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POTASSIUM SILICATE DRILLING FLUID AS A LAND RECLAMATION AMENDMENT

by

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ABSTRACT

Drilling fluid, also referred to as drilling mud, is used to lubricate and cool the drilling apparatus, transport drill cuttings to the surface and seal off porous geologic formations. Disposal of drilling fluid is a growing concern to the global oil and gas industry as it can require extensive waste management and result in significant land disturbance. The oil and gas industry has developed potassium silicate drilling fluids (PSDF), a relatively new advanced chemical gel drilling fluid system, to reduce sodium concentrations, which are considered to have a detrimental environmental impact. Hypothetically, high concentrations of potassium in PSDF could serve as a nutrient amendment for land reclamation, thus not only would land disposal be practical, but also provide benefits for reclamation. Environmental impacts associated with PSDF disposal are unclear. Before guidelines can be developed and modified, it is necessary to assess the intensity of disturbance on soil-plant-water systems by disposal of PSDF from land based operations. In this study, responses of soils of three textures, two plant species and leachate were studied when raw, spent once and spent twice PSDF were incorporated or sprayed at six rates with and without fertilizer.

Raw and spent PSDF had no detrimental effects on soil, vegetation and water at rates ≤ 60 m^3 ha^{-1}. Addition of PSDF to soil generally resulted in positive changes to soil physical, chemical and microbiological properties and enhanced plant establishment, development and yield. Slightly acidic and medium textured soils with inorganic fertilizer benefitted most by addition of PSDF. Both an agricultural crop, barley *(Hordeum vulgare* L.), and a native grass, slender wheat grass *(Agropyron trachycaulum* (Link) Malte ex H.F. Lewis) survived and developed through physiological growth stages. Constituents in PSDF significantly increased soil potassium concentrations, reduced hydraulic conductivity, stimulated populations of the microbial community and increased their diversity. Incorporating PSDF into soil provided greater benefits for vegetation than spraying it, increasing soil water content and macronutrients. Effects of PSDF recycling times on vegetation varied.

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CHAPTER 1. INTRODUCTION

1. Overview

Disturbances, both natural and anthropogenic, shape an ecosystem by influencing its composition, structure and functional processes (Dale et al. 2001). In Alberta, Canada, anthropogenic disturbances, such as surface coal mining, pipeline and well site construction and oil sands extraction, have had the greatest impacts on prairie and boreal forest. Post disturbance reclamation, commonly includes construction of topographic, soil and plant conditions which may not be identical to predisturbance, but which can function adequately in the ecosystem of which is was a part (Munshower 1994). Understanding disturbance processes and mechanisms is essential to successful land reclamation (Bradshaw 1987).

During the past few decades, extensive oil and gas well drilling has occurred and will continue to occur in western Canada. Drilling produces large volumes of drilling waste, which can pose significant threats to soil and water quality and plant establishment and development. The oil and gas industry has developed technologies and practices to reduce environmental damage with advanced chemical gel drilling fluid systems rather than traditional drilling fluids. Environmental impacts associated with advanced drilling wastes disposal are unclear. Although no two drilling wastes are identical, the disturbances cause similar problems for land reclamation but with varying intensity. The intensity of disturbance by disposal of new drilling wastes from land based operations remains a key issue for land reclamation and policy making.

2. Research Background

2.1 Drilling Waste

In oil and gas operations, drilling fluids, also referred to as drilling muds, are used to lubricate and cool the drilling apparatus, transport drill cuttings to the surface and seal off porous geologic formations. Drilling fluids typically consist of bentonite and a range of additives mixed with fresh water or hydrocarbons. Mud types are classified as water based, oil based and synthetic based. Water based muds are used for most types of drilling; oil based and synthetic based muds are most used in offshore wells or other water sensitive formations. Advanced gel chemical drilling fluids result from a water based drilling mud system in which a significant primary component such as a salt or polymer has been added to aid in drilling (Energy Resources Conservation Board 2012). Examples include potassium silicate, potassium sulphate, potassium nitrate and amines.

Drilling waste, including spent drilling fluids and cuttings generated while drilling oil and gas wells, have been identified as an environmental issue because of the pollutants they carry. A large drilling waste volume is the first obstacle to an environmentally friendly discharge. Theoretically, all drilling cuttings could be mechanically removed, and fluid recycled in the system. In reality, an increased fluid volume results from disintegration of cuttings during transport to the surface and limited efficiency of cuttings removal by the solids control separators (Wojtanowicz 2008). The way to dispose of drilling waste will greatly depend on characteristics of the area, including climate, land use, proximity to sensitive areas and regulatory requirements of the jurisdiction in which the drilling occurs.

Disposal costs increase with disposal area increases with stringent disposal limitations. The American Petroleum Institute, for example, has limited depth for land spreading of drilling fluids based on soil infiltration rates in the disposal area that would not cause fluid run off (American Petroleum Institute 1993). In Alberta, the Energy Resources Conservation Board allows discharging some types of drilling waste directly into the environment, but there are compliance criteria for loading rates, disposal area soils, methods and soil endpoints after discharge (Energy Resources Conservation Board 2012). Spent advanced gel chemical drilling fluid systems are only allowed to be deep well disposed by a third party which would cost approximately \$128 per m^3 in Alberta before 2012 (Alberta Environment and Sustainable Resource Development 2010a). The volume of drilling wastes to drill a hole is approximately three times the volume of the hole generally (United States Army Corps of Engineers 2001) and a typical shallow gas well (250 to 650 m deep) in Alberta will generate around 68 m^3 of spent drilling fluid (Zvomuya et al. 2009). Canada has the second highest number of active oil and gas wells worldwide (Industry Canada 2010). In Alberta alone, approximately 213,000 well sites were active up to December 2012 (Alberta

Environment and Sustainable Resource Development 2012). From these well sites an estimated 14.5 million m^3 of drilling wastes in Alberta will be disposed of by deep well application, costing approximately 1.9 billion dollars.

2.2 Drilling Waste Disposal Regulatory Requirements

Disposal of drilling waste from oil and gas activities can require extensive waste management and result in significant land disturbance. Regulatory requirements exist in Alberta that define drilling licenses, disposal of drilling wastes and reclamation of affected sites to equivalent land capability. These requirements consist of various federal and provincial acts, regulations and criteria that address land application of drilling waste.

In 2007 when this research was initiated, there were very few regulatory requirements dealing exclusively with disposal of advanced gel chemical drilling fluid systems, such as potassium silicate drilling fluids used in this study. However, much of the drilling waste management regulatory requirements included a special approval for advanced gel chemical drilling waste disposal in addition to disposal of water based drilling fluids. Therefore, drilling waste regulatory requirements constitute the primary framework for addressing advanced gel chemical drilling waste disposal from a regulatory perspective.

2.2.1 Federal requirements

There are numerous acts, regulations, standards and guidelines governing oil and gas activity in Canada. However, this section addresses only the ones that are specifically relevant to the disposal of drilling waste.

The Canadian Environmental Assessment Act applies a process for environmental assessment and review of projects proposed on federal lands that require federal action or decisions (Government of Canada 2012). This act is administered by the Canadian Environmental Assessment Agency. In practice, a proponent or operator of an oil and gas project on federal lands will interact with a federal responsible authority - the National Energy Board. Sections 15 and 19 of the act state the need for the authority to ensure appropriate measures are implemented to mitigate any significant adverse environmental effects of a project. This includes measures of environmental effects by drilling waste disposal, as deemed necessary and appropriate for the project by the authority. Environmental factors are considered, including physical and meteorological environment; soil, soil productivity and vegetation; wetlands, water quality and quantity; fish, wildlife, and their habitat; species at risk; heritage resources; traditional land and resource use; human health, aesthetics and noise. Typically these authorities for projects apply the appropriate provincial standards for drilling waste disposal.

The Canadian Environmental Quality Guidelines are nationally endorsed science based goals for the quality of atmospheric, aquatic and terrestrial ecosystems, including the Canadian Water Quality Guidelines and Canadian Soil Quality Guidelines for the Protection of Environment and Human Health (Canadian Council of Ministers of the Environment 2012). They are defined as numerical concentrations or narrative statements that are recommended as levels that should result in negligible risk to biota, their functions or any interactions that are integral to sustaining the health of ecosystems and the designated resource uses they support. These guidelines are not mandatory across the country, and provincial and territorial jurisdictions may have or may develop their own science based environmental assessment tools (e.g., criteria, guidelines, objectives, standards), which may be implemented within their respective jurisdictions. The legislative authority for implementation of such guidelines and other environmental assessment tools lies primarily with each provincial or territorial jurisdiction, with the exception of federal lands.

2.2.2 Alberta provincial requirements

Pursuant to the Oil and Gas Conservation Act and its regulations, the Energy Resources Conservation Board is responsible for developing and maintaining requirements for treatment and disposal of drilling waste generated in Alberta (Alberta Energy Resources Conservation Board 2012). Drilling waste disposal has been regulated in Alberta since 1975, when the Energy Resources Conservation Board released Interim Directive ID-OG-75-2 for sump fluid disposal requirements. Interim Directive (ID) 93-1 and Guide 50 for drilling waste management superseded this document in June 1993. After review of data from over 4,200 wells in 1995, the Energy Resources Conservation Board issued a revision to Guide 50 in 1996, which was introduced with Information Letter (IL)

96-13: Revision of Guide 50 Drilling Waste Management. Guide 50 has been renamed Directive 50. However, as environmental impacts from disposal of advanced gel chemical drilling waste were unknown, specific requirements were needed pursuant to Informational Letter (IL) 2001-3 for management of drilling wastes associated with advanced gel chemical systems. IL 2001-3 requires prior written approval from the Alberta Energy and Utilities Board for any land application of advanced gel chemical drilling wastes (e.g. potassium silicate, potassium sulphate). When applying for such approval, the waste producer must detail the type of advanced gel chemical drilling fluid to be used and the requested disposal option.

In May 2, 2012, Directive 50 was revised and brought into alignment with Alberta Tier 1 Soil and Groundwater Remediation Guidelines (Alberta Environment and Sustainable Resource Development 2010b), Alberta Soil and Water Quality Guidelines for Hydrocarbons at Upstream Petroleum Oil and Gas Sites (Alberta Environment 2001) and Soil Remediation Guidelines for Barite: Environmental Health and Human Health (Alberta Environment 2009).

IL 2001-3 was rescinded with the publication of the 2012 edition of Directive 050, and drilling waste from advanced gel chemical mud systems must now be managed following requirements of the 2012 edition. Requirements for land spraying drilling waste, and soil salt, hydrocarbon and metal endpoints from the newest edition are presented in Figure 1.1 and Tables 1.1, 1.2 and 1.3.

