29.4	0.1	0.5	0.0	0.0	0.0	0.0	0.0
3	20-Mar	11	3-6	42.0	62.3	26.1	5.5	22.2	0.0	0.5	0.0	0.0	0.0	0.0	0.0

Table B2: Continued

				Day 30 (metal concentrations; mg/L)											
Rep	App. Date	Trt	Depth (in)	Na	Ca	Mg	K	S-SO_4	B	P	Fe	Zn	Cu	Mn	Al
1	Cont	1	0-3	.	.	.	.	.	.	.	.	.	.	.	.
2	Cont	1	0-3	.	.	.	.	.	.	.	.	.	.	.	.
3	Cont	1	0-3	.	.	.	.	.	.	.	.	.	.	.	.
1	16-Oct	2	0-3	.	.	.	.	.	.	.	.	.	.	.	.
2	16-Oct	2	0-3	.	.	.	.	.	.	.	.	.	.	.	.
3	16-Oct	2	0-3	.	.	.	.	.	.	.	.	.	.	.	.
1	16-Oct	3	0-3	.	.	.	.	.	.	.	.	.	.	.	.
2	16-Oct	3	0-3	.	.	.	.	.	.	.	.	.	.	.	.
3	16-Oct	3	0-3	.	.	.	.	.	.	.	.	.	.	.	.
1	6-Dec	4	0-3	2032.2	1110.2	387.6	32.7	216.8	0.0	0.5	0.0	0.1	0.1	1.3	0.0
2	6-Dec	4	0-3	2179.3	787.2	451.2	45.7	172.3	0.1	0.6	0.0	0.1	0.0	11.0	0.0
3	6-Dec	4	0-3	2149.1	1268.9	423.8	26.9	183.6	0.0	0.4	0.0	0.0	0.0	0.3	0.0
1	6-Dec	5	0-3	1544.6	852.7	323.2	25.7	179.0	0.0	0.4	0.0	0.0	0.0	0.6	0.0
2	6-Dec	5	0-3	1632.5	1022.7	351.2	28.3	163.5	0.0	0.3	0.0	0.0	0.0	0.6	0.0
3	6-Dec	5	0-3	1557.8	582.5	286.2	44.7	125.7	0.0	0.6	0.0	0.0	0.0	6.9	0.0
1	14-Jan	6	0-3	2140.5	1192.9	350.5	37.4	252.4	0.0	0.4	0.0	0.0	0.0	0.6	0.0
2	14-Jan	6	0-3	3926.4	950.9	539.7	50.4	331.3	0.1	0.4	0.0	0.2	0.1	6.2	0.0
3	14-Jan	6	0-3	2553.5	813.3	332.9	29.7	231.5	0.0	0.5	0.0	0.1	0.1	1.3	0.0
1	14-Jan	7	0-3	832.6	539.9	142.3	16.8	108.1	0.0	0.2	0.0	0.0	0.0	0.1	0.0
2	14-Jan	7	0-3	566.9	450.7	161.4	17.3	81.4	0.0	0.3	0.0	0.0	0.0	0.1	0.0
3	14-Jan	7	0-3	1625.2	528.2	234.6	40.9	140.4	0.0	0.6	0.0	0.1	0.0	7.4	0.0
1	15-Feb	8	0-3	958.4	269.3	113.7	17.7	174.2	0.0	0.5	0.0	0.0	0.0	0.1	0.0
2	15-Feb	8	0-3	663.2	64.7	38.2	13.9	83.6	0.1	0.4	0.0	0.1	0.1	0.5	0.0
3	15-Feb	8	0-3	1356.1	201.9	65.7	12.5	161.9	0.0	0.8	0.0	0.0	0.0	0.0	0.0
1	15-Feb	9	0-3	766.1	142.4	79.5	19.4	100.0	0.1	0.6	0.0	0.0	0.1	1.4	0.0
2	15-Feb	9	0-3	784.1	151.7	67.9	12.4	119.4	0.0	0.4	0.0	0.0	0.0	0.1	0.0
3	15-Feb	9	0-3	1109.3	206.6	99.9	19.6	139.9	0.0	0.6	0.0	0.0	0.0	1.5	0.0

Table B2: Continued

				Day 30 (metal concentrations; mg/L)											
Rep	App. Date	Trt	Depth (in)	Na	Ca	Mg	K	S-SO_4	B	P	Fe	Zn	Cu	Mn	Al
1	20-Mar	10	0-3	566.1	184.9	52.5	9.5	95.1	0.0	0.5	0.0	0.0	0.0	0.1	0.0
2	20-Mar	10	0-3	467.8	154.4	54.4	8.4	67.1	0.0	0.3	0.0	0.0	0.0	0.0	0.0
3	20-Mar	10	0-3	1306.4	407.0	133.2	14.5	152.6	0.0	0.3	0.0	0.0	0.0	0.1	0.0
1	20-Mar	11	0-3	399.5	252.0	84.8	11.6	51.8	0.0	0.4	0.0	0.0	0.0	0.0	0.0
2	20-Mar	11	0-3	580.6	177.0	127.6	9.0	58.3	0.1	0.6	0.0	0.0	0.0	0.1	0.0
3	20-Mar	11	0-3	871.8	260.3	98.6	13.3	115.3	0.0	0.4	0.0	0.0	0.0	0.1	0.0
1	Cont	1	3-6	.	.	.	.	.	.	.	.	.	.	.	.
2	Cont	1	3-6	.	.	.	.	.	.	.	.	.	.	.	.
3	Cont	1	3-6	.	.	.	.	.	.	.	.	.	.	.	.
1	16-Oct	2	3-6	.	.	.	.	.	.	.	.	.	.	.	.
2	16-Oct	2	3-6	.	.	.	.	.	.	.	.	.	.	.	.
3	16-Oct	2	3-6	.	.	.	.	.	.	.	.	.	.	.	.
1	16-Oct	3	3-6	.	.	.	.	.	.	.	.	.	.	.	.
2	16-Oct	3	3-6	.	.	.	.	.	.	.	.	.	.	.	.
3	16-Oct	3	3-6	.	.	.	.	.	.	.	.	.	.	.	.
1	6-Dec	4	3-6	86.6	90.0	39.3	5.5	38.2	0.0	0.5	0.0	0.0	0.0	0.0	0.0
2	6-Dec	4	3-6	70.7	42.1	25.8	8.8	24.4	0.1	0.6	0.0	0.1	0.0	0.2	0.0
3	6-Dec	4	3-6	46.6	96.9	39.0	5.9	29.7	0.0	0.4	0.0	0.0	0.0	0.0	0.0
1	6-Dec	5	3-6	44.8	60.3	26.6	4.6	26.2	0.0	0.4	0.0	0.0	0.0	0.0	0.0
2	6-Dec	5	3-6	75.8	156.8	67.0	7.1	31.4	0.0	0.3	0.0	0.0	0.0	0.1	0.0
3	6-Dec	5	3-6	31.3	52.0	27.0	7.1	21.3	0.1	0.5	0.0	0.0	0.0	0.2	0.0
1	14-Jan	6	3-6	64.1	112.5	39.2	6.4	39.5	0.0	0.3	0.0	0.0	0.0	0.0	0.0
2	14-Jan	6	3-6	227.9	86.5	59.7	7.8	27.3	0.1	0.2	0.0	0.0	0.0	0.0	0.0
3	14-Jan	6	3-6	91.5	129.8	60.3	5.2	31.5	0.0	0.4	0.0	0.0	0.0	0.1	0.0
1	14-Jan	7	3-6	52.8	77.7	29.8	4.1	32.4	0.0	0.3	0.0	0.0	0.0	0.0	0.0
2	14-Jan	7	3-6	50.2	72.7	33.2	4.8	42.7	0.1	0.5	0.0	0.0	0.1	0.0	0.0
3	14-Jan	7	3-6	106.1	112.6	56.1	12.0	31.1	0.1	0.4	0.0	0.0	0.0	0.5	0.0

Table B2: Continued

				Day 30 (metal concentrations; mg/L)											
Rep	App. Date	Trt	Depth (in)	Na	Ca	Mg	K	S-SO_4	B	P	Fe	Zn	Cu	Mn	Al
1	15-Feb	8	3-6	515.8	412.1	212.6	11.2	68.5	0.0	0.3	0.0	0.1	0.1	0.1	0.0
2	15-Feb	8	3-6	236.8	76.2	54.6	7.9	31.1	0.1	0.3	0.0	0.0	0.0	0.0	0.0
3	15-Feb	8	3-6	499.5	805.8	278.3	13.0	63.8	0.0	0.4	0.0	0.0	0.0	0.0	0.0
1	15-Feb	9	3-6	261.3	195.1	123.8	10.1	52.0	0.1	0.5	0.0	0.1	0.0	0.1	0.0
2	15-Feb	9	3-6	230.4	398.9	181.5	12.9	47.7	0.0	0.2	0.0	0.0	0.0	0.0	0.0
3	15-Feb	9	3-6	204.7	329.6	162.4	12.6	30.4	0.0	0.3	0.0	0.0	0.0	0.4	0.0
1	20-Mar	10	3-6	139.9	295.8	104.1	7.9	42.3	0.0	0.3	0.0	0.0	0.0	0.0	0.0
2	20-Mar	10	3-6	413.4	156.4	54.6	7.6	59.0	0.0	0.5	0.0	0.0	0.0	0.0	0.0
3	20-Mar	10	3-6	226.2	766.0	243.6	13.0	50.6	0.0	0.4	0.0	0.0	0.0	0.1	0.0
1	20-Mar	11	3-6	94.6	196.5	91.8	7.4	31.3	0.0	0.4	0.0	0.0	0.1	0.0	0.0
2	20-Mar	11	3-6	510.9	174.3	94.7	14.5	90.8	0.1	0.5	0.0	0.0	0.0	0.2	0.0
3	20-Mar	11	3-6	89.0	298.8	126.7	10.9	34.2	0.0	0.3	0.0	0.0	0.0	0.0	0.0

Table B2: Continued

					Day 90 (Metal concentrations; mg/L)											
Rep	App. Date	Trt	Rate	Depth (in)	Na	Ca	Mg	K	S-SO_4	B	P	Fe	Zn	Cu	Mn	Al
1	Cont	1	0X	0-3	34.5	98.7	38.2	8.4	46.2	0.0	0.4	0.0	0.0	0.1	0.1	0.0
2	Cont	1	0X	0-3	53.9	101.8	48.9	8.8	51.1	0.0	0.4	0.0	0.0	0.0	0.2	0.0
3	Cont	1	0X	0-3	29.4	99.5	41.3	8.2	48.4	0.0	0.6	0.0	0.0	0.0	0.1	0.0
1	16-Oct	2	1X	0-3	2044.9	864.6	418.9	39.2	213.7	0.1	0.4	0.0	0.2	0.1	5.7	0.0
2	16-Oct	2	1X	0-3	1462.0	671.7	337.3	21.5	184.5	0.0	0.4	0.0	0.1	0.0	0.6	0.0
3	16-Oct	2	1X	0-3	3282.7	1171.0	469.2	32.4	362.9	0.0	0.6	0.0	0.1	0.1	3.3	0.0
1	16-Oct	3	0.66X	0-3	1861.9	858.3	319.8	23.9	195.7	0.0	0.4	0.0	0.0	0.0	0.7	0.0
2	16-Oct	3	0.66X	0-3	923.8	490.3	235.9	19.2	109.2	0.0	0.4	0.0	0.1	0.6	0.4	0.0
3	16-Oct	3	0.66X	0-3	792.4	574.2	210.2	18.9	78.6	0.0	0.3	0.0	0.0	0.0	0.3	0.0
1	6-Dec	4	1X	0-3	1138.2	247.6	82.8	17.9	204.8	0.0	0.6	0.0	0.0	0.0	0.4	0.0
2	6-Dec	4	1X	0-3	1368.7	199.6	111.9	25.7	164.7	0.1	0.7	0.0	0.2	0.1	3.8	0.0
3	6-Dec	4	1X	0-3	1037.9	161.2	53.3	13.2	143.2	0.0	0.7	0.0	0.0	0.0	0.1	0.0
1	6-Dec	5	0.66X	0-3	505.8	95.6	36.6	9.5	97.8	0.0	0.5	0.0	0.0	0.0	0.0	0.0
2	6-Dec	5	0.66X	0-3	970.2	117.3	39.1	11.7	182.7	0.0	0.4	0.0	0.0	0.1	0.2	0.0
3	6-Dec	5	0.66X	0-3	888.4	108.3	50.7	18.7	147.7	0.0	0.7	0.0	0.0	0.0	1.4	0.0
1	14-Jan	6	1X	0-3	602.0	80.7	24.8	9.1	113.5	0.0	0.9	0.0	0.0	0.0	0.1	0.0
2	14-Jan	6	1X	0-3	983.9	129.5	84.9	14.4	130.0	0.1	0.4	0.0	0.0	0.0	0.6	0.0
3	14-Jan	6	1X	0-3	2121.8	421.9	169.3	23.7	253.2	0.0	0.6	0.0	0.0	0.0	1.2	0.0
1	14-Jan	7	0.66X	0-3	318.0	89.7	23.2	6.7	74.2	0.0	0.5	0.0	0.0	0.1	0.0	0.0
2	14-Jan	7	0.66X	0-3	218.2	145.6	94.3	6.3	26.8	0.0	0.5	0.0	0.0	0.0	0.0	0.0
3	14-Jan	7	0.66X	0-3	1170.5	156.6	69.9	21.3	131.7	0.0	1.0	0.0	0.0	0.0	3.0	0.0
1	15-Feb	8	1X	0-3	1236.3	283.0	108.1	12.9	154.5	0.0	0.4	0.0	0.0	0.0	0.0	0.0
2	15-Feb	8	1X	0-3	586.9	66.2	42.8	12.5	87.1	0.1	0.2	0.0	0.0	0.0	0.3	0.0
3	15-Feb	8	1X	0-3	1972.4	273.4	84.1	13.5	232.2	0.1	0.4	0.0	0.0	0.0	0.0	0.0
1	15-Feb	9	0.66X	0-3	232.9	158.7	103.5	28.8	37.4	0.1	1.8	0.0	0.0	0.0	0.2	0.0
2	15-Feb	9	0.66X	0-3	1290.9	418.2	155.0	15.9	150.1	0.0	0.3	0.0	0.0	0.0	0.1	0.0
3	15-Feb	9	0.66X	0-3	877.4	101.1	46.7	11.2	162.9	0.0	0.3	0.0	0.0	0.0	0.6	0.0