In Alberta, when issuing reclamation certificates, oil and gas operators are required to show compliance with Alberta Environment and Sustainable Resource Development standards by submitting disposal information for drilling waste that has been disposed of on a well site or at a remote sump or land treatment site. There is a guideline, Assessing Drilling Waste Disposal Areas: Compliance Options for Reclamation Certification, for assessing drilling waste disposal areas for reclamation (Alberta Environment 2012).

Three compliance options have been developed to address the diversity of drilling wastes and available information types. All three compliance options are intended to deliver the same environmental results, which are based mainly on Directive 50. Each of the three options is discussed in the document and appendices are included to support technical procedures included in the options.

The first two compliance options exempt drilling waste disposal areas from a Phase 2 environmental site assessment. If the drilling waste disposal area does not meet the requirements specified in either of these options, compliance option 3 requires a Phase 2 environmental site assessment of the drilling waste disposal area. For drilling waste disposal that occurred before November 1, 2012, the guideline harmonizes Environment and Sustainable Resource Development requirements for drilling waste disposal areas with the 1996 Directive 50 whenever possible. For drilling waste disposal that occurred on or after November 1, 2012, the guideline harmonizes with the 2012 Directive 50.

2.2.3 Other western provincial requirements

In British Columbia and Saskatchewan, criteria for drilling waste disposal by method includes maximum disposal rates and acceptable increases in electrical conductivity, sodium adsorption ratio, sodium loading and nitrogen loading for water based drilling waste (Saskatchewan Ministry of Energy and Resources 2011, British Columbia Oil and Gas Commission 2006). In these provinces, advanced gel chem muds could be disposed in the same way as other water based drilling fluids at maximum rates of 40 m^3 ha^{-1} in summer or 20 m^3 ha^{-1} in winter or on frozen ground. The sodium loading rate must be < 150 kg ha^{-1} in British Columbia and 250 kg ha^{-1} in Saskatchewan. Spent drilling fluid can be disposed on cultivated land, where it is incorporated by cultivation, or spread on vegetated land without incorporation. Saskatchewan has special conditions for potassium sulphate and ammonium sulphate drilling muds, in which total loading sulphate rate for land spraying cannot exceed 33 kg ha^{-1} for oilseed crops and 22 kg ha^{-1} for cereal crops (Saskatchewan Ministry of Energy and Resources 2011).

2.3 Drilling Waste Environmental Impacts

Few studies have addressed the impact of disposal of spent drilling fluids on soil-plant-water systems. Some researchers found high soluble salts, heavy metals and petroleum residue content of used drilling fluids were detrimental to soil quality and plant growth (Zvomuya et al. 2011, Wojtanowicz 2008, Zvomuya et al. 2008, McFarland et al. 1994, McFarland et al. 1992a, Nelson et al. 1984, Miller et al. 1980). Others found positive or no impacts from drilling fluids applied at low rates to coarse textured soils native to arid regions (Bauder et al. 2005, Bauder et al. 1999, Macyk et al. 1990, Lesky et al. 1989, Tucker 1985, Moseley Jr. 1983, Miller et al. 1980). Differences among studies primarily resulted from waste properties, disposal method and disposal location.

Miller et al. (1980) studied effects of 31 drilling fluid components on green beans (*Phaseolus vulgaris* L.) and sweet corn (*Zea mays* L.) in a greenhouse. Plant growth was impacted with some components, such as phytotoxicity from diesel oil, high salt concentrations from potassium chloride (KCl) or sodium dichromate ($Na_2Cr_2O_7$), soil dispersion from sodium hydroxide (NaOH) and poor soil physical conditions and phytotoxins from lignosulfonate or starches. Miller and Pesaran (1980) tested the same plants with seven typical drilling fluid mixtures and soil at 1:1 and 1:4 volume ratios. Reduced plant growth was attributed to high soluble salts or high exchangeable sodium, but there may be a beneficial effect when high pH drilling fluid is applied to strongly acidic, medium textured soils.

Bioavailability to plants of trace elements in drilling fluids was investigated in a greenhouse study by Nelson et al. (1984). They assessed 0, 200 and 500 g kg^{-1} of drilling fluids with three barite sources on growth of swiss chard (*Beta vulgaris* L.) and ryegrass (*Lolium perenne* L.) in two soils. Barite drilling fluid mixture with trace metals dramatically increased plant tissue concentrations of zinc, copper, cadmium, lead and arsenic; barium, mercury and chromium were not plant available. Growth of both plant species was depressed, possibly resulting from increased soil alkalinity which decreased trace metal availability. Similar results were found in a laboratory study on chemical forms and mobility of heavy metals in drilling fluid when pH was adjusted (Deeley and Canter 1986).

Bauder et al. (1999) studied spent drilling fluids effects on sorghum (*Sorghum bicolour* L. Moench) and corn by applying 5 to 60 g dry drilling fluid kg^{-1} of soil. Plant dry matter yield increased as drilling fluid rates increased, attributed to increased zinc and iron available from drilling fluid. Sodium adsorption ratio increased slightly; pH and electrical conductivity had no significant increase.

Influence of total petroleum hydrocarbons from drilling fluid and crude oil on crop growth and soil, was studied in four experiments between 2003 and 2007 in a Croatia greenhouse (Kisic et al. 2009). Clean soil was mixed with drilling fluids at weight ratios of 1:1, 1:2 and 1:3, and mixed with crude oil at weight ratios of 1:2

and 1:3. The influence of total petroleum hydrocarbons on winter wheat (*Triticum aestivum* L.) density and yield was strongest in the first year. Drilling fluids had a stronger impact on soil chemical properties, while plant emergence, density and yield were more strongly affected by crude oil. Upon application of drilling fluids and crude oil, soil pH, organic matter and heavy metals varied little throughout the study, whereas soil concentrations of total petroleum hydrocarbons, mineral oils and polycyclic aromatic hydrocarbons were significantly reduced through bioremediation or aeration by tilling practices after the first year.

In an Alberta greenhouse, Macyk et al. (1990) assessed effects on soils and on growth and trace element content of brome grass (*Bromus inermis* Leyss.) from spent water based and oil based mud mixed with soil based on chloride content and liming potential. The chloride rates generally included 0, 200, 400, 600, 800, 1200 and 2400 kg ha^{-1}. Plant growth was enhanced when most water based mud was applied, with greatest height and yield occurring at lower waste application rates due to pH increase, potential micronutrient addition and/or improved soil physical properties. Lime content of the wastes was considered to be beneficial to acidic soils, but high electrical conductivity, pH, sodium adsorption ratio and chloride in water based mud mixtures and high petroleum hydrocarbons in oil based mud mixtures limited plant growth.

Fates of zinc, barium, chromium and chloride as a result of land farming reserve pit drilling fluids in silt loam and sandy textured soil with a volume ratio 1:8 were investigated in columns (Bates 1988). Ion uptake by bermuda grass (*Cynodon dactylon* (L.) Pers.) and transport were measured. Pollutant movement was a function of soil type retention capacity. Chloride added to soils was readily transported, affecting mobility of barium. There were higher available, but not detrimental, concentrations of zinc in sandy soils than in loam soils.

Columns were used to study remediation of drilling fluid affected soils (Kim 2003, Kim et al. 2002, Kim 1998). Hydraulic conductivity was adequate for leaching excessive salts without mobilizing chromium and contaminating ground water. Calcium carbonate ($CaCO_3$), gypsum, cationic organic polymers and calcium hydrogen phosphate ($CaHPO_4$) were added to sandy loam texture soils mixed with 3 % water based drilling fluid. Calcium carbonate, gypsum and cationic organic polymers increased hydraulic conductivity but decreased chromium mobility. Phosphate addition increased chromium movement. Soils with higher organic matter, cation exchange capacity and low manganese oxide were most suitable for drilling fluid disposal.

Little field research has been conducted on effects of drilling fluids on soils, plants and ground water. Vegetation cover decreased and soil erosion increased at drilling fluid disposal sites in Texas (McFarland et al. 1987). Burying spent drilling fluids at 30, 90 and 150 cm, covering with topsoil, and seeding to buffalo grass (*Buchloe dactyloides* (Nutt.) Engelm.) and four wing saltbush (*Atiplex canescens* (Pursh) Nutt.) led to upward soluble salt migration 30 to 60 cm into soil overlying drilling fluid after 44 months. Plant tissue salt concentrations increased, indicating shallow burial of drilling fluid may restrict plant growth (McFarland et al. 1994, McFarland et al. 1992a, McFarland et al. 1992b).

In a field study in southern Alberta, land spraying spent water based drilling fluid at 80 m^3 ha^{-1} increased bare ground but decreased lichen cover relative to the untreated control (Zvomuya et al. 2011). A single water based drilling fluid spraying at ≤ 20 m^3 ha^{-1} was considered unlikely to adversely affect native prairie vegetation in western Canada. Soil hydraulic conductivity decreased by 54 and 61 %, respectively, at 80 m^3 ha^{-1} water based spent drilling fluids relative to no drilling fluid and rates of 40 m^3 ha^{-1}, but these hydraulic effects did not persist one year following drilling mud application (Zvomuya et al. 2009).

In a field experiment to determine effects of water based drilling fluid on winter wheat (*Triticum aestivum* L.) soil was compacted by trucks, and pH, electrical conductivity and sodium adsorption ratio increased with increased application rate from 0 to 94 Mg ha^{-1} (Bauder et al. 2005). These increases did not affect crop production. Three years after land application of drilling fluids, most trace metals and nutrients did not significantly increase. Land application of water based muds at appropriate rates was deemed acceptable on well drained soils.

2.4 Potential Drilling Waste Uses

Potassium silicate drilling fluids (PSDF) are relatively new water based mud systems developed to reduce environmental impacts. Sodium concentrations in traditional drilling fluids were considered to have the most detrimental environmental impact; thus replacing sodium with potassium would reduce concentrations of sodium. Hypothetically, high concentrations of potassium in PSDF could serve as a nutrient amendment for reclamation, thus not only would land disposal be practical, but also provide benefits for reclamation.

Potassium silicate as a main component in drilling fluid may serve the same purpose as silicate slag and potassium nutrient for conventional agriculture and revegetation. Use of silica slag fertilizers in agriculture is widespread, particularly in sugar cane fields and rice paddies. In conventional agriculture, calcium silicate slag ($CaAl_2Si_2O_8$ or $CaSiO_3$) is commonly used as a silica fertilizer (Tisdale et al. 1999). Silicate slag applied at a rate of 1.5 to 3.0 t ha^{-1} is common practice in degraded paddy fields in Japan (Takahashi and Miyake 1977) and other foreign organic operations. One organic agriculture research farm in Taiwan that used silica slag mixed with manure and soybean meal reported a 25 % yield increase of high quality sponge gourd (*Luffa aegyptiaca* Mill.) (Hsieh and Hsieh 1989). Application wastewater from agricultural and industrial processes with high potassium concentrations increased overall soil fertility, particularly potassium and decreased hydraulic conductivity of the receiving soils (Arienzo et al. 2009).

Research has been conducted on foliar spraying with soluble potassium silicate on cucumber (*Cucumis sativus* L.), muskmelon (*C. melo* L.), and zucchini squash (*Cucurbita* pepo L.) reduced the severity of fungal diseases (*Sphaerotheca fuliginea*) in Canada (Menzies et al. 1992). Negative correlations between the potassium silicate content of barley (*Hordeum vulgare* L.) tissues and powdery mildew diseases (*Erysiphe gruminis* DC. f. sp. *Hordei* Em. Marchal) have been reported (Jiang et al. 1989).

Applications of drilling waste as an amendment to coarse textured soils in arid regions, or as a substrate for restoring coastal wetlands could be considered. In an arid zone field study drilling waste was land spread at low, medium and high rates (Moseley 1983). Vegetation growth increased with low application rates, from clay and sodium in drilling waste.

Drilling wastes are associated with drilling cuttings from the rock or shale parent material being drilled. Laboratory mesocosm experiments found mixes of drilling cuttings with residues of spent drilling fluids showed similar soil physical-chemical properties as single dredge spoil treatments, and were comparable to topsoil (control) in supporting wetland vegetation with big hog cane (*Spartina alterniflora* Loisel.), wiregrass (*Spartina patens* Aiton (Muhl)), oystergrass (*Spartina cynosuroides* L. Roth) and black mangrove (*Avicennia germinans* L.) growth (Willis et al. 2005, Veil 2003, Richardson and Hanson 2001, Kelley and Mendelssohn 1995). However, potassium silicate drilling fluid properties and effects on vegetation and soil are not well known. Such environmental impacts must be determined before land disposal of spent drilling fluids and its use in reclamation could be regulated.

3. Research Objectives And Hypotheses

3.1 Research Objectives

The objective of this research was to evaluate the effect of PSDF on soil chemical, physical and microbiological properties, and on plant growth and development to determine its potential for use in land reclamation. Research was conducted in the greenhouse and in the laboratory in several stages. Specific objectives were:

- To determine if application of raw and spent potassium PSDF affects plant emergence, establishment, growth, development and productivity.
- To determine whether application of raw and spent PSDF affects soil physical, chemical and microbiological properties.
- To determine mechanisms and intensity by which PSDF affects vegetation and soil.
- To determine disposal options and rates that could minimize negative impacts and maximize positive impacts of PSDF on soil and plant properties.
- To determine if the existing Energy Resources Conservation Board drilling waste disposal guidelines for non cultivated land are appropriate for PSDF.
- To determine whether spent PSDF has potential to be used as an amendment in land reclamation.

3.2 Research Hypotheses

- PSDF will have negative and positive effects on soil, plants and water. High concentrations of potassium and other constituents could be beneficial to plant growth by improving soil nutrient availability and water holding capacity. However high soluble salts may inhibit plant water uptake, and high potassium concentrations may have negative effects on soil hydraulic properties (Chapter 2, 3, 4).

- PSDF may have an inhibitory effect on the soil microbial community. The drilling fluid may control certain microbiological species or may have a negative impact on native microbial populations and components that are of benefit to the soil-plant community (Chapter 5).
- PSDF may have an effect on ground water quality by altering nutrients, salinity, heavy metals and petroleum hydrocarbons (Chapter 4).
- Raw drilling fluids and spent drilling fluids will have different impacts on soil and plants, as the components in raw drilling fluids may be changed under high pressure and temperature during drilling, and spent drilling fluids will incorporate some drilling cuttings (Chapter 3).
- Soils of different textures will respond differently to PSDF application (Chapter 2, 3, 4, 5).
- Plant species will have different response to PSDF (Chapter 2, 3).
- Inorganic nitrogen and phosphate fertilizer will have a potentially crucial impact on plant growth under PSDF application scenarios (Chapter 2).
- PSDF application method, spraying on the soil surface or incorporating into the soil will have impacts on soil, vegetation and water (Chapter 2, 3, 4).
- PSDF application rate will have effects on soil, vegetation and water responses (Chapter 2, 3, 4).

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Table 1.1. Soil endpoint requirements for land spraying drilling wastes.

Pre-Application Conditions	Waste Application Rates or Limits	Post-Application Conditions	Testing Requirements
Receiving topsoil with electrical conductivity < 2 dS m^{-1}, sodium adsorption ratio < 4 Site Slope must be < 5 % in summer operations and < 3 % in winter operations 50 m setback from water well 100 m setback from water body 10 m setback from a road ditch or property line Landowner or Crown department or agency consent for disposal Waste Limited to water based drilling wastes Segregate cement returns and drill stem fluids from drilling waste Separated solids or drill cuttings to be managed by another method	Fluids or total waste Must pass toxicity assessment The pH between 6 and 10.5 Maximum spread rate of 20 m^3 ha^{-1} in winter and 40 m^3 ha^{-1} in summer Maximum solids application rate for vegetated lands 6 t ha^{-1} Minimum spread rate 10 m^3 ha^{-1} If cumulative concentration of metals in mud additives and products exceed values in Table 1.3 or are unknown, analyze drilling waste; use results to calculate spread rates If hydrocarbon flag encountered, analyze drilling waste and use BTEX and F1 to F4 hydrocarbon results to calculate spread rates to prevent exceedances of criteria in Table 1.2	Soil and waste mix limited to maximum increase of one unit beyond background soil electrical conductivity and sodium adsorption ratio, not to exceed electrical conductivity of 2 dS m^{-1} or sodium adsorption ratio of 4 Maximum sodium loading rate 250 kg ha^{-1} Maximum nitrogen loading rate 25 kg ha^{-1} Soil and waste mix must not exceed soil hydrocarbon and metal endpoints in Tables 1.2 and 1.3	Receiving soil electrical conductivity and sodium adsorption ratio Fluids or total waste Electrical conductivity, pH, sodium adsorption ratio and sodium Nitrogen, if an unknown amount or ≥ 100 kg added to drilling mud or waste system Metals, if added to mud system in excess of concentrations in Table 1.3 or unknown Hydrocarbons, if hydrocarbon flag encountered Toxicity assessment Waste and soil mix (post-disposal) Electrical conductivity, sodium adsorption ratio, sodium and nitrogen, if drilling waste exceeds electrical conductivity of 10 dS m^{-1}, nitrogen loading rate exceeds 20 kg ha^{-1}, or if sodium loading rate exceeds 150 kg ha^{-1} Metals, if concentrations in drilling waste exceed concentrations listed in Table 1.3 Hydrocarbons, if concentrations in drilling waste exceed concentrations in Table 1.2

Table summarizes land spray requirements, but details within numbered requirements of Section 9 in Directive 50 will be used for assessing compliance with the land spray disposal method.
BTEX = benzene, toluene, ethylbenzene, xylenes.
Adapted from Alberta Energy Resources Conservation Board (2012).

Table 1.2. Soil hydrocarbon endpoint requirements to manage drilling waste.

	Natural Land		Agricultural Land		Residential or Parkland Land	
Hydrocarbon Fraction	Fine Soil (mg kg^{-1})	Coarse Soil (mg kg^{-1})	Fine Soil (mg kg^{-1})	Coarse Soil (mg kg^{-1})	Fine Soil (mg kg^{-1})	Coarse Soil (mg kg^{-1})
F1 (C_6 - C_{10}, no BTEX)	210	210	210	24	210	24
F2 (> C_{10} - C_{16})	150	150	150	130	150	130
F3 (> C_{16} - C_{34})	1300	300	1300	300	1300	300
F4 (> C_{34})	5600	2800	5600	2800	5600	2800
Benzene	0.046	0.078	0.046	0.073	0.046	0.073
Toluene	0.520	0.490	0.520	0.490	0.520	0.490
Ethylbenzene	0.110	0.210	0.110	0.210	0.110	0.210
Xylenes	15	28	15	12	15	12

BTEX = benzene, toluene, ethylbenzene, xylenes.
Adapted from Alberta Energy Resources Conservation Board (2012).

Table 1.3. Soil metal endpoint requirements to manage drilling waste.

	Guideline Value (mg kg^{-1})		
	Agricultural Land	Natural Land	Residential or Parkland Land
Antimony	20.0	20.0	20.0
Arsenic (inorganic)	17.0	17.0	17.0
Barium	750.0	750.0	500.0
Barite - barium†	10000.0	10000.0	10000.0
Beryllium	5.0	5.0	5.0
Boron (hot water soluble)	2.0	2.0	2.0
Cadmium	1.4	3.8	10.0
Chromium (total)	64.0	64.0	64.0
Cobalt	20.0	20.0	20.0
Copper	63.0	63.0	63.0
Lead	70.0	70.0	140.0
Mercury (inorganic)	6.6	12.0	6.6
Molybdenum	4.0	4.0	4.0
Nickel	50.0	50.0	50.0
Selenium	1.0	1.0	1.0
Silver	20.0	20.0	20.0
Thallium	1.0	1.0	1.0
Tin	5.0	5.0	5.0
Uranium	23.0	33.0	23.0
Vanadium	130.0	130.0	130.0
Zinc	200.0	200.0	200.0

† Alberta Soil Remediation Guidelines for Barite (2009) must be followed to determine if the site qualifies as a barite site. If it does not, then the 750 mg kg^{-1} total barium value applies.

Adapted from Alberta Energy Resources Conservation Board (2012).