Table B2: Continued

					Day 90 (Metal concentrations; mg/L)											
Rep	App. Date	Trt	Rate	Depth (in)	Na	Ca	Mg	K	$S\text{-}SO_4$	B	P	Fe	Zn	Cu	Mn	Al
1	20-Mar	10	1X	0-3	792.9	196.2	57.0	15.3	126.9	0.1	0.3	0.0	0.0	0.0	0.1	0.0
2	20-Mar	10	1X	0-3	631.6	148.8	45.9	7.6	99.7	0.0	0.3	0.0	0.0	0.0	0.0	0.0
3	20-Mar	10	1X	0-3	2328.6	570.8	161.9	18.4	253.5	0.0	0.4	0.0	0.0	0.0	0.2	0.0
1	20-Mar	11	0.66X	0-3	254.1	63.0	21.2	5.8	57.1	0.0	0.4	0.0	0.0	0.0	0.0	0.0
2	20-Mar	11	0.66X	0-3	458.2	75.8	41.7	7.8	82.3	0.1	0.2	0.0	0.0	0.0	0.3	0.0
3	20-Mar	11	0.66X	0-3	1529.6	487.7	174.3	15.4	195.3	0.0	0.4	0.0	0.0	0.0	0.3	0.0
1	Cont	1	0X	3-6	37.0	54.4	23.2	5.0	24.5	0.0	0.3	0.0	0.0	0.0	0.0	0.0
2	Cont	1	0X	3-6	44.9	36.4	20.2	3.6	25.5	0.1	0.4	0.0	0.0	0.0	0.0	0.0
3	Cont	1	0X	3-6	14.0	52.5	23.4	5.1	25.8	0.0	0.5	0.0	0.0	0.0	0.0	0.0
1	16-Oct	2	1X	3-6	190.7	242.6	110.9	11.4	38.3	0.1	0.3	0.0	0.0	0.0	0.2	0.0
2	16-Oct	2	1X	3-6	136.1	102.5	64.7	6.7	26.0	0.1	0.4	0.0	0.0	0.2	0.1	0.0
3	16-Oct	2	1X	3-6	188.7	323.3	142.6	11.7	28.0	0.0	0.3	0.0	0.0	0.0	0.3	0.0
1	16-Oct	3	0.66X	3-6	166.3	219.7	97.3	8.1	26.3	0.0	0.3	0.0	0.0	0.0	0.0	0.0
2	16-Oct	3	0.66X	3-6	68.6	53.3	32.5	5.2	21.7	0.1	0.5	0.0	0.2	0.3	0.0	0.0
3	16-Oct	3	0.66X	3-6	35.3	100.4	43.4	5.5	27.0	0.0	0.4	0.0	0.0	0.0	0.0	0.0
1	6-Dec	4	1X	3-6	380.0	285.7	108.9	9.2	65.9	0.0	0.4	0.0	0.2	0.1	0.2	0.0
2	6-Dec	4	1X	3-6	457.1	239.4	158.7	12.2	39.4	0.1	0.4	0.0	0.0	0.0	0.4	0.0
3	6-Dec	4	1X	3-6	529.5	630.0	217.9	13.0	66.9	0.0	0.4	0.0	0.0	0.0	0.0	0.0
1	6-Dec	5	0.66X	3-6	281.0	233.5	102.8	8.0	58.3	0.0	0.3	0.0	0.0	0.0	0.0	0.0
2	6-Dec	5	0.66X	3-6	375.7	360.5	148.6	9.9	61.2	0.0	0.2	0.0	0.0	0.0	0.0	0.0
3	6-Dec	5	0.66X	3-6	339.2	279.0	148.8	14.2	38.2	0.0	0.4	0.0	0.1	0.0	0.8	0.0
1	14-Jan	6	1X	3-6	347.4	413.5	133.1	9.8	76.0	0.0	0.4	0.0	0.0	0.1	0.0	0.0
2	14-Jan	6	1X	3-6	792.3	105.6	68.6	10.6	104.4	0.1	0.6	0.0	0.0	0.0	0.4	0.0
3	14-Jan	6	1X	3-6	745.7	830.8	384.4	13.3	74.4	0.0	0.3	0.0	0.0	0.0	0.2	0.0
1	14-Jan	7	0.66X	3-6	122.1	279.5	98.0	7.7	54.1	0.0	0.3	0.0	0.0	0.0	0.0	0.0
2	14-Jan	7	0.66X	3-6	313.5	77.6	28.0	6.0	80.3	0.0	0.6	0.0	0.0	0.0	0.0	0.0
3	14-Jan	7	0.66X	3-6	506.1	528.7	248.0	16.2	41.3	0.0	0.5	0.0	0.1	0.0	2.6	0.0

Table B2: Continued

					Day 90 (Metal concentrations; mg/L)											
Rep	App. Date	Trt	Rate	Depth (in)	Na	Ca	Mg	K	S-SO_4	B	P	Fe	Zn	Cu	Mn	Al
1	15-Feb	8	1X	3-6	537.0	293.8	149.1	8.3	59.1	0.1	0.2	0.0	0.0	0.0	0.0	0.0
2	15-Feb	8	1X	3-6	401.0	122.5	90.0	7.7	44.0	0.1	0.2	0.0	0.0	0.0	0.1	0.0
3	15-Feb	8	1X	3-6	876.1	633.5	213.7	9.0	103.2	0.0	0.2	0.0	0.0	0.0	0.0	0.0
1	15-Feb	9	0.66X	3-6	476.7	74.4	41.2	10.5	85.4	0.1	0.4	0.0	0.0	0.0	0.7	0.0
2	15-Feb	9	0.66X	3-6	445.8	817.9	332.2	16.7	77.2	0.1	0.2	0.0	0.1	0.0	0.1	0.0
3	15-Feb	9	0.66X	3-6	484.4	384.6	190.0	11.4	58.5	0.0	0.2	0.0	0.0	0.0	0.8	0.0
1	20-Mar	10	1X	3-6	251.2	401.9	135.6	7.4	51.4	0.0	0.2	0.0	0.0	0.0	0.0	0.0
2	20-Mar	10	1X	3-6	263.6	351.1	125.9	6.8	47.9	0.0	0.2	0.0	0.0	0.0	0.0	0.0
3	20-Mar	10	1X	3-6	576.3	913.5	275.6	10.0	66.1	0.0	0.3	0.0	0.0	0.0	0.0	0.0
1	20-Mar	11	0.66X	3-6	115.4	167.4	78.2	6.0	41.9	0.1	0.3	0.0	0.2	0.0	0.0	0.0
2	20-Mar	11	0.66X	3-6	370.3	169.5	118.4	5.8	41.6	0.1	0.2	0.0	0.0	0.0	0.0	0.0
3	20-Mar	11	0.66X	3-6	249.1	444.6	177.1	8.9	44.3	0.0	0.2	0.0	0.0	0.0	0.0	0.0

Table B2: Continued

					Post-havest (Metal concentrations; mg/L)											
Rep	App. Date	Trt	Rate	Depth (in)	Na	Ca	Mg	K	S-SO_4	B	P	Fe	Zn	Cu	Mn	Al
1	Cont	1	0X	0-3	42.5	57.7	21.6	6.3	32.1	0.0	0.6	0.0	0.0	0.0	0.0	0.0
2	Cont	1	0X	0-3	33.9	68.0	31.9	6.4	35.2	0.1	0.5	0.0	0.0	0.0	0.1	0.0
3	Cont	1	0X	0-3	24.3	74.9	29.4	7.3	28.0	0.1	0.4	0.0	0.0	0.0	0.1	0.0
1	16-Oct	2	1X	0-3	499.0	78.8	38.6	11.0	107.8	0.1	0.7	0.0	0.2	0.1	0.6	0.0
2	16-Oct	2	1X	0-3	775.5	154.7	78.0	10.0	145.6	0.1	0.7	0.0	0.0	0.1	0.2	0.0
3	16-Oct	2	1X	0-3	849.6	196.7	82.5	10.7	127.2	0.0	0.5	0.0	0.0	0.0	0.9	0.0
1	16-Oct	3	0.66X	0-3	489.2	93.0	33.3	7.6	98.3	0.0	0.8	0.0	0.0	0.1	0.1	0.0
2	16-Oct	3	0.66X	0-3	373.6	72.1	33.8	7.5	96.3	0.1	0.9	0.0	0.0	0.3	0.1	0.0
3	16-Oct	3	0.66X	0-3	367.1	89.2	29.7	7.0	79.5	0.0	0.8	0.0	0.0	0.0	0.1	0.0
1	6-Dec	4	1X	0-3	690.4	127.3	44.0	11.3	136.7	0.1	0.6	0.0	0.1	0.1	0.4	0.0
2	6-Dec	4	1X	0-3	1075.8	158.5	86.5	17.0	146.4	0.1	0.6	0.0	0.1	0.0	6.4	0.0
3	6-Dec	4	1X	0-3	1007.1	327.9	105.7	12.4	177.3	0.1	0.6	0.0	0.0	0.0	0.2	0.0
1	6-Dec	5	0.66X	0-3	475.6	85.4	31.0	7.8	93.9	0.0	0.7	0.0	0.0	0.0	0.1	0.0
2	6-Dec	5	0.66X	0-3	618.4	173.0	63.9	9.6	106.4	0.0	0.6	0.0	0.0	0.2	0.2	0.0
3	6-Dec	5	0.66X	0-3	522.2	78.1	38.3	12.9	79.4	0.1	0.7	0.0	0.1	0.0	2.1	0.0
1	14-Jan	6	1X	0-3	1122.6	298.8	88.4	16.4	200.0	0.1	0.6	0.0	0.1	0.4	0.6	0.0
2	14-Jan	6	1X	0-3	1146.1	137.6	81.3	13.8	165.1	0.1	0.5	0.0	0.1	0.1	1.8	0.0
3	14-Jan	6	1X	0-3	1299.8	242.0	105.1	12.3	162.7	0.0	0.6	0.0	0.1	0.0	1.6	0.2
1	14-Jan	7	0.66X	0-3	167.3	49.5	12.7	4.9	57.1	0.1	0.5	0.1	0.0	0.1	0.0	0.1
2	14-Jan	7	0.66X	0-3	250.4	77.7	29.8	7.0	94.1	0.1	0.6	0.0	0.0	0.1	0.1	0.0
3	14-Jan	7	0.66X	0-3	880.1	106.7	48.0	16.9	119.8	0.1	0.9	0.0	0.2	0.1	4.6	0.0
1	15-Feb	8	1X	0-3	762.2	146.9	53.3	9.9	123.2	0.1	0.6	0.0	0.0	0.1	0.1	0.0
2	15-Feb	8	1X	0-3	554.6	72.5	42.4	12.5	104.1	0.1	0.8	0.0	0.1	0.2	1.2	0.0
3	15-Feb	8	1X	0-3	1253.7	298.8	94.6	11.6	148.2	0.0	0.4	0.0	0.0	0.0	0.1	0.0
1	15-Feb	9	0.66X	0-3	556.7	110.1	63.1	15.3	89.1	0.1	0.6	0.0	0.3	0.1	2.0	0.0
2	15-Feb	9	0.66X	0-3	808.6	194.2	71.6	10.4	115.9	0.1	0.5	0.0	0.0	0.0	0.1	0.1
3	15-Feb	9	0.66X	0-3	740.6	123.4	57.8	11.1	103.6	0.1	0.7	0.0	0.1	0.0	1.5	0.2