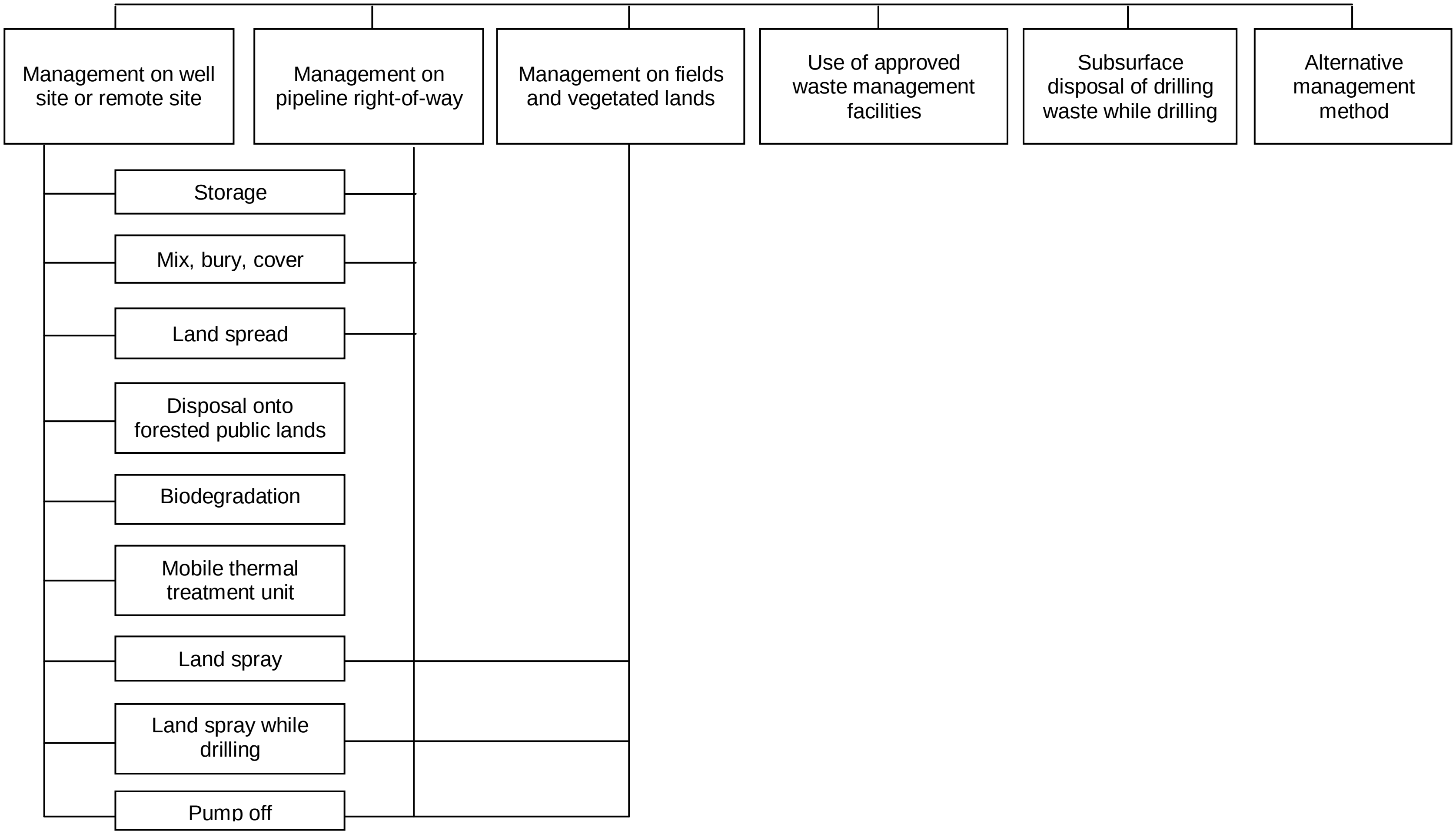

Figure 1.1. Overview of drilling waste disposal methods. Adapted from Alberta Energy Resources Conservation Board (2012).

CHAPTER 2. SOIL AND PLANT RESPONSE TO RAW POTASSIUM SILICATE DRILLING FLUID APPLICATION IN A GREENHOUSE

1. Introduction

The path to economic and environmental sustainability requires a new way of thinking about oil and gas industry waste streams. Drilling fluid, also referred to as drilling mud, is one of the primary drilling wastes generated. It is used to lubricate and cool the drilling apparatus, transport drill cuttings to the surface and seal off porous geologic formations. Drilling fluids typically consist of bentonite and various additives mixed with fresh water or hydrocarbons, and are classified as water based, oil based and synthetic based. Disposal of drilling fluid is a growing concern to the global oil and gas industry as it can require extensive waste management and result in significant land disturbance. Land application has been suggested as a possible disposal method, but before guidelines can be developed and modified it is necessary to determine environmental effects of drilling fluid on soils, plants and ground water.

Disposal of drilling muds varies with type and jurisdiction. For example, in western Canada, land spraying while drilling is a common disposal approach with criteria for maximum disposal rate and acceptable increases in electrical conductivity, sodium absorption ratio and sodium and nitrogen loading for water based drilling waste (Alberta Energy Resources Conservation Board 2012, Saskatchewan Ministry of Energy and Resources 2011, British Columbia Oil and Gas Commission 2006). Spent drilling fluid could be disposed and incorporated on cultivated land or spread on vegetated land without incorporation. Before 2012, advanced gel chemical muds (e.g. potassium silicate, potassium sulphate), water based drilling mud systems, were not included in Alberta regulations for drilling waste land spraying, and required site specific approval for disposal.

Few studies have addressed the impact of disposal of spent drilling fluids on soil-plant-water systems and results are variable. Some researchers found high soluble salts, heavy metals and petroleum residue in used drilling fluids were detrimental to soil quality and plant growth (Zvomuya et al. 2009, Wojtanowicz 2008, Zvomuya et al. 2008, McFarland et al. 1994, McFarland et al. 1992, Nelson et al. 1984, Miller et al. 1980). Others found positive or no impacts from drilling fluids applied at low rates in coarse textured soils in arid regions (Bauder et al. 2005, Bauder et al. 1999, Macyk et al. 1990, Lesky et al. 1989, Tucker 1985, Moseley Jr. 1983, Miller et al. 1980). Differences among studies primarily resulted from waste properties, disposal method and disposal location.

Previous studies (Bauder et al. 2005, Bauder et al. 1999, Macyk et al. 1990, Miller et al. 1980) have focused on agricultural or horticultural crops, such as green beans (*Phaseolus vulgaris* L.), sweet corn (*Zea mays* L.), sorghum (*Sorghum bicolour* L. Moench) and winter wheat (*Triticum aestivum* L.), or forage species such as smooth brome grass (*Bromus inermis* Leyss.). Response of these plants to drilling fluid application varied with species and drilling fluids.

Fertilizer plays an important role in land reclamation, often dramatically improving ecosystem restoration (Marrs and Bradshaw 1982). Drilling fluid normally has high pH and sodicity which can cause nutrient deficiencies by modifying availability of elements that play a major role in plant nutrition, such as phosphorus, potassium and magnesium (Taiz and Zeiger 1998). Thus investigating fertilizer use under drilling fluid application scenarios is important.

Potassium silicate drilling fluids (PSDF) are relatively new water based mud systems developed to reduce environmental impacts. Sodium concentrations in traditional drilling fluids were considered to have the most detrimental environmental impact; thus replacing sodium with potassium in newer drilling fluids would reduce concentrations of sodium. Hypothetically, high concentrations of potassium in PSDF could serve as a nutrient amendment for land reclamation, thus not only would land disposal be practical, but also provide benefits for reclamation. However, PSDF properties and effects on vegetation and soil are not well known. Such environmental impacts must be determined before land disposal of PSDF and its use in reclamation could be regulated.

The objective of this greenhouse experiment was to evaluate whether application of raw PSDF to three different textured soils, at three different rates and with two different application methods would affect soil properties and establishment and development of selected agricultural and native grass species. By using raw drilling fluid, the influence of the soils being drilled could be removed, and the impact of the drilling fluid itself could be more clearly interpreted.

2. Materials And Methods

2.1 Experimental Design

A greenhouse experiment was conducted in a completely randomized experimental design, with treatments replicated 5 times. Treatments represented application scenarios under various end land uses. Four soil textures were sand, loam, clay loam 1 and clay loam 2 (more clay than clay loam 1), covering a range of soils with potential for potassium silicate drilling fluid disposal. Two application methods were spraying over the soil and spraying followed by incorporation, approximating likely application methods on a field basis. Three potassium silicate drilling fluid application rates were developed around the current Energy Resources Conservation Board (2007) summer maximum loading rate of 40 m^3 ha^{-1}; they were 30, 45 and 60 m^3 ha^{-1}.

Plant species used were slender wheat grass (*Agropyron trachycaulum* (Link) Malte ex H.F. Lewis) and common barley (*Hordeum vulgare* L.), representing a common native species and a common agricultural crop widely used in reclamation. These two species are relatively alkali and salinity tolerant (Wentz 2001, McKenzie 1988) and have been successfully used in a wide range of land reclamation scenarios.

Two fertilizer treatments were with and without fertilizer. Amount of fertilizer was based on optimum concentrations of macronutrients required by agronomic species. Potassium fertilizer was not applied due to the high potassium content in PSDF. Fertilizer was ammonium nitrate (NH_4NO_3) at 0.06 g pot^{-1} and triple super phosphate ($3Ca_3(PO_4)_2{\cdot}CaF_2 + 4H_3PO_4 + 9H_2O \dashrightarrow 9Ca(H_2PO_4)_2 + CaF_2$) at 0.07 g pot^{-1}, equivalent to 34 and 45 kg ha^{-1}, respectively.

2.2 Potassium Silicate Drilling Fluid, Soil Collection and Analyses

Raw PSDF was manufactured by Marquis Alliance Ltd. and refrigerated until used. Soils were collected from three Alberta locations where drilling was active. Soil and PSDF properties were determined by Exova Laboratory Group. One sample was randomly procured from each treatment (56 samples), except the no fertilizer treatment due to budget limitations.

Soil pH, sodium adsorption ratio and electrical conductivity were determined from saturated paste extracts (Carter and Gregorich 2008). Routine ions were determined by ion chromatography with chemical suppression (Clesceri et al. 1992). Cation exchange capacity was determined by exchange with ammonium acetate (NH_4OA_C) at pH 7 (McKeague 1978). Available nitrate (NO_3^-) and ammonium (NH_4^+) were determined by extraction with 2.0 M potassium chloride (KCl) (Carter and Gregorich 2008). Available phosphorus and potassium were determined by modified Kelowna extraction (Ashworth and Mrazek 1995) and available sulphate by extraction with 0.1 M calcium chloride ($CaCl_2$) (McKeague 1978). Sand, silt and clay were determined by hydrometer after treatment with calgon (Carter and Gregorich 2008). Total nitrogen was determined by Kjeldahl digestion distillation (Bremner 1996). Total carbon was determined by dry combustion (Nelson and Sommers 1996) and total organic carbon by the Walkley-Black wet dichromate oxidation method (Nelson and Sommers 1996).

2.3 Greenhouse Methods and Plant Measurements

Soil was placed in 15 cm diameter pots. PSDF was applied at rates of 30, 45 and 60 m^3 ha^{-1} (34.11, 51.17 and 68.22 Mg ha^{-1}). Untreated controls were included for each soil. Land spraying treatments of PSDF were hand applied evenly on the soil surface from glass beakers. PSDF was incorporated into the soil with small shovels for incorporated treatments. Pots were left to settle for 1 week before seedling to represent a field scenario.

Pots were seeded with 20 wheat grass or 10 barley seeds, then randomly placed on a greenhouse bench. Seeding rates were based on species morphology and length of time of the experiment. Pots were watered regularly twice a day for the first 4 weeks, then once a day thereafter. Sufficient water was added to keep the soil at field capacity by gravimetric methods throughout the growth period (weight basis of pots). Greenhouse temperature was maintained at 21 ^oC during the day and 15 ^oC at night, with a 16 hour photoperiod.

Number of plants established and their survival in each pot were recorded weekly. At the end of the four month greenhouse session, seeded plant density, height of the tallest and shortest plants, length and width of the widest and narrowest leaves and number of seed heads were determined in each pot. Above ground biomass from each pot was clipped at ground level. Roots were removed from the substrate by hand and lightly brushed to remove residual soil. All biomass was oven dried at 80 ^oC for 48 hours to constant weight.

2.4 Statistical Analyses

Analyses were conducted with R software (R Development Core Team 2012). Effects of soil, fertilizer, plant species, PSDF application method and rate were considered fixed. Plant establishment and development were analyzed with five way factorial analysis of variance (ANOVA). Plant density and above ground biomass variances were heterogeneous among species and thus were analyzed as survival percentage (density divided by number of seeds) and above ground biomass per plant (above ground biomass divided by density). Data were checked for violations of normality assumption with Shapiro-Wilk test and equal variances assumption with Bartlett's test. Soil chemical data were compared using means and standard errors. With no replication of soil samples within treatments due to budget limitations, five way ANOVA without interaction was completed rather than five way factorial ANOVA. Where significant treatment effects were determined, an $LSD_{0.05}$ was calculated to provide a method of comparing means and measures of precision. Only when the F test was significant were statistical differences among means determined. To investigate soil chemical property responses to PSDF application, principal components analysis (PCA) was performed.

3. Results

3.1 Soil Response to PSDF

Raw PSDF components and other properties are presented in Tables 2.1, 2.2, 2.3. PSDF had a density of 1137 kg m^{-3}, equivalent to a dry bulk density of 220 kg m^{-3}, as determined by the manufacturer. Most notably, raw PSDF had high pH and electrical conductivity and did not contain hydrocarbons.

Soil variables work together to create a dynamic system that has significant impacts on plant growth and development. Principal component analysis can be used to assist in revealing soil response to PSDF application with fertilizer for the different plant species studied (Figure 2.1). Axis 1 explained 44.4 % of the response to PSDF and fertilizer, while axis 2 explained 19.6 %; the cumulative proportion explained 64 %.

Electrical conductivity, pH, sodium adsorption ratio and cation exchange capacity varied with soil and PSDF (Table 2.4). Loam soil had significantly lowest pH, clay loam 2 had highest electrical conductivity, and sand had highest sodium adsorption ratio and lowest cation exchange capacity. This led to groupings of highest clay content soils with greater electrical conductivity, and highest sand content soils with highest sodium adsorption ratio and lowest electrical conductivity (Figure 2.2 A, B). Electrical conductivity and pH did not respond significantly to PSDF application method or rate (Table 2.4), although sodium adsorption ratio increased significantly with increased PSDF (Figure 2.2 B). Sodium adsorption ratio and cation exchange capacity were significantly greater in soil with barley than wheat grass (Table 2.4).

Available nitrogen, phosphorus, potassium and sulphur were significantly different among soils after PSDF application (Table 2.5). Available nitrogen, phosphorus and potassium were highest in clay loam 1 soil (Table 2.5, Figure 2.1 B). Clay loam 2 soil had significantly highest available sulphate, 30 times higher than that of sandy soil (Table 2.2). PSDF application method and rate and plant species had no significant effect on available nitrogen and sulphur (Table 2.5, Figure 2.1 A, C, D). Available potassium increased significantly with increasing PSDF rate of application (Table 2.5), being highest at the highest PSDF rate in clay loam 1 soils (Figure 2.2 C).

Soils had significantly different soluble sodium, calcium, magnesium, potassium, sulphate and chloride following PSDF application (Table 2.6). All soluble cations and anions except potassium were significantly greatest in clay loam 2 soils; calcium sulphate was the dominant soluble salt with concentrations at least 4.5 times higher than that in the other soils (Table 2.6). Soil with different plant species or PSDF application methods had no significant effect on soluble salts (Figure 2.1 A, C). PSDF rate significantly affected soluble sodium and potassium; potassium concentrations increased rapidly with increased application rates of PSDF (Table 2.6, Figure 2.2 D).

3.2 Plant Response to PSDF

Plant survival was significantly affected by soil and its interactions with PSDF application method and by plant species and its interaction with fertilizer (Table 2.7). Plant measurements with each main factor level are shown in Table 2.8. There was a significant interaction between soil and PSDF application method on plant survival; clay loam 1 and loam soils with incorporated PSDF had significantly greater plant survival than with sprayed PSDF (Figure 2.3). Plant survival was lowest with sprayed PSDF on clay loam 1 and greatest on loam soil with incorporated PSDF. Fertilizer and plant species had a significant interaction on plant survival (Table 2.7). Wheat grass survival was significantly higher with fertilizer, while barley survival was lowest in the same treatment (Figure 2.4). There were no significant main effects of PSDF application rate on plant survival (Table 2.7). Survival was not significantly different with or without PSDF, except it decreased significantly in clay loam 2 with PSDF (Figure 2.5 A).

Soil, plant species, PSDF application method and rate and their respective interactions significantly affected plant height; fertilizer did not (Table 2.7). There was a significant interaction among species, PSDF application method and rate. Barley height with PSDF was significantly greater than that of wheat grass and PSDF at 30 m^3 ha^{-1} yielded tallest barley plants (Figure 2.6). There were significant interactions among soil type, PSDF application method and rates (Table 2.7). Clay loam 1 soil sprayed with PSDF at 30 m^3 ha^{-1} yielded tallest plants (data not shown). Plant height was not significantly affected by PSDF application method at 30 and 45 m^3 ha^{-1}, but sprayed PSDF at 60 m^3 ha^{-1} yielded significantly taller plants than incorporated PSDF (Figure 2.7). Relative to no PSDF, highest application rates of PSDF significantly decreased plant height in clay loam 2, but increased it in loam soil (Figure 2.5 B).

Soil, fertilizer, PSDF application method and rate significantly affected above ground biomass (Table 2.7). There was a significant interaction among soil type, PSDF application method and species. Greatest barley biomass occurred with sprayed PSDF at 45 m^3 ha^{-1}; significantly greatest on clay loam 1 (Figure 2.8). Fertilizer significantly increased biomass, with that of barley significantly greater than wheat grass (Figure 2.9). Biomass was significantly greater with PSDF than without, except at 60 m^3 ha^{-1} in clay loam 2 soil (Figure 2.5 C).

4. Discussion

There was no evidence of detrimental effects of raw PSDF on soil and vegetation. Application of PSDF to soils benefitted plants, as evidenced by increased height and above ground biomass.

4.1 Soil Responses to PSDF

Significantly increased available and soluble potassium in soil with PSDF have implications for plant growth and development. Plants cannot grow or survive without potassium (Mengel 2007). It plays an important role in plant enzyme activation, charge balance and osmoregulation, and helps plants exclude sodium (Taiz and Zeiger 1998). Soluble potassium plays a critical role in salt tolerance in barley, which is highly correlated with physiology (Chen et al. 2007).

Soil potassium occurs as mineral, fixed, exchangeable and solution, with only exchangeable and soluble potassium readily available to plants (Carey et al. 2011). Most potassium in PSDF was immediately available to plants, a desirable situation for land reclamation. Clay loam and loam soil with 24 to 38 % clay could retain exchangeable potassium at much higher concentrations than sandy soils with 2 % clay, since clay content affects exchangeable potassium with clay minerals containing fixed potassium and releasing it when soil solution potassium concentration is low (Ogaard et al. 2002, Sharpley and Buol 1987, Ma and Hanway 1969). Loam soil had the lowest pH before and after PSDF application, further impacting potassium availability as it is strongly affected by pH, with neutral and slightly acidic soils having more available potassium to plants (Arienzo et al. 2009).

PSDF slightly increased sodium adsorption ratio but did not increase pH and electrical conductivity. This increased sodium adsorption ratio was likely due to soluble sodium and potassium in PSDF, which was not high enough to severely impact plants. When saline PSDF is applied to soil, competitive adsorption of cations occurs on soil exchange sites and ions of highest valence, such as calcium and magnesium, are preferentially adsorbed. Increased solution concentration in soil tends to enhance adsorption of monovalents, such as sodium and potassium (Smiles 2006).

Responses of soils to drilling fluid application were not always consistent with those reported in other studies. Bauder et al. (2005) found increasing pH, electrical conductivity and sodium adsorption ratio with increasing drilling fluid rates, but not at levels detrimental to winter wheat (*Triticum aestivum* L.) production in the field. Application rates were 94 Mg ha^{-1}, higher than the maximum rate of 60 m^3 ha^{-1} (68.22 Mg ha^{-1}) in our study. Macyk et al. (1990) found significant increases in chloride and sodium, potentially limiting to brome grass in a greenhouse. Although fresh PSDF had high pH, electrical conductivity and sodium adsorption ratio, all our soils with PSDF ≤ 60 m^3 ha^{-1} were classified as good quality for revegetation relative to disturbance and reclamation for unrestricted land use except clay loam 2 (electrical conductivity < 2, sodium adsorption ratio < 4) (Alberta Environmental Sciences Division 2001). Electrical conductivity in clay loam 2 soil before PSDF application led to low quality classification. Kim et al. (2002) utilized calcium salts to decrease sodium adsorption ratio and lower chromium mobility. Highest calcium in clay loam 2 with poor plant response, suggests calcium salts may improve soil quality by decreasing sodicity, but baseline soil values must be considered.

4.2 Plant Responses to PSDF

Effect of PSDF on plants depends on plant species, fertilizer, soil type, PSDF rate and application method. These five factors showed strong interactions with plant establishment and growth. Wheat grass survived better than barley with PSDF, but barley had greater height and biomass. Either of these species is compatible with PSDF use.