Table B2: Continued

					Post-havest (Metal concentrations; mg/L)											
Rep	App. Date	Trt	Rate	Depth (in)	Na	Ca	Mg	K	S-SO$_4$	B	P	Fe	Zn	Cu	Mn	Al
1	20-Mar	10	1X	0-3	574.8	174.2	49.2	11.1	127.0	0.1	0.5	0.0	0.0	0.1	0.2	0.0
2	20-Mar	10	1X	0-3	511.9	133.3	41.0	8.3	93.0	0.0	0.6	0.0	0.0	0.0	0.1	0.0
3	20-Mar	10	1X	0-3	622.9	154.0	47.1	8.5	99.2	0.0	0.6	0.0	0.0	0.0	0.1	0.0
1	20-Mar	11	0.66X	0-3	239.8	73.6	24.0	6.5	72.4	0.1	0.7	0.0	0.0	0.2	0.1	0.0
2	20-Mar	11	0.66X	0-3	413.8	91.3	48.7	8.8	92.2	0.1	0.4	0.0	0.0	0.0	0.4	0.0
3	20-Mar	11	0.66X	0-3	610.7	259.2	95.0	11.5	100.1	0.0	0.4	0.0	0.0	0.0	0.3	0.0
1	Cont	1	0X	3-6	54.9	60.7	26.1	5.0	36.8	0.1	0.6	0.0	0.0	0.2	0.0	0.0
2	Cont	1	0X	3-6	60.3	50.6	29.9	4.7	32.9	0.1	0.5	0.0	0.0	0.0	0.1	0.0
3	Cont	1	0X	3-6	26.8	71.3	31.6	5.2	25.1	0.1	0.3	0.0	0.0	0.0	0.0	0.1
1	16-Oct	2	1X	3-6	352.0	148.3	88.1	7.6	68.7	0.1	0.4	0.0	0.0	0.0	0.2	0.0
2	16-Oct	2	1X	3-6	535.6	275.3	181.3	10.5	94.1	0.1	0.5	0.0	0.0	0.2	0.1	0.0
3	16-Oct	2	1X	3-6	447.1	302.6	145.4	9.3	61.3	0.0	0.5	0.0	0.0	0.0	0.8	0.0
1	16-Oct	3	0.66X	3-6	508.3	233.0	105.8	8.0	71.9	0.0	0.5	0.0	0.0	0.2	0.1	0.0
2	16-Oct	3	0.66X	3-6	265.6	141.1	87.6	7.1	75.3	0.1	0.5	0.0	0.0	0.0	0.0	0.0
3	16-Oct	3	0.66X	3-6	229.4	174.5	69.5	7.1	63.0	0.0	0.5	0.0	0.0	0.0	0.1	0.2
1	6-Dec	4	1X	3-6	388.6	239.0	109.6	9.4	79.7	0.1	0.7	0.0	0.0	0.0	0.1	0.0
2	6-Dec	4	1X	3-6	733.9	219.6	133.2	12.8	78.4	0.1	0.7	0.0	0.1	0.0	3.2	0.0
3	6-Dec	4	1X	3-6	464.2	438.5	168.3	10.8	75.2	0.1	0.3	0.0	0.0	0.0	0.1	0.1
1	6-Dec	5	0.66X	3-6	331.4	141.8	62.3	6.9	72.9	0.0	0.4	0.0	0.0	0.0	0.0	0.0
2	6-Dec	5	0.66X	3-6	342.8	275.1	129.0	8.9	70.1	0.1	0.4	0.0	0.0	0.0	0.2	0.0
3	6-Dec	5	0.66X	3-6	315.9	184.5	96.3	11.3	36.3	0.1	0.5	0.0	0.0	0.0	1.5	0.0
1	14-Jan	6	1X	3-6	355.4	281.2	105.4	9.7	83.0	0.1	0.5	0.0	0.1	0.5	0.3	0.0
2	14-Jan	6	1X	3-6	760.7	181.3	126.6	11.0	81.7	0.1	0.6	0.0	0.1	0.2	0.7	0.0
3	14-Jan	6	1X	3-6	680.9	485.5	225.0	9.9	64.4	0.0	0.6	0.0	0.0	0.0	0.8	0.0
1	14-Jan	7	0.66X	3-6	112.9	68.6	26.9	4.8	56.0	0.1	0.4	0.0	0.0	0.0	0.0	0.0
2	14-Jan	7	0.66X	3-6	173.0	153.0	73.8	7.0	64.8	0.1	0.5	0.0	0.0	0.3	0.1	0.0
3	14-Jan	7	0.66X	3-6	443.9	241.7	121.2	12.9	43.0	0.1	0.5	0.0	0.1	0.0	5.3	0.0

Table B2: Continued

					Post-havest (Metal concentrations; mg/L)											
Rep	App. Date	Trt	Rate	Depth (in)	Na	Ca	Mg	K	$S\text{-}SO_4$	B	P	Fe	Zn	Cu	Mn	Al
1	15-Feb	8	1X	3-6	536.5	237.8	118.8	9.8	82.3	0.1	0.5	0.0	0.0	0.2	0.1	0.0
2	15-Feb	8	1X	3-6	376.1	93.6	62.7	9.1	64.4	0.1	0.6	0.0	0.1	0.3	0.6	0.0
3	15-Feb	8	1X	3-6	586.3	498.7	183.1	10.6	67.1	0.1	0.4	0.0	0.0	0.0	0.1	0.0
1	15-Feb	9	0.66X	3-6	261.6	96.9	64.3	7.6	54.1	0.1	0.7	0.0	0.5	0.0	0.3	0.0
2	15-Feb	9	0.66X	3-6	399.9	292.9	137.9	11.5	62.5	0.1	0.5	0.2	0.0	0.0	0.1	0.1
3	15-Feb	9	0.66X	3-6	293.0	227.5	114.6	9.6	45.8	0.1	0.5	0.0	0.1	0.0	1.5	0.0
1	20-Mar	10	1X	3-6	222.6	255.0	98.1	9.2	68.9	0.1	0.4	0.0	0.1	0.3	0.3	0.0
2	20-Mar	10	1X	3-6	291.1	323.7	119.5	9.3	57.7	0.1	0.4	0.0	0.1	0.0	0.1	0.0
3	20-Mar	10	1X	3-6	390.3	484.7	164.3	10.1	64.5	0.1	0.3	0.0	0.0	0.0	0.2	0.0
1	20-Mar	11	0.66X	3-6	143.3	119.9	55.5	6.4	66.1	0.1	0.6	0.0	0.0	0.3	0.0	0.0
2	20-Mar	11	0.66X	3-6	336.3	103.6	71.3	6.2	52.0	0.1	0.5	0.0	0.0	0.0	0.2	0.0
3	20-Mar	11	0.66X	3-6	191.2	290.9	121.9	8.9	59.1	0.1	0.2	0.0	0.0	0.0	0.1	0.0

Table B3: Wheat yield harvested on June 20th, 2013 after WBM application in Lahoma, OK. Corrected for 12.5% moisture.

Rep	App. Date	Trt	Rate	kg/ha (12.5%)
1	Control	1	0X	3062
1	16-Oct	2	1X	3341
1	16-Oct	3	0.66X	2488
1	6-Dec	4	1X	2621
1	6-Dec	5	0.66X	2406
1	14-Jan	6	1X	3467
1	14-Jan	7	0.66X	2721
1	15-Feb	8	1X	3140
1	15-Feb	9	0.66X	3286
1	20-Mar	10	1X	1505
1	20-Mar	11	0.66X	2259
2	Control	1	0X	3262
2	16-Oct	2	1X	1915
2	16-Oct	3	0.66X	2692
2	6-Dec	4	1X	3016
2	6-Dec	5	0.66X	1969
2	14-Jan	6	1X	1750
2	14-Jan	7	0.66X	3002
2	15-Feb	8	1X	2029
2	15-Feb	9	0.66X	3054
2	20-Mar	10	1X	2090
2	20-Mar	11	0.66X	2099
3	Control	1	0X	2564
3	16-Oct	2	1X	2679
3	16-Oct	3	0.66X	2357
3	6-Dec	4	1X	1327
3	6-Dec	5	0.66X	3050
3	14-Jan	6	1X	1763
3	14-Jan	7	0.66X	3016
3	15-Feb	8	1X	2395
3	15-Feb	9	0.66X	3080
3	20-Mar	10	1X	1998
3	20-Mar	11	0.66X	1284

APPENDIX C

Sample data was collected for the BTEX column leaching study where oil-base mud (OBM) was surface applied (non-incorporated) at five TPH rates (0, 22,000, 45,000, 67,000, and 90,000 kg TPH ha^{-1}). All treatments were subjected to four different moisture regimes which all produced one leaching event per month (1.5 pore volumes). Moisture regimes were tested by applying 1, 2, 3, and 4 wetting events per month which consisted of 0.5 pore volumes that did not produce leachate. The OBM was sampled 0, 7, 30, 60, and 91 days after application to analyze TPH and BTEX degradation. Leachate was analyzed for BTEX, EC, pH, and metal concentrations at each leaching event.

Table C1: OBM TPH concentrations in mg TPH kg^{-1} for the BTEX column leaching study.

BTEX column leaching study				
rep	rate	moisture	day	TPH (mg/kg)
1	1	1	0	161558
1	1	2	0	161558
1	1	3	0	161558
1	1	4	0	161558
1	2	1	0	161558
1	2	2	0	161558
1	2	3	0	161558
1	2	4	0	161558
1	3	1	0	161558
1	3	2	0	161558
1	3	3	0	161558
1	3	4	0	161558
1	4	1	0	161558
1	4	2	0	161558
1	4	3	0	161558
1	4	4	0	161558
2	1	1	0	161558
2	1	2	0	161558
2	1	3	0	161558
2	1	4	0	161558
2	2	1	0	161558
2	2	2	0	161558
2	2	3	0	161558
2	2	4	0	161558
2	3	1	0	161558
2	3	2	0	161558
2	3	3	0	161558
2	3	4	0	161558
2	4	1	0	161558
2	4	2	0	161558
2	4	3	0	161558
2	4	4	0	161558
3	1	1	0	161558
3	1	2	0	161558
3	1	3	0	161558
3	1	4	0	161558
3	2	1	0	161558

Table C1: Continued

BTEX column leaching study				
rep	rate	moisture	day	TPH (mg/kg)
3	2	2	0	161558
3	2	3	0	161558
3	2	4	0	161558
3	3	1	0	161558
3	3	2	0	161558
3	3	3	0	161558
3	3	4	0	161558
3	4	1	0	161558
3	4	2	0	161558
3	4	3	0	161558
3	4	4	0	161558
1	1	1	7	121756
1	1	2	7	145835
1	1	3	7	147680
1	1	4	7	149074
1	2	1	7	143807
1	2	2	7	140387
1	2	3	7	136493
1	2	4	7	127933
1	3	1	7	146995
1	3	2	7	155268
1	3	3	7	149690
1	3	4	7	152743
1	4	1	7	167126
1	4	2	7	162961
1	4	3	7	145349
1	4	4	7	155770
2	1	1	7	125185
2	1	2	7	142989
2	1	3	7	138427
2	1	4	7	136117
2	2	1	7	134905
2	2	2	7	135628
2	2	3	7	135704
2	2	4	7	126215
2	3	1	7	143655
2	3	2	7	133871

Table C1: Continued

BTEX column leaching study

rep	rate	moisture	day	TPH (mg/kg)
2	3	3	7	79023
2	3	4	7	133505
2	4	1	7	153109
2	4	2	7	146089
2	4	3	7	149114
2	4	4	7	143479
3	1	1	7	115268
3	1	2	7	112663
3	1	3	7	154554
3	1	4	7	149036
3	2	1	7	144975
3	2	2	7	143449
3	2	3	7	124089
3	2	4	7	138977
3	3	1	7	156775
3	3	2	7	152564
3	3	3	7	148688
3	3	4	7	137021
3	4	1	7	146350
3	4	2	7	104841
3	4	3	7	141419
3	4	4	7	129298
1	1	1	30	81941
1	1	2	30	115713
1	1	3	30	105023
1	1	4	30	114559
1	2	1	30	126228
1	2	2	30	114846
1	2	3	30	121857
1	2	4	30	122781
1	3	1	30	136488
1	3	2	30	134225
1	3	3	30	130386
1	3	4	30	126758
1	4	1	30	146308
1	4	2	30	104726
1	4	3	30	115768

Table C1: Continued

BTEX column leaching study				
rep	rate	moisture	day	TPH (mg/kg)
1	4	4	30	139224
2	1	1	30	88079
2	1	2	30	131586
2	1	3	30	111425
2	1	4	30	123697
2	2	1	30	121296
2	2	2	30	113457
2	2	3	30	110085
2	2	4	30	130820
2	3	1	30	154835
2	3	2	30	129694
2	3	3	30	137575
2	3	4	30	153091
2	4	1	30	144739
2	4	2	30	148300
2	4	3	30	150641
2	4	4	30	152535
3	1	1	30	109548
3	1	2	30	112772
3	1	3	30	125674
3	1	4	30	128141
3	2	1	30	138489
3	2	2	30	127888
3	2	3	30	137872
3	2	4	30	154838
3	3	1	30	160719
3	3	2	30	131032
3	3	3	30	157313
3	3	4	30	132609
3	4	1	30	150042
3	4	2	30	141363
3	4	3	30	140246
3	4	4	30	136065
1	1	1	60	106265
1	1	2	60	85893
1	1	3	60	98512
1	1	4	60	90478

Table C1: Continued

BTEX column leaching study				
rep	rate	moisture	day	TPH (mg/kg)
1	2	1	60	118992
1	2	2	60	109798
1	2	3	60	107797
1	2	4	60	104671
1	3	1	60	110791
1	3	2	60	120704
1	3	3	60	101280
1	3	4	60	110952
1	4	1	60	138825
1	4	2	60	117418
1	4	3	60	128342
1	4	4	60	153978
2	1	1	60	93145
2	1	2	60	79018
2	1	3	60	95375
2	1	4	60	97158
2	2	1	60	121140
2	2	2	60	101096
2	2	3	60	104726
2	2	4	60	104349
2	3	1	60	118000
2	3	2	60	117292
2	3	3	60	113417
2	3	4	60	127144
2	4	1	60	125974
2	4	2	60	98329
2	4	3	60	117720
2	4	4	60	124294
3	1	1	60	75481
3	1	2	60	86895
3	1	3	60	89724
3	1	4	60	66906
3	2	1	60	86981
3	2	2	60	94333
3	2	3	60	97181
3	2	4	60	81921
3	3	1	60	101586

Table C1: Continued

BTEX column leaching study				
rep	rate	moisture	day	TPH (mg/kg)
3	3	2	60	110448
3	3	3	60	101373
3	3	4	60	108698
3	4	1	60	107298
3	4	2	60	96465
3	4	3	60	105366
3	4	4	60	117259
1	1	1	91	113736
1	1	2	91	72305
1	1	3	91	93732
1	1	4	91	90079
1	2	1	91	102288
1	2	2	91	104631
1	2	3	91	118261
1	2	4	91	115533
1	3	1	91	102764
1	3	2	91	107490
1	3	3	91	131937
1	3	4	91	126222
1	4	1	91	132006
1	4	2	91	115189
1	4	3	91	107433
1	4	4	91	139915
2	1	1	91	84570
2	1	2	91	92213
2	1	3	91	84091
2	1	4	91	89510
2	2	1	91	109187
2	2	2	91	68349
2	2	3	91	110773
2	2	4	91	104776
2	3	1	91	116786
2	3	2	91	104970
2	3	3	91	127116
2	3	4	91	135293
2	4	1	91	130937
2	4	2	91	132726

Table C1: Continued

BTEX column leaching study				
rep	rate	moisture	day	TPH (mg/kg)
2	4	3	91	120132
2	4	4	91	108637
3	1	1	91	73144
3	1	2	91	75678
3	1	3	91	97519
3	1	4	91	87342
3	2	1	91	107507
3	2	2	91	97907
3	2	3	91	103890
3	2	4	91	94425
3	3	1	91	112176
3	3	2	91	101419
3	3	3	91	108141
3	3	4	91	116803
3	4	1	91	121317
3	4	2	91	118392
3	4	3	91	117599
3	4	4	91	138201

Table C2: OBM BTEX concentrations in mg kg^{-1} for BTEX column leaching study.

				BTEX mud concentrations (mg/kg)				
rep	rate	moisture	day	Benzene	Ethylbenzene	Toluene	o-xylene	m,p-xylene
1	1	1	0	2.65	35.0	23.3	63.5	94.1
1	1	4	0	2.65	35.0	23.3	63.5	94.1
1	2	1	0	2.65	35.0	23.3	63.5	94.1
1	2	3	0	2.65	35.0	23.3	63.5	94.1
1	3	1	0	2.65	35.0	23.3	63.5	94.1
1	3	2	0	2.65	35.0	23.3	63.5	94.1
1	4	1	0	2.65	35.0	23.3	63.5	94.1
1	4	4	0	2.65	35.0	23.3	63.5	94.1
2	1	1	0	2.65	35.0	23.3	63.5	94.1
2	1	2	0	2.65	35.0	23.3	63.5	94.1
2	2	1	0	2.65	35.0	23.3	63.5	94.1
2	2	2	0	2.65	35.0	23.3	63.5	94.1
2	3	1	0	2.65	35.0	23.3	63.5	94.1
2	3	3	0	2.65	35.0	23.3	63.5	94.1
2	4	1	0	2.65	35.0	23.3	63.5	94.1
2	4	2	0	2.65	35.0	23.3	63.5	94.1
3	1	1	0	2.65	35.0	23.3	63.5	94.1
3	1	3	0	2.65	35.0	23.3	63.5	94.1
3	2	1	0	2.65	35.0	23.3	63.5	94.1
3	2	4	0	2.65	35.0	23.3	63.5	94.1
3	3	1	0	2.65	35.0	23.3	63.5	94.1
3	3	4	0	2.65	35.0	23.3	63.5	94.1
3	4	1	0	2.65	35.0	23.3	63.5	94.1
3	4	3	0	2.65	35.0	23.3	63.5	94.1
1	1	1	7	0	0.4	0.5	0.8	1.3
1	1	4	7	0	1.4	0.8	2.9	4.0
1	2	1	7	0	4.1	1.9	7.9	11.8
1	2	3	7	0	2.9	1.5	5.6	8.2
1	3	1	7	0	5.7	2.8	10.3	15.7
1	3	2	7	0	6.4	3.9	10.9	17.3
1	4	1	7	0.27	8.8	5.6	13.9	22.8
1	4	4	7	0	4.5	2.4	8.2	12.4
2	1	1	7	0	0.6	0.6	1.4	2.0
2	1	2	7	0	0.8	0.6	2.1	2.7
2	2	1	7	0	3.4	1.5	6.9	9.9
2	2	2	7	0	4.7	2.4	8.9	13.4
2	3	1	7	0.24	6.8	4.5	11.2	17.6

Table C2: Continued

BTEX mud concentrations (mg/kg)								
rep	rate	moisture	day	Benzene	Ethylbenzene	Toluene	o-xylene	m,p-xylene
2	3	3	7	0.31	9.0	5.8	14.9	23.7
2	4	1	7	0.24	8.0	5.1	13.4	21.4
2	4	2	7	0.31	9.2	5.8	15.3	24.8
3	1	1	7	0	0.8	0.6	1.6	2.3
3	1	3	7	0	1.5	0.9	3.1	4.5
3	2	1	7	0	2.2	1.1	4.6	6.5
3	2	4	7	0	3.3	1.7	6.1	9.3
3	3	1	7	0	5.4	2.6	9.9	15.0
3	3	4	7	0	4.9	2.6	8.7	13.4
3	4	1	7	0	5.8	3.4	10.1	15.7
3	4	3	7	0	5.2	3.4	8.8	14.0

Table C3: BTEX leachate concentrations in ug L^{-1} for the BTEX column leaching study.

					BTEX leachate (ug/L)				
rep	rate	moisture	day	leaching event	Benzene	Ethylbenzene	Toluene	o-xylene	m,p-xylene
1	1	1	0	1	0	1.44	2.23	2.9	3.93
1	2	1	0	1	1.14	2.82	5.38	5	7.46
1	3	1	0	1	1.76	5.6	11.4	7.64	12.9
1	4	1	0	1	4.08	13.9	27.7	15.7	30.3
1	5	1	0	1	0	0	0	0	0
1	1	2	14	1	0	0	0	0	0
1	2	2	14	1	0	0	0	0	0
1	3	2	14	1	0	1.45	1.23	4.51	0
1	4	2	14	1	0	1.38	1.62	4.09	2.53
1	5	2	14	1	0	0	0	0	0
1	1	3	28	1	0	0	0	0	0
1	2	3	28	1	0	0	0	0	0
1	3	3	28	1	0	0	0	2.47	0
1	4	3	28	1	0	1.72	1.01	4.81	4.09
1	5	3	28	1	0	0	0	0	0
1	1	4	28	1	0	0	0	0	0
1	2	4	28	1	0	0	0	0	0
1	3	4	28	1	0	0	0	0	0
1	4	4	28	1	0	0	0	1.71	0
1	5	4	28	1	0	0	0	0	0
1	1	1	35	2	0	0	0	0	0
1	2	1	35	2	0	0	0	0	0
1	3	1	35	2	0	0	0	0	0
1	4	1	35	2	0	0	0	0	0
1	5	1	35	2	0	0	0	0	0
1	1	2	49	2	0	0	0	0	0
1	2	2	49	2	0	0	0	0	0
1	3	2	49	2	0	0	0	0	0
1	4	2	49	2	0	0	0	0	0
1	5	2	49	2	0	0	0	0	0
1	1	3	56	2	0	0	0	0	0
1	2	3	56	2	0	0	0	0	0
1	3	3	56	2	0	0	0	0	0
1	4	3	56	2	0	0	0	1.28	0
1	5	3	56	2	0	0	0	0	0
1	1	4	56	2	0	0	0	0	0
1	2	4	56	2	0	0	0	0	0

Table C3: Continued

					BTEX leachate (ug/L)				
rep	rate	moisture	day	leaching event	Benzene	Ethylbenzene	Toluene	o-xylene	m,p-xylene
1	3	4	56	2	0	0	0	0	0
1	4	4	56	2	0	0	0	0	0
1	5	4	56	2	0	0	0	0	0
1	1	1	63	3	0	0	0	0	0
1	2	1	63	3	0	0	0	0	0
1	3	1	63	3	0	0	0	0	0
1	4	1	63	3	0	0	0	0	0
1	5	1	63	3	0	0	0	0	0
1	1	2	77	3	0	0	0	0	0
1	2	2	77	3	0	0	0	0	0
1	3	2	77	3	0	0	0	0	0
1	4	2	77	3	0	0	0	0	0
1	5	2	77	3	0	0	0	0	0
1	1	3	84	3	0	0	0	0	0
1	2	3	84	3	0	0	0	0	0
1	3	3	84	3	0	0	0	0	0
1	4	3	84	3	0	0	0	0	0
1	5	3	84	3	0	0	0	0	0
1	1	4	84	3	0	0	0	0	0
1	2	4	84	3	0	0	0	0	0
1	3	4	84	3	0	0	0	0	0
1	4	4	84	3	0	0	0	0	0
1	5	4	84	3	0	0	0	0	0
2	1	1	0	1	0	1.45	1.98	2.8	3.95
2	2	1	0	1	2.26	7.5	13.1	10.6	17.8
2	3	1	0	1	0	1.62	3.45	2.67	4.08
2	4	1	0	1	3.43	9.99	19.7	13.4	23
2	5	1	0	1	0	0	0	0	0
2	1	2	14	1	0	0	0	0	0
2	2	2	14	1	0	0	0	1.33	0
2	3	2	14	1	0	1.03	0	4.01	2.93
2	4	2	14	1	0	0	0	1.36	0
2	5	2	14	1	0	0	0	0	0
2	1	3	28	1	0	0	0	0	0
2	2	3	28	1	0	0	0	0	0
2	3	3	28	1	0	0	0	1.2	0
2	4	3	28	1	0	0	0	2.04	0

Table C3: Continued

					BTEX leachate (ug/L)				
rep	rate	moisture	day	leaching event	Benzene	Ethylbenzene	Toluene	o-xylene	m,p-xylene
2	5	3	28	1	0	0	0	0	0
2	1	4	28	1	0	0	0	0	0
2	2	4	28	1	0	0	0	0	0
2	3	4	28	1	0	0	0	1.03	0
2	4	4	28	1	0	2.26	1.27	5.99	6.12
2	5	4	28	1	0	0	0	0	0
2	1	1	35	2	0	0	0	0	0
2	2	1	35	2	0	0	0	0	0
2	3	1	35	2	0	0	0	0	0
2	4	1	35	2	0	0	0	1.19	0
2	5	1	35	2	0	0	0	0	0
2	1	2	49	2	0	0	0	0	0
2	2	2	49	2	0	0	0	0	0
2	3	2	49	2	0	0	0	0	0
2	4	2	49	2	0	0	0	1.34	0
2	5	2	49	2	0	0	0	0	0
2	1	3	56	2	0	0	0	0	0
2	2	3	56	2	0	0	0	0	0
2	3	3	56	2	0	0	0	0	0
2	4	3	56	2	0	0	0	0	0
2	5	3	56	2	0	0	0	0	0
2	1	4	56	2	0	0	0	0	0
2	2	4	56	2	0	0	0	0	0
2	3	4	56	2	0	0	0	0	0
2	4	4	56	2	0	0	0	1.06	0
2	5	4	56	2	0	0	0	0	0
2	1	1	63	3	0	0	0	0	0
2	2	1	63	3	0	0	0	0	0
2	3	1	63	3	0	0	0	0	0
2	4	1	63	3	0	0	0	0	0
2	5	1	63	3	0	0	0	0	0
2	1	2	77	3	0	0	0	0	0
2	2	2	77	3	0	0	0	0	0
2	3	2	77	3	0	0	0	0	0
2	4	2	77	3	0	0	0	0	0
2	5	2	77	3	0	0	0	0	0
2	1	3	84	3	.	.	.	.	.