Soil to which PSDF is applied will significantly affect vegetation. Loam soil with PSDF had significantly higher plant survival than other soils, likely due to more available potassium and lower pH. Higher available nutrients (nitrogen, phosphorus, potassium) in clay loam 1 soil contributed to greatest plant height and biomass. Moseley Jr. (1983) also found high pH drilling fluids containing sodium salts caused the least detrimental effects when added to acidic, medium texture soils (silt, clay loams) in areas with over 63.5 cm annual precipitation.

Plant response to spraying and incorporating PSDF gave similar results, unlike results of Zvomuya et al. (2009), who found water based drilling fluid sprayed at the surface could impede near saturated hydraulic conductivity, due to clogging of conductive macrospores in the receiving soil. Water that fails to infiltrate and remains on the soil surface is subject to runoff or evaporation losses, and therefore not available for plant uptake or soil storage. Seeding in our study, after a low rate of drilling fluid was applied then left to settle for 1 week, led to cracked PSDF on the soil surface which may have led to less reduction of water infiltration rate. Zvomuya et al. (2011) reported land spraying spent water based drilling fluid at 40 and 80 m^3 ha^{-1} did not adversely affect nutrient uptake and plant growth. Since hydrologic effects did not persist one year following drilling mud application or high rainfall after application washed the vegetation before it could impart any adverse effects on the vegetation. Thus PSDF effects on hydrologic properties are also less likely to be impacted long term.

PSDF application at ≤ 45 m^3 ha^{-1} in loam soil, was associated with the most significant positive effects on vegetation. Lower rates could also enhance plant growth with greatest height and biomass from pH increases in acidic soils, potential micronutrient (zinc, iron) addition or improved soil physical properties as determined in other similar studies (Bauder et al. 2005, Bauder et al. 1999, Macyk et al. 1990). A 12 month field study found no negative effect on agriculture productivity with water based drilling fluid up to 80 m^3 ha^{-1} in Saskatchewan, Canada (Lesky et al. 1989). Zvomuya et al. (2011) suggested a single water based drilling fluid spraying at ≤ 20 m^3 ha^{-1} is unlikely to adversely affect native prairie vegetation in western Canada. Miller et al. (1980) found low amounts of organic materials, such as starches, gum and tannin in drilling fluids stimulated plant growth, although this was not assessed in our study.

5. Conclusions

Raw PSDF had no detrimental effects on soil and plants at rates ≤ 45 m^3 ha^{-1}. Available potassium for plants increased significantly with PSDF application. Barley and wheat grass plants survived and developed through their physiological growth stages in different textured soils with spread and incorporated PSDF. Inorganic nitrogen and phosphorus fertilizer and potassium fertilizer from PSDF had a significant effect on vegetation. Raw PSDF as a land reclamation amendment will likely give best results on acidic and well drained soils with inorganic fertilizer.

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Table 2.1. Raw PSDF components.

Freshwater (m^3)	1
Pregelatinized starch (kg m^{-3})	6
Polyanionic cellulose (kg m^{-3})	2
Anionic water soluble polymer (kg m^{-3})	1
Xanthan gum (kg m^{-3})	2
Potassium hydroxide (kg m^{-3})	3
Raw silicate (kg m^{-3})	5.5

Source: Marquis Alliance Ltd. (Ma 2008)

Table 2.2. Selected raw PSDF chemical properties.

Hydrogen ion activity (pH)	11
Electrical conductivity (dS m^{-1})	14
Sodium adsorption ratio	43
Soluble calcium (mg L^{-1})	93
Soluble magnesium (mg L^{-1})	20
Soluble sodium (mg L^{-1})	290
Soluble potassium (mg L^{-1})	6860
Soluble chloride (mg L^{-1})	104
Soluble sulphate (mg L^{-1})	97
Total petroleum hydrocarbons	0

Table 2.3. Selected soil properties before PSDF application.

	Sand	Loam	Clay Loam 1	Clay Loam 2
Available nitrogen (mg kg^{-1})	1.5	36.5	85.0	29.5
Available phosphorus (mg kg^{-1})	15.5	< 5.0	56.5	21.5
Available potassium (mg kg^{-1})	32.5	120.0	572.5	392.5
Available sulphur (mg kg^{-1})	1.5	11.0	18.8	1019.5
Cation exchange capacity (meq 100 g^{-1})	4.0	21.5	22.0	28.5
Total carbon (%)	0.2	2.3	1.8	2.4
Total organic carbon (%)	0.2	2.3	1.7	2.1
Total nitrogen (%)	< 0.02	0.2	0.1	0.2
Hydrogen ion concentration (pH)	6.2	5.9	7.5	7.1
Electrical conductivity (dS m^{-1})	0.1	0.7	1.7	3.2
Sodium adsorption ratio	0.2	0.3	0.2	0.6
Soluble sodium (mg kg^{-1})	0.8	6.0	8.0	46.0
Soluble calcium (mg kg^{-1})	1.6	50.7	132.0	428.8
Soluble magnesium (mg kg^{-1})	0.5	10.1	23.5	120.0
Soluble potassium (mg kg^{-1})	1.5	1.3	20.3	25.0
Soluble chloride (mg kg^{-1})	1.3	7.3	4.3	16.0
Soluble sulphate (mg kg^{-1})	1.0	8.5	15.1	400.0
Sand (%)	95.9	30.0	30.5	21.5
Silt (%)	1.6	46.0	38.8	40.2
Clay (%)	2.5	24.0	30.8	38.3

Table 2.4. Electrical conductivity, pH, sodium adsorption ratio and cation exchange capacity in soil with fertilizer and PSDF.

		Hydrogen Ion Activity (pH)	Electrical Conductivity (dS m^{-1})	Sodium Adsorption Ratio	Cation Exchange Capacity (meq $100g^{-1}$)
Soil	Sand	7.6 ± 0.0	0.8 ± 0.1	1.1 ± 0.1	4.3 ± 0.3
	Loam	6.3 ± 0.0	0.8 ± 0.0	0.8 ± 0.0	33.8 ± 3.3
	Clay Loam 1	7.8 ± 0.0	1.1 ± 0.1	0.7 ± 0.0	36.4 ± 3.6
	Clay Loam 2	7.7 ± 0.0	2.9 ± 0.0	0.7 ± 0.0	25.8 ± 0.3
Application Method	Incorporated	7.3 ± 0.1	1.4 ± 0.2	0.8 ± 0.1	25.8 ± 3.2
	Sprayed	7.3 ± 0.1	1.4 ± 0.2	0.8 ± 0.0	24.4 ± 3.0
Rate (m^3 ha^{-1})	0	7.2 ± 0.2	1.3 ± 0.2	0.6 ± 0.0	22.5 ± 3.2
	30	7.3 ± 0.2	1.4 ± 0.2	0.8 ± 0.1	24.2 ± 3.7
	45	7.4 ± 0.2	1.4 ± 0.2	0.8 ± 0.1	25.6 ± 4.1
	60	7.4 ± 0.2	1.5 ± 0.2	0.9 ± 0.1	25.4 ± 3.9
Plant Species	Barley	7.3 ± 0.1	1.4 ± 0.2	0.9 ± 0.0	30.3 ± 3.8
	Wheat grass	7.4 ± 0.1	1.4 ± 0.2	0.8 ± 0.0	19.9 ± 1.8

Means within a column, within treatments of the same variable, followed by standard errors.

Table 2.5. Available nitrogen, phosphorus, potassium and sulphur in soil with fertilizer and PSDF.

		Nitrogen (mg kg^{-1})	Phosphorus (mg kg^{-1})	Potassium (mg kg^{-1})	Sulphur (mg kg^{-1})
Soil	Sand	2.1 ± 0.8	61.5 ± 2.7	183.3 ± 16.9	23.5 ± 2.8
	Loam	11.8 ± 2.1	35.3 ± 1.7	238.3 ± 15.3	53.0 ± 4.2
	Clay Loam 1	11.9 ± 3.2	108.3 ± 4.4	700.0 ± 40.8	58.6 ± 5.0
	Clay Loam 2	5.8 ± 2.6	43.6 ± 3.2	446.7 ± 18.1	678.3 ± 66.3
Application Method	Incorporated	9.0 ± 2.0	59.5 ± 5.6	388.3 ± 46.7	176.1 ± 48.7
	Sprayed	6.7 ± 1.6	64.9 ± 6.9	395.8 ± 44.8	230.6 ± 71.9
Rate (m^3 ha^{-1})	0	13.4 ± 4.3	59.3 ± 7.1	217.5 ± 38.6	241.4 ± 91.7
	30	7.5 ± 2.2	61.2 ± 7.6	323.8 ± 47.3	215.3 ± 85.3
	45	5.9 ± 2.0	62.3 ± 8.3	385.6 ± 54.3	192.2 ± 66.8
	60	10.2 ± 2.4	63.0 ± 7.5	466.9 ± 61.3	202.6 ± 75.9
Plant Species	Barley	7.6 ± 1.7	58.9 ± 6.6	375.8 ± 41.6	185.0 ± 50.6
	Wheat grass	8.2 ± 2.0	65.4 ± 5.9	408.3 ± 49.3	221.7 ± 70.9

Means within a column, within treatments of the same variable, followed by standard errors.

Table 2.6. Soluble elements in soil with fertilizer and PSDF.