Table C3: Continued

					BTEX leachate (ug/L)				
rep	rate	moisture	day	leaching event	Benzene	Ethylbenzene	Toluene	o-xylene	m,p-xylene
2	2	3	84	3	0	0	0	0	0
2	3	3	84	3	0	0	0	0	0
2	4	3	84	3	0	0	0	0	0
2	5	3	84	3	0	0	0	0	0
2	1	4	84	3	0	0	0	0	0
2	2	4	84	3	0	0	0	0	0
2	3	4	84	3	0	0	0	0	0
2	4	4	84	3	0	0	0	0	0
2	5	4	84	3	0	0	0	0	0
3	1	1	0	1	1.33	8.53	14.9	11.8	20.3
3	2	1	0	1	4.04	15.7	28.2	21.6	36.9
3	3	1	0	1	7	31.3	59.3	36.7	68.1
3	4	1	0	1	5.09	19.5	33.6	23.6	44
3	5	1	0	1	0	0	0	0	0
3	1	2	14	1	0	0	0	0	0
3	2	2	14	1	0	0	0	0	0
3	3	2	14	1	0	0	0	0	0
3	4	2	14	1	0	0	0	1.4	0
3	5	2	14	1	0	0	0	0	0
3	1	3	28	1	0	0	0	0	0
3	2	3	28	1	0	0	0	0	0
3	3	3	28	1	0	0	0	0	0
3	4	3	28	1	0	0	0	3.05	2.69
3	5	3	28	1	0	0	0	0	0
3	1	4	28	1	0	0	0	0	0
3	2	4	28	1	0	0	0	0	0
3	3	4	28	1	0	0	0	0	0
3	4	4	28	1	0	0	0	2.55	2.1
3	5	4	28	1	0	0	0	0	0
3	1	1	35	2	0	0	0	0	0
3	2	1	35	2	0	0	0	0	0
3	3	1	35	2	0	0	0	0	0
3	4	1	35	2	0	0	0	2.35	0
3	5	1	35	2	0	0	0	0	0
3	1	2	49	2	0	0	0	0	0
3	2	2	49	2	0	0	0	0	0
3	3	2	49	2	0	0	0	0	0

Table C3: Continued

					BTEX leachate (ug/L)				
rep	rate	moisture	day	leaching event	Benzene	Ethylbenzene	Toluene	o-xylene	m,p-xylene
3	4	2	49	2	0	0	0	1.48	0
3	5	2	49	2	0	0	0	0	0
3	1	3	56	2	0	0	0	0	0
3	2	3	56	2	0	0	0	0	0
3	3	3	56	2	0	0	0	0	0
3	4	3	56	2	0	0	0	0	0
3	5	3	56	2	0	0	0	0	0
3	1	4	56	2	0	0	0	0	0
3	2	4	56	2	0	0	0	0	0
3	3	4	56	2	0	0	0	0	0
3	4	4	56	2	0	0	0	0	0
3	5	4	56	2	0	0	0	0	0
3	1	1	63	3	0	0	0	0	0
3	2	1	63	3	0	0	0	0	0
3	3	1	63	3	0	0	0	0	0
3	4	1	63	3	0	0	0	0	0
3	5	1	63	3	0	0	0	0	0
3	1	2	77	3	0	0	0	0	0
3	2	2	77	3	0	0	0	0	0
3	3	2	77	3	0	0	0	0	0
3	4	2	77	3	0	0	0	0	0
3	5	2	77	3	0	0	0	0	0
3	1	3	84	3	0	0	0	0	0
3	2	3	84	3	0	0	0	0	0
3	3	3	84	3	0	0	0	0	0
3	4	3	84	3	0	0	0	0	0
3	5	3	84	3	0	0	0	0	0
3	1	4	84	3	0	0	0	0	0
3	2	4	84	3	.	.	.	.	.
3	3	4	84	3	0	0	0	0	0
3	4	4	84	3	0	0	0	0	0
3	5	4	84	3	0	0	0	0	0

Table C4: BTEX leachate loads (ug) for the BTEX column leaching study.

					BTEX leach loads (ug)				
rep	rate	moisture	day	Leaching Event	Benzene	Ethylbenzene	Toluene	o-xylene	m,p-xylene
1	1	1	0	1	0.00	0.57	0.89	1.16	1.57
1	2	1	0	1	0.54	1.34	2.56	2.38	3.55
1	3	1	0	1	0.83	2.66	5.41	3.62	6.12
1	4	1	0	1	1.88	6.42	12.80	7.25	14.00
1	5	1	0	1	0.00	0.00	0.00	0.00	0.00
1	1	2	14	1	0.00	0.00	0.00	0.00	0.00
1	2	2	14	1	0.00	0.00	0.00	0.00	0.00
1	3	2	14	1	0.00	0.79	0.67	2.47	0.00
1	4	2	14	1	0.00	0.79	0.93	2.34	1.45
1	5	2	14	1	0.00	0.00	0.00	0.00	0.00
1	1	3	28	1	0.00	0.00	0.00	0.00	0.00
1	2	3	28	1	0.00	0.00	0.00	0.00	0.00
1	3	3	28	1	0.00	0.00	0.00	2.06	0.00
1	4	3	28	1	0.00	1.61	0.94	4.50	3.83
1	5	3	28	1	0.00	0.00	0.00	0.00	0.00
1	1	4	28	1	0.00	0.00	0.00	0.00	0.00
1	2	4	28	1	0.00	0.00	0.00	0.00	0.00
1	3	4	28	1	0.00	0.00	0.00	0.00	0.00
1	4	4	28	1	0.00	0.00	0.00	1.78	0.00
1	5	4	28	1	0.00	0.00	0.00	0.00	0.00
1	1	1	35	2	0.00	0.00	0.00	0.00	0.00
1	2	1	35	2	0.00	0.00	0.00	0.00	0.00
1	3	1	35	2	0.00	0.00	0.00	0.00	0.00
1	4	1	35	2	0.00	0.00	0.00	0.00	0.00
1	5	1	35	2	0.00	0.00	0.00	0.00	0.00
1	1	2	49	2	0.00	0.00	0.00	0.00	0.00
1	2	2	49	2	0.00	0.00	0.00	0.00	0.00
1	3	2	49	2	0.00	0.00	0.00	0.00	0.00
1	4	2	49	2	0.00	0.00	0.00	0.00	0.00
1	5	2	49	2	0.00	0.00	0.00	0.00	0.00
1	1	3	56	2	0.00	0.00	0.00	0.00	0.00
1	2	3	56	2	0.00	0.00	0.00	0.00	0.00
1	3	3	56	2	0.00	0.00	0.00	0.00	0.00
1	4	3	56	2	0.00	0.00	0.00	0.80	0.00
1	5	3	56	2	0.00	0.00	0.00	0.00	0.00
1	1	4	56	2	0.00	0.00	0.00	0.00	0.00

Table C4: Continued

					BTEX leach loads (ug)				
rep	rate	moisture	day	Leaching Event	Benzene	Ethylbenzene	Toluene	o-xylene	m,p-xylene
1	2	4	56	2	0.00	0.00	0.00	0.00	0.00
1	3	4	56	2	0.00	0.00	0.00	0.00	0.00
1	4	4	56	2	0.00	0.00	0.00	0.00	0.00
1	5	4	56	2	0.00	0.00	0.00	0.00	0.00
1	1	1	63	3	0.00	0.00	0.00	0.00	0.00
1	2	1	63	3	0.00	0.00	0.00	0.00	0.00
1	3	1	63	3	0.00	0.00	0.00	0.00	0.00
1	4	1	63	3	0.00	0.00	0.00	0.00	0.00
1	5	1	63	3	0.00	0.00	0.00	0.00	0.00
1	1	2	77	3	0.00	0.00	0.00	0.00	0.00
1	2	2	77	3	0.00	0.00	0.00	0.00	0.00
1	3	2	77	3	0.00	0.00	0.00	0.00	0.00
1	4	2	77	3	0.00	0.00	0.00	0.00	0.00
1	5	2	77	3	0.00	0.00	0.00	0.00	0.00
1	1	3	84	3	0.00	0.00	0.00	0.00	0.00
1	2	3	84	3	0.00	0.00	0.00	0.00	0.00
1	3	3	84	3	0.00	0.00	0.00	0.00	0.00
1	4	3	84	3	0.00	0.00	0.00	0.00	0.00
1	5	3	84	3	0.00	0.00	0.00	0.00	0.00
1	1	4	84	3	0.00	0.00	0.00	0.00	0.00
1	2	4	84	3	0.00	0.00	0.00	0.00	0.00
1	3	4	84	3	0.00	0.00	0.00	0.00	0.00
1	4	4	84	3	0.00	0.00	0.00	0.00	0.00
1	5	4	84	3	0.00	0.00	0.00	0.00	0.00
2	1	1	0	1	0.00	0.66	0.91	1.28	1.81
2	2	1	0	1	1.17	3.89	6.80	5.50	9.24
2	3	1	0	1	0.00	0.67	1.42	1.10	1.68
2	4	1	0	1	2.00	5.81	11.46	7.80	13.38
2	5	1	0	1	0.00	0.00	0.00	0.00	0.00
2	1	2	14	1	0.00	0.00	0.00	0.00	0.00
2	2	2	14	1	0.00	0.00	0.00	1.07	0.00
2	3	2	14	1	0.00	0.87	0.00	3.39	2.48
2	4	2	14	1	0.00	0.00	0.00	0.95	0.00
2	5	2	14	1	0.00	0.00	0.00	0.00	0.00
2	1	3	28	1	0.00	0.00	0.00	0.00	0.00
2	2	3	28	1	0.00	0.00	0.00	0.00	0.00
2	3	3	28	1	0.00	0.00	0.00	1.11	0.00

Table C4: Continued

					BTEX leach loads (ug)				
rep	rate	moisture	day	Leaching Event	Benzene	Ethylbenzene	Toluene	o-xylene	m,p-xylene
2	4	3	28	1	0.00	0.00	0.00	1.87	0.00
2	5	3	28	1	0.00	0.00	0.00	0.00	0.00
2	1	4	28	1	0.00	0.00	0.00	0.00	0.00
2	2	4	28	1	0.00	0.00	0.00	0.00	0.00
2	3	4	28	1	0.00	0.00	0.00	1.18	0.00
2	4	4	28	1	0.00	2.24	1.26	5.94	6.06
2	5	4	28	1	0.00	0.00	0.00	0.00	0.00
2	1	1	35	2	0.00	0.00	0.00	0.00	0.00
2	2	1	35	2	0.00	0.00	0.00	0.00	0.00
2	3	1	35	2	0.00	0.00	0.00	0.00	0.00
2	4	1	35	2	0.00	0.00	0.00	0.57	0.00
2	5	1	35	2	0.00	0.00	0.00	0.00	0.00
2	1	2	49	2	0.00	0.00	0.00	0.00	0.00
2	2	2	49	2	0.00	0.00	0.00	0.00	0.00
2	3	2	49	2	0.00	0.00	0.00	0.00	0.00
2	4	2	49	2	0.00	0.00	0.00	0.78	0.00
2	5	2	49	2	0.00	0.00	0.00	0.00	0.00
2	1	3	56	2	0.00	0.00	0.00	0.00	0.00
2	2	3	56	2	0.00	0.00	0.00	0.00	0.00
2	3	3	56	2	0.00	0.00	0.00	0.00	0.00
2	4	3	56	2	0.00	0.00	0.00	0.00	0.00
2	5	3	56	2	0.00	0.00	0.00	0.00	0.00
2	1	4	56	2	0.00	0.00	0.00	0.00	0.00
2	2	4	56	2	0.00	0.00	0.00	0.00	0.00
2	3	4	56	2	0.00	0.00	0.00	0.00	0.00
2	4	4	56	2	0.00	0.00	0.00	0.66	0.00
2	5	4	56	2	0.00	0.00	0.00	0.00	0.00
2	1	1	63	3	0.00	0.00	0.00	0.00	0.00
2	2	1	63	3	0.00	0.00	0.00	0.00	0.00
2	3	1	63	3	0.00	0.00	0.00	0.00	0.00
2	4	1	63	3	0.00	0.00	0.00	0.00	0.00
2	5	1	63	3	0.00	0.00	0.00	0.00	0.00
2	1	2	77	3	0.00	0.00	0.00	0.00	0.00
2	2	2	77	3	0.00	0.00	0.00	0.00	0.00
2	3	2	77	3	0.00	0.00	0.00	0.00	0.00
2	4	2	77	3	0.00	0.00	0.00	0.00	0.00
2	5	2	77	3	0.00	0.00	0.00	0.00	0.00

Table C4: Continued

					BTEX leach loads (ug)				
rep	rate	moisture	day	Leaching Event	Benzene	Ethylbenzene	Toluene	o-xylene	m,p-xylene
2	2	3	84	3	0.00	0.00	0.00	0.00	0.00
2	3	3	84	3	0.00	0.00	0.00	0.00	0.00
2	4	3	84	3	0.00	0.00	0.00	0.00	0.00
2	5	3	84	3	0.00	0.00	0.00	0.00	0.00
2	1	4	84	3	0.00	0.00	0.00	0.00	0.00
2	2	4	84	3	0.00	0.00	0.00	0.00	0.00
2	3	4	84	3	0.00	0.00	0.00	0.00	0.00
2	4	4	84	3	0.00	0.00	0.00	0.00	0.00
2	5	4	84	3	0.00	0.00	0.00	0.00	0.00
3	1	1	0	1	0.62	4.00	6.99	5.53	9.52
3	2	1	0	1	1.67	6.48	11.65	8.92	15.24
3	3	1	0	1	2.81	12.58	23.83	14.75	27.37
3	4	1	0	1	2.41	9.22	15.89	11.16	20.81
3	5	1	0	1	0.00	0.00	0.00	0.00	0.00
3	1	2	14	1	0.00	0.00	0.00	0.00	0.00
3	2	2	14	1	0.00	0.00	0.00	0.00	0.00
3	3	2	14	1	0.00	0.00	0.00	0.00	0.00
3	4	2	14	1	0.00	0.00	0.00	0.97	0.00
3	5	2	14	1	0.00	0.00	0.00	0.00	0.00
3	1	3	28	1	0.00	0.00	0.00	0.00	0.00
3	2	3	28	1	0.00	0.00	0.00	0.00	0.00
3	3	3	28	1	0.00	0.00	0.00	0.00	0.00
3	4	3	28	1	0.00	0.00	0.00	2.28	2.01
3	5	3	28	1	0.00	0.00	0.00	0.00	0.00
3	1	4	28	1	0.00	0.00	0.00	0.00	0.00
3	2	4	28	1	0.00	0.00	0.00	0.00	0.00
3	3	4	28	1	0.00	0.00	0.00	0.00	0.00
3	4	4	28	1	0.00	0.00	0.00	2.94	2.42
3	5	4	28	1	0.00	0.00	0.00	0.00	0.00
3	1	1	35	2	0.00	0.00	0.00	0.00	0.00
3	2	1	35	2	0.00	0.00	0.00	0.00	0.00
3	3	1	35	2	0.00	0.00	0.00	0.00	0.00
3	4	1	35	2	0.00	0.00	0.00	1.13	0.00
3	5	1	35	2	0.00	0.00	0.00	0.00	0.00
3	1	2	49	2	0.00	0.00	0.00	0.00	0.00
3	2	2	49	2	0.00	0.00	0.00	0.00	0.00
3	3	2	49	2	0.00	0.00	0.00	0.00	0.00

Table C4: Continued

					BTEX leach loads (ug)				
rep	rate	moisture	day	Leaching Event	Benzene	Ethylbenzene	Toluene	o-xylene	m,p-xylene
3	4	2	49	2	0.00	0.00	0.00	0.92	0.00
3	5	2	49	2	0.00	0.00	0.00	0.00	0.00
3	1	3	56	2	0.00	0.00	0.00	0.00	0.00
3	2	3	56	2	0.00	0.00	0.00	0.00	0.00
3	3	3	56	2	0.00	0.00	0.00	0.00	0.00
3	4	3	56	2	0.00	0.00	0.00	0.00	0.00
3	5	3	56	2	0.00	0.00	0.00	0.00	0.00
3	1	4	56	2	0.00	0.00	0.00	0.00	0.00
3	2	4	56	2	0.00	0.00	0.00	0.00	0.00
3	3	4	56	2	0.00	0.00	0.00	0.00	0.00
3	4	4	56	2	0.00	0.00	0.00	0.00	0.00
3	5	4	56	2	0.00	0.00	0.00	0.00	0.00
3	1	1	63	3	0.00	0.00	0.00	0.00	0.00
3	2	1	63	3	0.00	0.00	0.00	0.00	0.00
3	3	1	63	3	0.00	0.00	0.00	0.00	0.00
3	4	1	63	3	0.00	0.00	0.00	0.00	0.00
3	5	1	63	3	0.00	0.00	0.00	0.00	0.00
3	1	2	77	3	0.00	0.00	0.00	0.00	0.00
3	2	2	77	3	0.00	0.00	0.00	0.00	0.00
3	3	2	77	3	0.00	0.00	0.00	0.00	0.00
3	4	2	77	3	0.00	0.00	0.00	0.00	0.00
3	5	2	77	3	0.00	0.00	0.00	0.00	0.00
3	1	3	84	3	0.00	0.00	0.00	0.00	0.00
3	2	3	84	3	0.00	0.00	0.00	0.00	0.00
3	3	3	84	3	0.00	0.00	0.00	0.00	0.00
3	4	3	84	3	0.00	0.00	0.00	0.00	0.00
3	5	3	84	3	0.00	0.00	0.00	0.00	0.00
3	1	4	84	3	0.00	0.00	0.00	0.00	0.00
3	2	4	84	3	.	.	.	.	.
3	3	4	84	3	0.00	0.00	0.00	0.00	0.00
3	4	4	84	3	0.00	0.00	0.00	0.00	0.00
3	5	4	84	3	0.00	0.00	0.00	0.00	0.00

Table C5: Leachate EC and pH for the BTEX column study.

rep	rate	moisture	day	leaching event	EC (mS/cm)	pH
1	1	1	0	1	0.70	7.6
1	2	1	0	1	0.85	7.8
1	3	1	0	1	1.18	7.8
1	4	1	0	1	.	.
1	5	1	0	1	0.75	7.9
1	1	2	14	1	0.72	7.0
1	2	2	14	1	0.91	6.4
1	3	2	14	1	0.79	7.6
1	4	2	14	1	.	.
1	5	2	14	1	.	.
1	1	3	28	1	1.21	7.0
1	2	3	28	1	1.32	7.1
1	3	3	28	1	1.28	7.4
1	4	3	28	1	1.07	7.0
1	5	3	28	1	1.23	5.9
1	1	4	28	1	1.31	7.0
1	2	4	28	1	1.29	7.0
1	3	4	28	1	1.23	7.2
1	4	4	28	1	1.16	7.5
1	5	4	28	1	1.23	6.5
1	1	1	35	2	0.98	7.1
1	2	1	35	2	1.08	7.6
1	3	1	35	2	0.85	7.3
1	4	1	35	2	0.85	7.2
1	5	1	35	2	1.13	7.3
1	1	2	49	2	1.26	7.6
1	2	2	49	2	1.10	8.0
1	3	2	49	2	1.24	8.1
1	4	2	49	2	1.14	8.2
1	5	2	49	2	1.13	7.9
1	1	3	56	2	1.18	7.9
1	2	3	56	2	1.71	7.5
1	3	3	56	2	1.27	7.1
1	4	3	56	2	1.23	8.1
1	5	3	56	2	1.06	7.3
1	1	4	56	2	1.07	7.3
1	2	4	56	2	1.28	7.9

Table C5: Continued

rep	rate	moisture	day	leaching event	EC (mS/cm)	pH
1	3	4	56	2	1.34	7.8
1	4	4	56	2	1.34	8.0
1	5	4	56	2	0.73	7.7
1	1	1	63	3	1.23	8.3
1	2	1	63	3	1.87	8.0
1	3	1	63	3	1.16	8.3
1	4	1	63	3	1.45	8.2
1	5	1	63	3	1.01	8.2
1	1	2	77	3	.	.
1	2	2	77	3	1.53	7.6
1	3	2	77	3	1.91	8.3
1	4	2	77	3	2.30	7.3
1	5	2	77	3	.	.
1	1	3	84	3	1.28	7.2
1	2	3	84	3	1.79	7.3
1	3	3	84	3	1.10	7.2
1	4	3	84	3	1.52	7.2
1	5	3	84	3	0.61	8.0
1	1	4	84	3	0.76	8.3
1	2	4	84	3	1.19	8.4
1	3	4	84	3	1.33	7.5
1	4	4	84	3	1.42	7.2
1	5	4	84	3	0.43	8.2
2	1	1	0	1	0.74	7.7
2	2	1	0	1	0.95	7.5
2	3	1	0	1	0.69	7.4
2	4	1	0	1	0.72	7.7
2	5	1	0	1	0.59	7.4
2	1	2	14	1	0.83	6.9
2	2	2	14	1	0.92	6.7
2	3	2	14	1	0.89	7.1
2	4	2	14	1	1.20	6.7
2	5	2	14	1	0.93	7.2
2	1	3	28	1	1.22	7.5
2	2	3	28	1	1.34	6.5
2	3	3	28	1	1.35	6.9
2	4	3	28	1	1.60	7.0
2	5	3	28	1	1.24	6.8

Table C5: Continued

rep	rate	moisture	day	leaching event	EC (mS/cm)	pH
2	1	4	28	1	1.07	6.9
2	2	4	28	1	1.53	7.1
2	3	4	28	1	1.24	6.8
2	4	4	28	1	1.43	6.8
2	5	4	28	1	1.17	6.8
2	1	1	35	2	1.14	4.9
2	2	1	35	2	1.11	7.2
2	3	1	35	2	0.79	7.9
2	4	1	35	2	1.08	7.4
2	5	1	35	2	.	.
2	1	2	49	2	1.14	8.2
2	2	2	49	2	1.62	8.0
2	3	2	49	2	1.07	8.2
2	4	2	49	2	1.67	8.1
2	5	2	49	2	1.27	7.9
2	1	3	56	2	1.73	7.7
2	2	3	56	2	1.38	7.4
2	3	3	56	2	1.47	7.8
2	4	3	56	2	1.78	7.5
2	5	3	56	2	0.80	7.7
2	1	4	56	2	1.08	7.4
2	2	4	56	2	1.46	7.5
2	3	4	56	2	1.37	7.2
2	4	4	56	2	1.57	7.9
2	5	4	56	2	0.86	7.8
2	1	1	63	3	1.63	8.4
2	2	1	63	3	1.49	8.3
2	3	1	63	3	1.05	8.4
2	4	1	63	3	1.80	8.2
2	5	1	63	3	0.82	8.3
2	1	2	77	3	1.45	7.7
2	2	2	77	3	1.71	8.3
2	3	2	77	3	1.43	7.5
2	4	2	77	3	2.20	6.9
2	5	2	77	3	.	.
2	1	3	84	3	1.46	7.5
2	2	3	84	3	1.48	7.6
2	3	3	84	3	1.34	7.6

Table C5: Continued

rep	rate	moisture	day	leaching event	EC (mS/cm)	pH
2	4	3	84	3	1.85	7.9
2	5	3	84	3	0.40	7.6
2	1	4	84	3	1.10	7.2
2	2	4	84	3	1.52	7.4
2	3	4	84	3	1.22	7.4
2	4	4	84	3	1.26	7.4
2	5	4	84	3	0.55	8.4
3	1	1	0	1	0.93	7.8
3	2	1	0	1	1.00	7.6
3	3	1	0	1	1.01	7.6
3	4	1	0	1	0.93	7.6
3	5	1	0	1	0.63	7.5
3	1	2	14	1	0.89	7.7
3	2	2	14	1	0.94	7.6
3	3	2	14	1	1.19	7.1
3	4	2	14	1	1.69	7.0
3	5	2	14	1	1.06	7.1
3	1	3	28	1	1.23	6.9
3	2	3	28	1	1.43	7.0
3	3	3	28	1	1.64	7.3
3	4	3	28	1	1.75	7.2
3	5	3	28	1	1.54	6.7
3	1	4	28	1	1.16	7.5
3	2	4	28	1	1.34	7.4
3	3	4	28	1	1.66	7.2
3	4	4	28	1	1.76	6.6
3	5	4	28	1	1.56	6.7
3	1	1	35	2	1.06	7.6
3	2	1	35	2	1.42	7.8
3	3	1	35	2	1.24	6.8
3	4	1	35	2	1.43	7.9
3	5	1	35	2	1.45	8.6
3	1	2	49	2	1.60	8.1
3	2	2	49	2	1.59	8.2
3	3	2	49	2	1.77	8.2
3	4	2	49	2	1.68	8.1
3	5	2	49	2	1.12	8.0
3	1	3	56	2	1.50	7.7

Table C5: Continued

rep	rate	moisture	day	leaching event	EC (mS/cm)	pH
3	2	3	56	2	1.74	7.7
3	3	3	56	2	2.12	7.5
3	4	3	56	2	1.99	8.0
3	5	3	56	2	1.22	6.1
3	1	4	56	2	1.12	7.6
3	2	4	56	2	1.60	8.3
3	3	4	56	2	2.09	8.1
3	4	4	56	2	1.21	7.8
3	5	4	56	2	1.17	7.8
3	1	1	63	3	1.31	8.4
3	2	1	63	3	1.71	8.3
3	3	1	63	3	1.76	7.6
3	4	1	63	3	1.98	8.2
3	5	1	63	3	1.18	7.9
3	1	2	77	3	2.00	7.6
3	2	2	77	3	3.13	8.0
3	3	2	77	3	2.32	7.2
3	4	2	77	3	2.39	7.2
3	5	2	77	3	.	.
3	1	3	84	3	1.30	7.6
3	2	3	84	3	1.50	8.4
3	3	3	84	3	1.97	7.4
3	4	3	84	3	2.02	7.5
3	5	3	84	3	0.67	8.3
3	1	4	84	3	1.25	7.8
3	2	4	84	3	.	.
3	3	4	84	3	2.13	7.4
3	4	4	84	3	1.23	7.9
3	5	4	84	3	0.73	8.6

Table C6: Leachate metal concentrations in mg L^{-1} for the BTEX column leaching study.