		Sodium (mg kg^{-1})	Calcium (mg kg^{-1})	Magnesium (mg kg^{-1})	Potassium (mg kg^{-1})	Chloride (mg kg^{-1})	Sulphate (mg kg^{-1})	Nitrate (mg kg^{-1})
Soil	Sand	11.0 ± 1.1	16.6 ± 1.1	5.3 ± 0.4	28.4 ± 4.2	3.1 ± 0.1	20.9 ± 2.2	2.6 ± 0.9
	Loam	18.2 ± 1.0	56.1 ± 2.6	12.2 ± 0.6	4.9 ± 0.5	6.6 ± 0.5	49.0 ± 3.3	6.6 ± 2.1
	Clay Loam 1	20.1 ± 1.2	77.5 ± 5.2	14.1 ± 0.9	34.2 ± 2.8	7.1 ± 0.5	56.7 ± 4.9	10.4 ± 3.3
	Clay Loam 2	41.8 ± 2.4	340.5 ± 6.4	78.9 ± 2.5	28.6 ± 2.2	8.5 ± 1.2	362.3 ± 7.5	7.0 ± 1.9
Application Method	Incorporated	22.9 ± 2.6	121.6 ± 26.3	27.5 ± 6.2	24.0 ± 3.3	6.8 ± 0.7	119.3 ± 28.5	8.0 ± 1.9
	Sprayed	22.6 ± 2.6	123.8 ± 27.2	27.8 ± 6.4	24.1 ± 2.7	5.9 ± 0.5	125.1 ± 29.9	5.3 ± 1.3
Rate (m^3 ha^{-1})	0	15.9 ± 2.3	126.6 ± 33.0	29.5 ± 8.2	8.9 ± 1.8	5.4 ± 0.6	113.7 ± 36.1	12.6 ± 4.5
	30	20.9 ± 3.2	122.3 ± 33.5	27.5 ± 7.9	17.4 ± 2.4	5.9 ± 0.8	120.5 ± 36.6	5.8 ± 2.1
	45	22.3 ± 3.0	121.1 ± 32.6	27.4 ± 7.7	23.2 ± 3.0	6.1 ± 0.6	122.3 ± 36.1	5.7 ± 1.7
	60	25.1 ± 3.3	124.7 ± 33.4	27.9 ± 7.8	31.4 ± 4.6	6.9 ± 1.0	123.8 ± 36.0	8.5 ± 2.2
Plant Species	Barley	24.8 ± 2.8	122.1 ± 25.9	28.8 ± 6.4	24.6 ± 3.3	6.2 ± 0.6	123.3 ± 28.3	5.8 ± 1.4
	Wheat grass	20.7 ± 2.4	123.2 ± 27.6	26.4 ± 6.1	23.5 ± 2.7	6.4 ± 0.7	121.1 ± 30.2	7.5 ± 1.9

Means within a column within treatments of same variable followed by standard errors.

Table 2.7. P values of five factor ANOVA (soil, PSDF application method and rate, fertilizer, plant species) for plant survival, height and biomass in soils with fertilizer and PSDF after 4 months.

	Survival (%)	Height (cm)	Biomass (g plant^{-1})
Soil (S)	< 0.001	< 0.001	< 0.001
Application Method (M)	0.060	0.175	0.031
Rate (R, m^3 ha^{-1})	0.575	0.010	0.666
Fertilizer (F)	0.941	0.203	0.038
Plant Species (P)	< 0.001	< 0.001	< 0.001
S × M	< 0.001	0.197	0.001
S × F	0.169	0.388	0.359
M × F	0.988	0.210	0.716
S × P	0.085	0.001	< 0.001
M × P	0.335	0.729	0.042
F × P	0.030	0.546	0.038
S × R	0.928	0.032	0.685
M × R	0.502	< 0.001	0.021
F × R	0.283	0.291	0.304
P × R	0.295	0.145	0.668
S × M × F	0.949	0.542	0.830
S × M × P	0.687	0.694	0.026
S × F × P	0.459	0.212	0.457
M × F × P	0.119	0.826	0.859
S × M × R	0.207	0.044	0.368
S × F × R	0.995	0.536	0.959
M × R × F	0.839	0.271	0.832
S × R × P	0.524	0.184	0.871
M × R × P	0.505	0.005	0.012
R × F × P	0.193	0.513	0.506
S × M × F × P	0.417	0.379	0.910
S × M × F × R	0.181	0.527	0.795
S × M × P × R	0.379	0.321	0.358
S × M × R × P	0.097	0.178	0.832
M × R × F × P	0.468	0.321	0.656
S × M × R × F × P	0.975	0.391	0.898

Table 2.8. Plant growth in soils with fertilizer and PSDF after 4 months.

		Survival (%)	Height (cm)	Biomass (g $plant^{-1}$)
Soil	Sand	42.3 ± 1.7	38.3 ± 1.2	0.8 ± 0.1
	Loam	48.8 ± 1.7	36.4 ± 1.1	0.7 ± 0.1
	Clay Loam 1	40.5 ± 1.6	43.4 ± 1.2	1.6 ± 0.2
	Clay Loam 2	42.3 ± 1.6	35.3 ± 1.3	0.7 ± 0.1
Application Method	Incorporated	44.8 ± 1.2	38.4 ± 0.9	0.9 ± 0.1
	Sprayed	42.1 ± 1.2	39.4 ± 0.9	1.0 ± 0.1
Rate (m^3 ha^{-1})	0	44.9 ± 1.4	36.9 ± 1.1	0.6 ± 0.1
	30	43.1 ± 1.5	39.2 ± 1.1	0.9 ± 0.1
	45	44.5 ± 1.4	40.0 ± 0.9	1.0 ± 0.1
	60	42.8 ± 1.6	37.4 ± 1.2	0.9 ± 0.1
Fertilizer	With	43.4 ± 1.2	39.3 ± 0.9	1.0 ± 0.1
	Without	43.5 ± 1.2	38.4 ± 0.9	0.9 ± 0.1
Plant Species	Barley	33.7 ± 1.1	48.9 ± 0.8	1.6 ± 0.1
	Wheat grass	53.3 ± 0.9	28.8 ± 0.3	0.3 ± 0.0

Biomass = above ground biomass.

Means within a column, within treatments of the same variable, followed by standard errors.

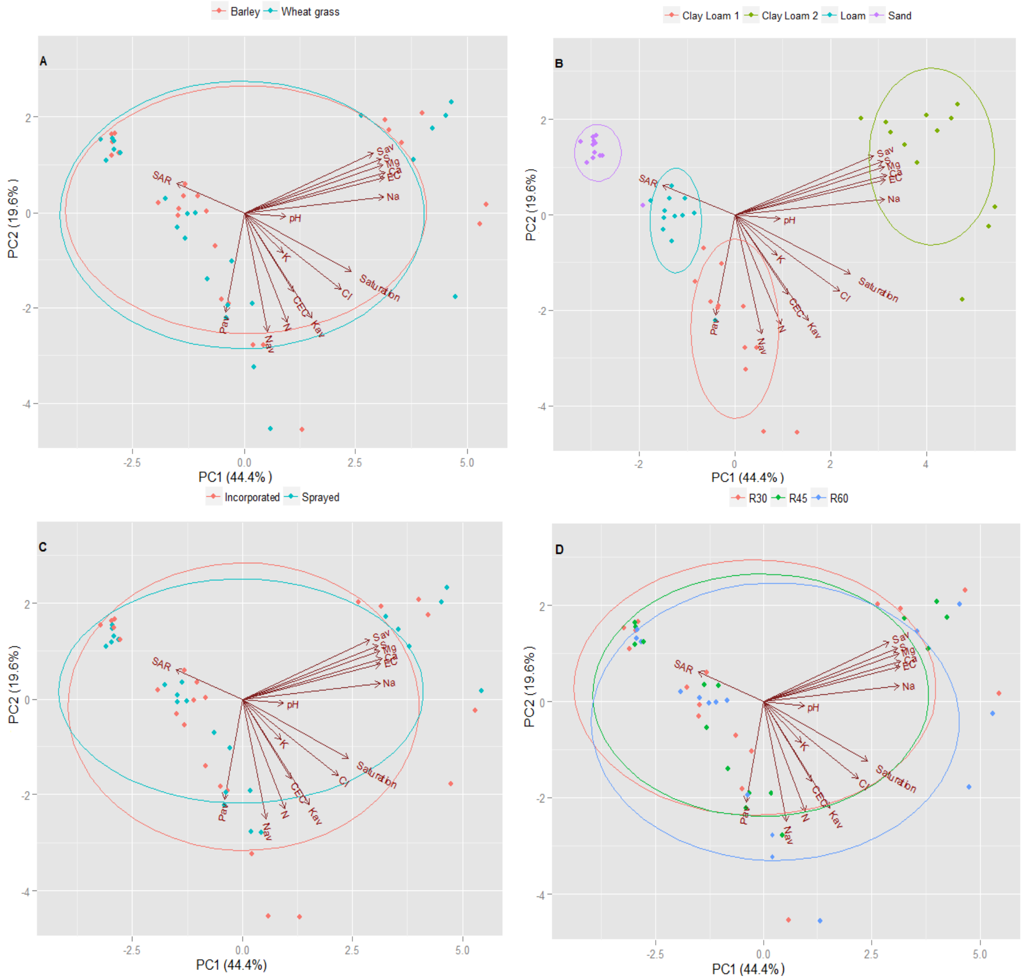

Figure 2.1. Principal component analysis. Score plot of PC1 vs PC2 illustrates distribution of soil chemical properties. Descriptions at the end of arrows represent soil chemical measurements (salinity, nutrients, and soluble ions). Ellipses represent treatments clustered by the same level of each main effect: plant species (A), soil types (B), PSDF application methods (C), PSDF application rates (D). R30, R45, R60 = PSDF at 30, 45, 60 m^3 ha^{-1}. Nav = available nitrogen. Pav = available phosphorus. Kav = available potassium. Sav = available sulphur, pH = hydrogen ion activity, EC = electrical conductivity, SAR = sodium adsorption ratio, CEC = cation exchange capacity, K = soluble potassium, Na = soluble sodium, Mg = soluble magnesium, Ca = soluble calcium, Cl = soluble chloride, S = soluble sulphate, N = soluble nitrate.