				Leachate metal concentrations (mg L^{-1})											
rep	rate	moisture	day	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al
3	1	1	0	26.6	116.6	30.5	13.8	84.5	0.4	0.1	0.0	0.0	0.0	0.0	0.0
3	2	1	0	27.8	124.6	31.6	15.6	100.8	0.4	0.2	0.0	0.0	0.0	0.0	0.0
3	3	1	0	23.8	136.3	34.8	15.4	120.8	0.3	0.2	0.0	0.0	0.0	0.0	0.0
3	4	1	0	25.0	116.4	30.0	13.5	101.8	0.5	0.2	0.0	0.0	0.0	0.0	0.0
3	5	1	0	18.5	81.9	20.4	11.3	89.2	0.3	0.2	0.0	0.0	0.0	0.0	0.0
2	1	1	0	24.1	89.6	20.4	11.0	79.5	0.2	0.2	0.0	0.0	0.0	0.0	0.0
2	2	1	0	18.4	120.6	30.5	13.7	95.7	0.3	0.2	0.0	0.0	0.0	0.0	0.0
2	3	1	0	19.4	87.5	21.8	12.0	83.5	0.2	0.2	0.0	0.0	0.0	0.0	0.0
2	4	1	0	21.2	90.0	22.5	11.7	82.8	0.2	0.2	0.0	0.0	0.0	0.0	0.0
2	5	1	0	22.1	73.9	18.3	10.6	83.2	0.2	0.1	0.0	0.0	0.0	0.0	0.0
1	1	1	0	27.7	81.1	20.8	11.6	85.3	0.4	0.2	0.0	0.0	0.0	0.0	0.0
1	2	1	0	42.3	101.9	25.3	12.8	98.8	0.8	0.3	0.0	0.0	0.0	0.0	0.0
1	3	1	0	54.4	141.9	35.6	15.5	107.5	0.8	0.3	0.0	0.0	0.0	0.0	0.0
1	4	1	0	.	.	.	.	.	.	.	.	.	.	.	.
1	5	1	0	44.7	87.0	21.2	11.8	96.0	0.9	0.4	0.0	0.0	0.0	0.0	0.0
cont	cont	cont	0	49.2	34.8	16.3	5.7	53.6	0.1	0.0	0.0	0.0	0.0	0.0	0.0
3	1	2	14	46.8	100.8	25.7	15.5	81.5	0.4	0.2	0.0	0.0	0.0	0.0	0.0
3	2	2	14	64.5	100.2	24.7	13.2	81.2	1.2	0.1	0.0	0.0	0.0	0.0	0.0
3	3	2	14	71.0	137.2	35.5	15.6	93.9	1.1	0.0	0.0	0.0	0.0	0.0	0.0
3	4	2	14	78.8	192.8	49.1	17.9	119.1	0.9	0.1	0.0	0.0	0.0	0.0	0.0
3	5	2	14	44.3	107.8	27.0	53.6	90.7	1.1	0.1	0.0	0.0	0.0	0.0	0.0
2	1	2	14	39.0	91.8	23.9	11.5	74.7	0.2	0.1	0.0	0.0	0.0	0.0	0.0
2	2	2	14	43.0	105.3	26.6	12.1	75.0	0.2	0.1	0.0	0.0	0.0	0.0	0.0
2	3	2	14	57.6	95.2	24.4	12.0	73.0	0.2	0.0	0.0	0.0	0.0	0.0	0.0
2	4	2	14	50.1	143.9	34.6	14.2	87.9	0.3	0.2	0.0	0.0	0.0	0.0	0.0
2	5	2	14	28.5	120.6	30.0	13.2	93.1	0.2	0.1	0.0	0.0	0.0	0.0	0.0
1	1	2	14	44.5	78.8	20.5	10.5	69.5	0.3	0.1	0.0	0.0	0.0	0.0	0.0

Table C6: Continued

				Leachate metal concentrations (mg L^{-1})											
rep	rate	moisture	day	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al
1	2	2	14	39.8	100.4	26.2	12.0	78.5	0.4	0.1	0.0	0.0	0.0	0.0	0.0
1	3	2	14	48.5	80.2	21.7	11.1	73.7	0.4	0.7	0.0	0.0	0.0	0.0	0.0
1	4	2	14	43.0	95.5	24.4	11.4	75.3	0.3	0.1	0.2	0.1	0.0	0.0	0.4
1	5	2	14	26.7	107.6	28.1	13.7	86.1	0.4	0.2	0.6	0.0	0.0	0.5	1.4
cont	cont	cont	14	46.0	31.6	15.9	5.5	50.5	0.1	0.0	0.0	0.0	0.0	0.0	0.0
3	1	3	28	70.1	123.2	32.4	10.8	83.1	2.3	0.1	0.0	0.0	0.0	0.0	0.0
3	1	4	28	84.8	105.7	28.5	10.1	77.6	4.1	0.1	0.0	0.0	0.0	0.0	0.0
3	2	3	28	94.5	126.9	33.6	11.5	83.1	3.0	0.1	0.0	0.0	0.0	0.0	0.0
3	2	4	28	89.4	120.8	32.6	11.7	73.3	3.2	0.1	0.0	0.0	0.0	0.0	0.0
3	3	3	28	100.4	156.0	40.6	12.1	91.5	2.3	0.1	0.0	0.0	0.0	0.0	0.0
3	3	4	28	100.8	154.1	40.8	12.6	77.1	3.7	0.1	0.0	0.0	0.0	0.0	0.0
3	4	3	28	90.4	174.9	45.0	13.0	102.5	2.3	0.1	0.0	0.0	0.0	0.0	0.0
3	4	4	28	112.1	157.0	42.1	13.3	91.6	3.5	0.1	0.0	0.0	0.0	0.0	0.0
3	5	3	28	79.4	153.9	40.1	12.5	109.9	2.4	0.2	0.0	0.0	0.0	0.0	0.0
3	5	4	28	84.2	155.4	40.7	12.9	129.4	3.6	0.2	0.0	0.0	0.0	0.0	0.0
2	1	3	28	58.9	120.4	31.3	9.8	76.3	0.8	0.1	0.0	0.0	0.0	0.0	0.0
2	1	4	28	55.2	105.1	27.9	11.1	73.1	0.8	0.1	0.0	0.0	0.0	0.0	0.0
2	2	3	28	62.1	136.9	34.9	11.1	78.0	0.7	0.1	0.0	0.1	0.0	0.0	0.0
2	2	4	28	68.9	155.8	40.1	12.0	77.4	0.5	0.1	0.0	0.0	0.0	0.0	0.0
2	3	3	28	62.5	142.4	33.8	11.1	76.4	0.5	0.1	0.0	0.1	0.0	0.0	0.0
2	3	4	28	60.9	119.6	30.7	10.5	68.3	0.3	0.0	0.0	0.0	0.0	0.0	0.0
2	4	3	28	51.6	181.5	44.1	12.3	86.3	0.2	0.2	0.0	0.0	0.0	0.0	0.0
2	4	4	28	73.8	134.1	34.7	11.3	79.9	0.7	0.0	0.0	0.0	0.0	0.0	0.0
2	5	3	28	40.9	137.0	34.7	10.4	98.0	0.3	0.1	0.0	0.0	0.0	0.0	0.0
2	5	4	28	37.2	129.0	32.6	10.5	102.6	0.9	0.1	0.0	0.0	0.0	0.0	0.0
1	1	3	28	58.5	115.5	30.2	11.2	73.6	1.0	0.1	0.0	0.0	0.0	0.0	0.0
1	1	4	28	58.2	124.2	32.9	11.3	68.3	0.7	0.1	0.0	0.0	0.0	0.0	0.0

Table C6: Continued

				Leachate metal concentrations (mg L^{-1})											
rep	rate	moisture	day	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al
1	2	3	28	53.6	131.8	35.1	37.7	73.5	0.4	0.1	0.0	0.0	0.0	0.0	0.0
1	2	4	28	58.5	120.7	32.2	12.0	72.2	0.9	0.1	0.0	0.0	0.0	0.0	0.0
1	3	3	28	62.3	126.1	33.2	10.8	81.8	1.0	0.1	0.0	0.0	0.0	0.0	0.0
1	3	4	28	59.3	119.2	31.4	11.1	79.7	1.0	0.1	0.0	0.0	0.0	0.0	0.0
1	4	3	28	56.2	99.9	27.1	9.1	73.2	0.7	0.1	0.0	0.0	0.0	0.0	0.0
1	4	4	28	63.0	103.7	28.7	10.3	75.7	0.9	0.1	0.0	0.0	0.0	0.0	0.0
1	5	3	28	33.1	131.7	34.2	11.1	92.3	0.4	0.1	0.0	0.0	0.0	0.0	0.0
1	5	4	28	38.3	131.5	34.7	11.3	101.6	0.9	0.1	0.0	0.0	0.0	0.0	0.0
cont	cont	cont	28	39.1	29.1	14.8	5.0	44.8	0.1	0.0	0.0	0.0	0.0	0.0	0.0
1	1	1	35	68.0	85.2	23.2	11.1	64.2	1.4	0.1	0.0	0.0	0.0	0.0	0.0
1	2	1	35	76.4	101.9	27.6	12.2	77.4	1.8	0.1	0.0	0.0	0.0	0.0	0.0
1	3	1	35	61.5	79.2	20.8	31.2	56.6	0.9	0.0	0.0	0.0	0.0	0.0	0.0
1	4	1	35	68.8	71.3	21.0	9.6	63.3	1.2	0.0	0.0	0.0	0.0	0.0	0.0
1	5	1	35	63.6	111.4	28.9	12.7	89.9	1.2	0.1	0.0	0.0	0.0	0.0	0.0
2	1	1	35	79.3	89.0	28.9	11.4	80.8	1.2	0.1	0.1	0.0	0.0	0.1	0.0
2	2	1	35	86.6	102.6	25.7	10.4	77.2	1.7	0.1	0.0	0.0	0.0	0.0	0.0
2	3	1	35	62.8	71.6	19.3	8.7	58.9	1.2	0.0	0.0	0.0	0.0	0.0	0.0
2	4	1	35	85.9	106.3	26.5	10.2	71.0	0.9	0.0	0.0	0.0	0.0	0.0	0.0
2	5	1	35	44.8	71.0	20.3	8.9	79.2	0.3	0.0	0.1	0.1	0.0	0.2	0.1
3	1	1	35	92.6	95.9	24.1	9.9	74.4	2.4	0.1	0.0	0.0	0.0	0.0	0.0
3	2	1	35	135.3	119.5	29.7	11.8	85.4	4.9	0.1	0.0	0.0	0.0	0.0	0.0
3	3	1	35	119.0	82.3	21.1	9.7	63.2	4.3	0.1	0.0	0.0	0.0	0.0	0.0
3	4	1	35	132.3	112.8	27.9	11.6	84.1	4.4	0.1	0.0	0.0	0.0	0.0	0.0
3	5	1	35	117.7	131.7	32.8	12.7	113.2	4.4	0.1	0.0	0.0	0.0	0.0	0.0
cont	cont	cont	35	41.6	28.7	14.5	5.2	44.1	0.2	0.0	0.0	0.0	0.0	0.0	0.0
1	1	2	49	76.8	101.8	29.1	37.4	72.2	1.4	0.0	0.0	0.0	0.0	0.0	0.0
1	2	2	49	84.4	96.5	26.3	11.6	73.2	2.3	0.1	0.0	0.0	0.0	0.0	0.0

Table C6: Continued

				Leachate metal concentrations (mg L^{-1})											
rep	rate	moisture	day	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al
1	3	2	49	92.8	112.8	31.2	12.5	82.2	2.6	0.1	0.0	0.0	0.0	0.0	0.0
1	4	2	49	86.2	111.4	30.0	11.0	64.7	1.8	0.1	0.0	0.0	0.0	0.0	0.0
1	5	2	49	58.2	124.6	32.9	11.9	121.9	1.3	0.1	0.0	0.0	0.0	0.0	0.0
2	1	2	49	75.2	118.0	31.3	9.9	75.7	0.9	0.0	0.0	0.0	0.0	0.0	0.0
2	2	2	49	107.2	147.0	37.7	10.9	67.3	0.9	0.1	0.0	0.0	0.0	0.0	0.0
2	3	2	49	78.4	109.8	29.4	9.8	69.7	0.8	0.0	0.0	0.0	0.0	0.0	0.0
2	4	2	49	104.9	162.7	42.1	13.4	85.2	1.8	0.0	0.0	0.0	0.0	0.0	0.0
2	5	2	49	56.2	145.1	37.3	11.2	118.4	0.5	0.1	0.0	0.0	0.0	0.0	0.0
3	1	2	49	126.2	138.0	36.9	12.7	84.6	2.1	0.0	0.0	0.0	0.0	0.0	0.0
3	2	2	49	141.8	136.2	33.7	12.2	83.5	5.0	0.1	0.0	0.0	0.0	0.0	0.0
3	3	2	49	139.4	154.7	41.7	13.6	99.1	4.5	0.1	0.0	0.0	0.0	0.0	0.0
3	4	2	49	146.6	201.9	50.9	14.5	100.1	3.8	0.0	0.0	0.0	0.0	0.0	0.0
3	5	2	49	83.0	119.7	29.1	10.5	121.6	2.4	0.1	0.0	0.0	0.0	0.0	0.0
cont	cont	cont	49	40.4	30.7	15.3	5.2	45.6	0.1	0.0	0.0	0.0	0.0	0.0	0.0
1	1	3	56	93.2	120.5	30.7	13.3	73.9	1.7	0.0	0.0	0.0	0.0	0.0	0.0
1	1	4	56	97.2	92.7	24.4	10.9	48.2	1.1	0.0	0.0	0.0	0.0	0.0	0.0
1	2	3	56	117.6	180.9	42.3	13.7	89.6	1.1	0.0	0.0	0.1	0.0	0.0	0.0
1	2	4	56	107.8	111.8	29.5	11.5	50.0	0.8	0.0	0.0	0.0	0.0	0.0	0.0
1	3	3	56	97.1	121.5	31.7	12.5	69.9	1.2	0.0	0.0	0.0	0.0	0.0	0.0
1	3	4	56	95.2	126.3	31.8	12.7	55.2	1.0	0.0	0.0	0.2	0.0	0.0	0.1
1	4	3	56	92.6	120.0	31.6	11.6	62.3	1.0	0.0	0.0	0.0	0.0	0.0	0.0
1	4	4	56	106.7	123.8	32.7	11.9	62.6	1.0	0.0	0.0	0.0	0.0	0.1	0.0
1	5	3	56	60.6	118.4	30.6	10.4	119.3	0.9	0.0	0.0	0.0	0.0	0.0	0.0
1	5	4	56	62.5	64.0	16.7	9.9	108.1	1.6	0.0	0.0	0.0	0.0	0.0	0.0
2	1	3	56	114.5	165.6	42.1	14.0	95.8	1.5	0.0	0.1	0.1	0.0	0.0	0.0
2	1	4	56	95.2	104.0	26.0	10.7	57.7	1.1	0.0	0.0	0.0	0.0	0.0	0.0
2	2	3	56	118.4	131.9	31.9	11.8	76.6	2.2	0.0	0.0	0.0	0.0	0.0	0.0

Table C6: Continued

				Leachate metal concentrations (mg L^{-1})											
rep	rate	moisture	day	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al
2	2	4	56	132.6	147.4	36.6	10.8	41.1	1.0	0.1	0.0	0.0	0.0	0.0	0.0
2	3	3	56	111.1	145.7	35.8	11.9	55.9	0.9	0.0	0.0	0.0	0.0	0.0	0.0
2	3	4	56	110.9	129.4	32.6	9.9	34.1	0.4	0.0	0.0	0.0	0.0	0.4	0.0
2	4	3	56	121.3	189.9	44.7	14.7	59.8	1.1	0.1	0.0	0.0	0.0	0.0	0.0
2	4	4	56	128.9	148.5	36.7	13.6	79.9	0.9	0.0	0.0	0.0	0.0	0.0	0.0
2	5	3	56	49.3	84.5	21.3	9.6	106.7	0.9	0.1	0.0	0.0	0.0	0.0	0.0
2	5	4	56	69.8	90.0	20.0	9.6	120.0	1.6	0.1	0.0	0.0	0.0	0.0	0.0
3	1	3	56	146.2	130.7	33.8	12.8	95.6	4.3	0.1	0.0	0.0	0.0	0.0	0.0
3	1	4	56	113.0	89.8	25.9	11.0	69.0	3.8	0.0	0.0	0.0	0.0	0.0	0.0
3	2	3	56	163.4	147.5	39.7	14.6	91.4	4.9	0.0	0.0	0.0	0.0	0.0	0.0
3	2	4	56	156.9	140.5	35.8	13.7	57.7	4.9	0.0	0.0	0.0	0.0	0.0	0.0
3	3	3	56	179.6	199.3	49.9	15.4	108.3	4.0	0.0	0.0	0.0	0.0	0.0	0.0
3	3	4	56	195.0	173.4	47.0	15.8	113.0	3.9	0.0	0.0	0.0	0.0	0.0	0.0
3	4	3	56	174.2	182.5	45.2	15.2	95.3	3.8	0.0	0.0	0.0	0.0	0.0	0.0
3	4	4	56	125.6	96.4	25.9	10.8	51.1	3.3	0.1	0.0	0.0	0.0	0.0	0.0
3	5	3	56	119.3	87.2	25.8	11.3	127.9	4.8	0.1	0.2	0.0	0.0	0.1	0.2
3	5	4	56	122.0	89.5	22.0	11.1	137.3	4.6	0.1	0.1	0.0	0.0	0.0	0.1
cont	cont	cont	56	40.2	37.7	15.9	5.3	47.2	0.1	0.0	0.0	0.0	0.0	0.0	0.0
1	1	1	63	100.5	103.4	28.1	11.8	71.1	1.6	0.0	0.0	0.0	0.0	0.0	0.0
1	2	1	63	144.7	162.8	41.1	14.7	103.2	2.2	0.0	0.0	0.0	0.0	0.0	0.0
1	3	1	63	92.9	102.0	27.1	10.1	42.2	2.2	0.0	0.0	0.0	0.0	0.0	0.0
1	4	1	63	116.8	130.2	35.7	12.4	87.4	2.2	0.0	0.0	0.0	0.0	0.0	0.0
1	5	1	63	64.4	102.0	26.4	9.7	120.0	1.2	0.0	0.0	0.0	0.0	0.0	0.0
2	1	1	63	122.9	154.8	41.3	12.7	98.0	1.8	0.0	0.0	0.0	0.0	0.0	0.0
2	2	1	63	118.5	134.3	33.9	12.2	80.2	1.8	0.0	0.0	0.0	0.0	0.0	0.0
2	3	1	63	72.4	100.5	25.9	8.5	76.1	0.8	0.0	0.0	0.0	0.0	0.0	0.0
2	4	1	63	136.6	162.2	40.4	11.7	95.1	1.2	0.0	0.0	0.0	0.0	0.0	0.2

Table C6: Continued

				Leachate metal concentrations (mg L^{-1})											
rep	rate	moisture	day	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al
2	5	1	63	50.9	81.2	21.4	7.4	104.5	0.8	0.0	0.0	0.0	0.0	0.0	0.0
3	1	1	63	98.8	123.9	31.9	9.0	87.9	1.7	0.0	0.0	0.0	0.0	0.0	0.0
3	2	1	63	170.3	140.0	35.5	12.7	93.5	4.4	0.0	0.0	0.0	0.0	0.0	0.0
3	3	1	63	166.0	136.1	34.1	11.6	82.0	3.6	0.0	0.0	0.0	0.0	0.0	0.0
3	4	1	63	189.0	165.7	40.1	13.8	97.4	5.4	0.0	0.0	0.0	0.0	0.0	0.0
3	5	1	63	115.9	97.5	23.6	9.6	114.1	4.1	0.1	0.0	0.0	0.0	0.0	0.0
cont	cont	cont	63	41.2	30.2	15.2	5.2	45.1	0.2	0.0	0.0	0.1	0.0	0.0	0.0
1	1	2	77	148.2	165.8	43.1	15.6	105.6	2.3	0.1	0.1	0.0	0.0	0.0	0.2
1	2	2	77	143.6	140.5	37.9	14.9	78.9	3.8	0.1	0.0	0.0	0.0	0.0	0.0
1	3	2	77	166.4	176.0	48.1	18.0	93.2	4.7	0.0	0.0	0.0	0.0	0.0	0.0
1	4	2	77	184.1	214.7	61.3	17.8	43.6	4.6	0.1	0.0	0.0	0.0	0.1	0.0
1	5	2	77	110.0	107.8	27.4	13.5	186.8	1.8	0.1	0.1	0.1	0.0	0.0	0.4
2	1	2	77	103.5	151.3	39.5	10.6	99.6	1.8	0.0	0.0	0.0	0.0	0.0	0.0
2	2	2	77	149.7	161.7	39.8	12.3	113.3	3.4	0.1	0.0	0.0	0.0	0.0	0.0
2	3	2	77	129.5	131.5	30.1	10.8	64.3	3.5	0.0	0.0	0.0	0.0	0.0	0.0
2	4	2	77	176.9	199.5	52.7	16.2	95.0	2.8	0.1	0.0	0.0	0.0	0.0	0.0
2	5	2	77	67.2	70.7	16.7	8.8	139.9	0.5	0.1	0.2	0.1	0.0	0.1	0.6
3	1	2	77	174.4	185.7	38.8	14.3	128.0	6.8	0.1	0.0	0.1	0.0	0.0	0.0
3	2	2	77	275.9	284.9	76.6	18.6	108.3	4.8	0.0	0.0	0.0	0.0	0.0	0.0
3	3	2	77	227.2	212.1	49.3	17.5	155.1	9.0	0.0	0.0	0.1	0.0	0.0	0.0
3	4	2	77	187.6	245.7	55.6	17.0	121.0	3.3	0.0	0.0	0.1	0.0	0.0	0.0
3	5	2	77	86.9	88.0	21.4	9.6	135.4	0.8	0.1	0.3	0.1	0.0	0.1	0.8
cont	cont	cont	77	41.9	30.4	15.4	5.2	50.5	0.2	0.0	0.0	0.0	0.0	0.0	0.0
1	1	3	84	114.2	119.4	29.3	13.7	73.2	1.7	0.0	0.0	0.0	0.0	0.0	0.0
1	1	4	84	97.3	70.6	18.1	10.2	52.3	1.3	0.0	0.0	0.0	0.0	0.0	0.0
1	2	3	84	147.9	162.9	40.5	13.9	101.0	1.4	0.0	0.0	0.0	0.0	0.0	0.0
1	2	4	84	112.9	100.8	26.0	10.3	39.8	0.8	0.0	0.0	0.0	0.0	0.0	0.0

Table C6: Continued

				Leachate metal concentrations (mg L^{-1})											
rep	rate	moisture	day	Na	Ca	Mg	K	S	B	P	Fe	Zn	Cu	Mn	Al
1	3	3	84	99.2	96.4	24.0	11.6	56.2	1.3	0.0	0.0	0.0	0.0	0.0	0.0
1	3	4	84	115.5	118.0	31.1	11.1	35.3	1.3	0.1	0.0	0.0	0.0	0.0	0.0
1	4	3	84	126.5	136.0	34.5	11.4	50.2	0.8	0.0	0.0	0.0	0.0	0.0	0.0
1	4	4	84	123.6	122.1	31.7	11.2	57.8	0.7	0.0	0.0	0.0	0.0	0.0	0.0
1	5	3	84	77.9	43.0	10.9	7.7	116.9	0.8	0.1	0.2	0.0	0.0	0.0	0.4
1	5	4	84	68.6	31.1	8.1	6.3	86.7	0.8	0.0	0.0	0.0	0.0	0.0	0.0
2	1	3	84	130.2	134.0	32.8	11.8	87.1	1.6	0.0	0.0	0.0	0.0	0.0	0.0
2	1	4	84	116.0	86.7	22.7	10.5	74.5	1.0	0.0	0.0	0.0	0.0	0.0	0.0
2	2	3	84	148.0	134.7	33.3	12.0	94.4	2.8	0.1	0.0	0.0	0.0	0.0	0.0
2	2	4	84	146.3	131.7	31.5	11.1	62.8	1.1	0.0	0.0	0.0	0.0	0.0	0.0
2	3	3	84	118.6	123.5	31.0	8.8	34.1	1.2	0.1	0.0	0.0	0.0	0.0	0.0
2	3	4	84	109.5	113.6	28.9	7.5	30.5	0.5	0.0	0.0	0.0	0.0	0.4	0.0
2	4	3	84	151.6	169.5	42.8	12.5	86.7	0.4	0.0	0.0	0.0	0.0	0.0	0.0
2	4	4	84	119.6	113.8	28.3	12.4	79.3	1.1	0.0	0.0	0.0	0.0	0.0	0.0
2	5	3	84	55.6	25.5	6.2	5.3	59.5	0.2	0.1	0.0	0.0	0.0	0.0	0.0
2	5	4	84	67.6	61.9	13.0	7.6	85.0	1.6	0.1	0.0	0.0	0.0	0.0	0.0
3	1	3	84	131.5	110.4	30.0	10.8	76.4	2.9	0.0	0.0	0.0	0.0	0.0	0.0
3	1	4	84	129.8	99.1	28.1	11.1	73.7	2.9	0.0	0.0	0.0	0.0	0.0	0.0
3	2	3	84	164.2	124.4	32.3	13.1	94.5	4.2	0.1	0.0	0.0	0.0	0.0	0.0
3	2	4	84	.	.	.	.	.	.	.	.	.	.	.	.
3	3	3	84	195.8	173.7	46.4	15.0	113.3	3.7	0.0	0.0	0.0	0.0	0.0	0.0
3	3	4	84	223.6	176.6	45.6	14.5	127.8	3.5	0.0	0.0	0.0	0.0	0.0	0.0
3	4	3	84	196.6	190.0	44.0	15.1	77.3	2.9	0.1	0.0	0.0	0.0	0.0	0.0
3	4	4	84	128.9	104.2	27.5	11.2	60.6	3.0	0.0	0.0	0.0	0.0	0.0	0.0
3	5	3	84	96.9	47.2	11.5	7.7	116.7	2.7	0.1	0.1	0.0	0.0	0.0	0.0
3	5	4	84	104.4	48.5	12.1	8.1	128.0	2.4	0.1	0.0	0.0	0.0	0.0	0.0
cont	cont	cont	84	42.4	30.8	15.3	5.1	50.6	0.1	0.0	0.0	0.0	0.0	0.0	0.0

VITA

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Candidate for the Degree of

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