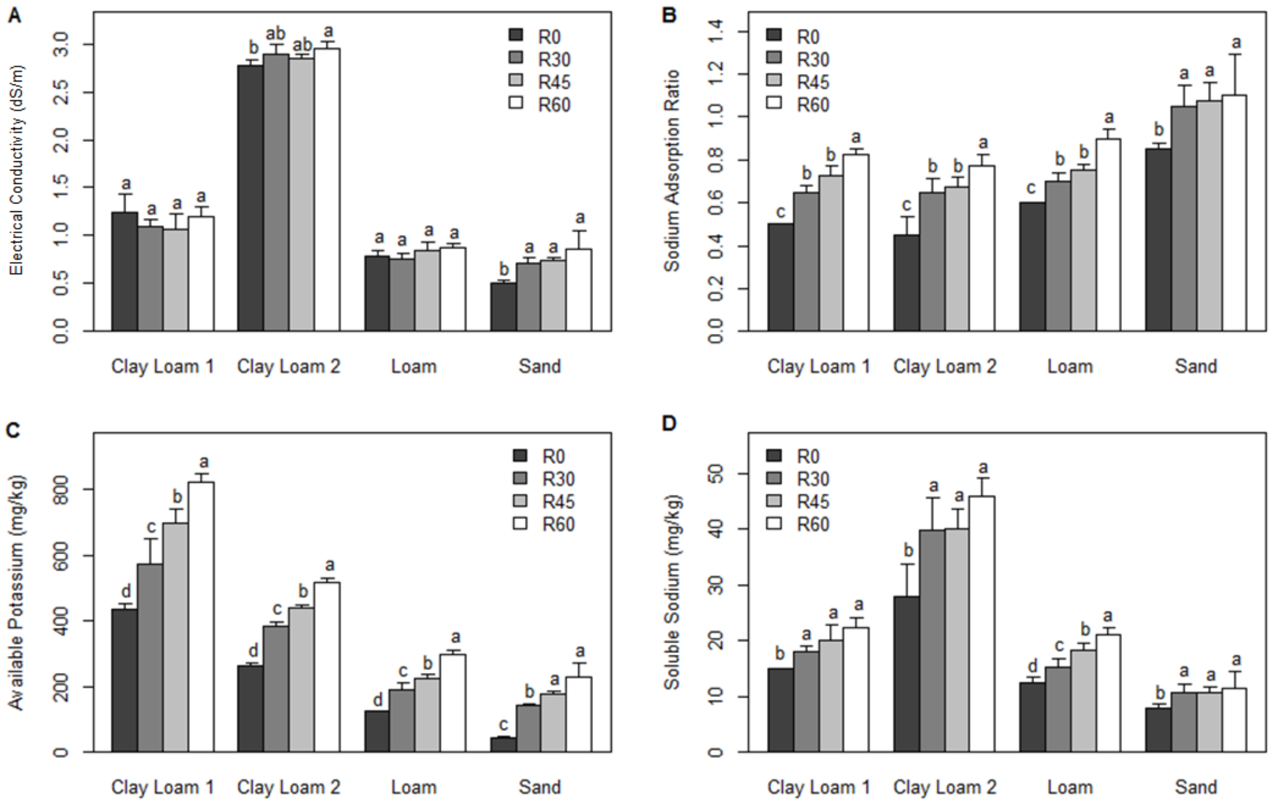

Figure 2.2. Mean soil electrical conductivity (A), sodium adsorption ratio (B), available potassium (C) soluble sodium (D) with and without PSDF. Errors bars are standard errors. Letters indicate significant differences among PSDF rates for the same soil. R0 = without PSDF; R30, R45, R60 = PSDF at 30, 45, 60 m^3 ha^{-1}.

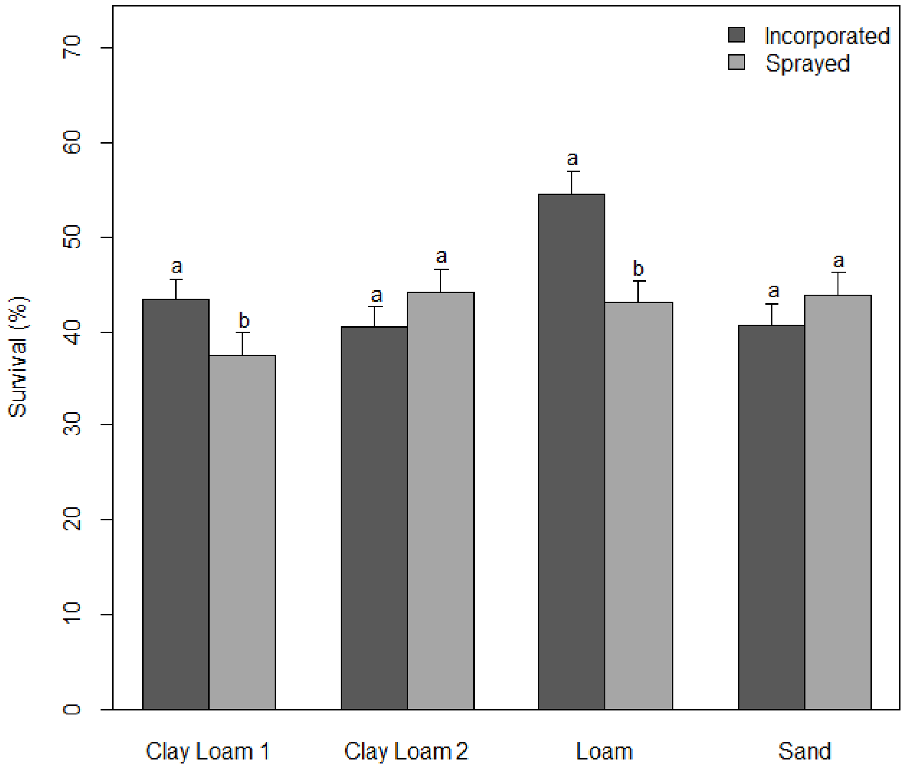

Figure 2.3. Significant interactions for survival (%) among soil type and PSDF application method. Error bars are standard error. Letters indicate significant differences between incorporated and sprayed PSDF for the same soil.

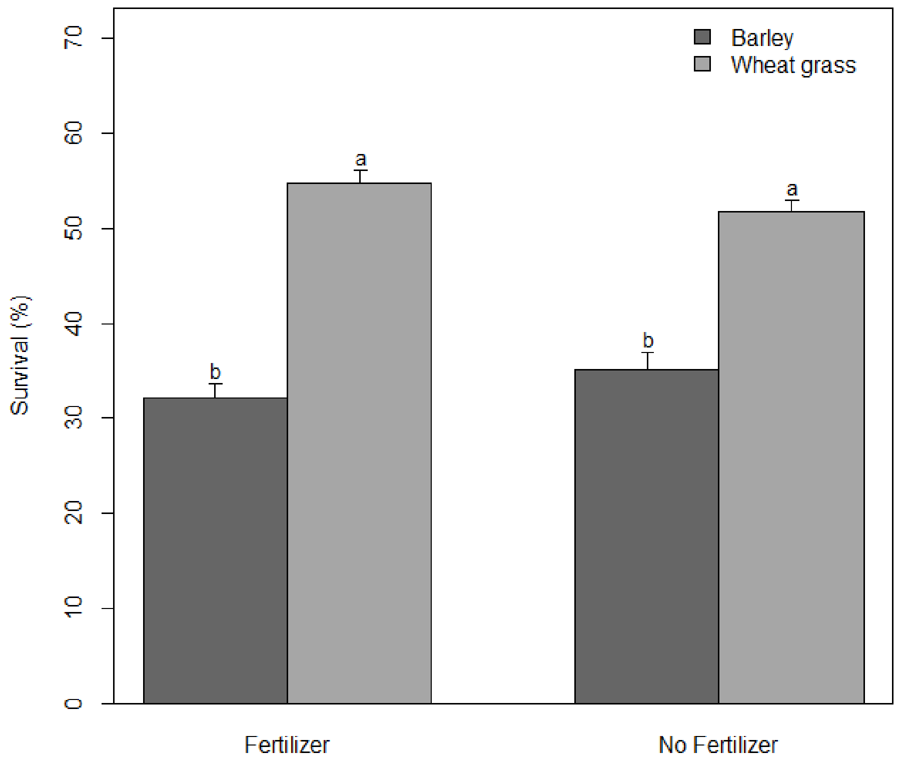

Figure 2.4. Significant interactions for plant survival (%) between barley and wheat grass with and without fertilizer. Error bars are standard error. Letters indicate significant differences between plant species with and without fertilizer.

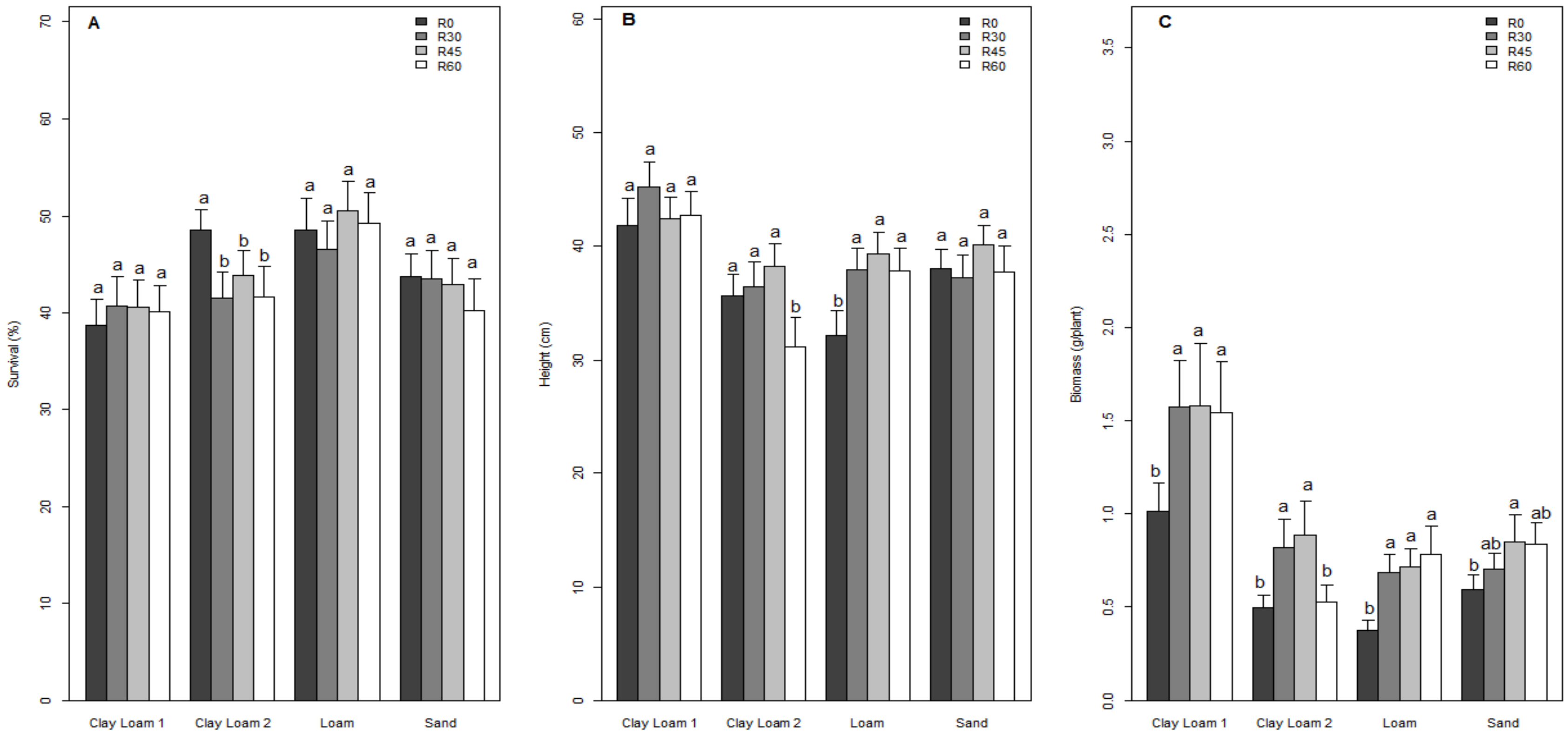

Figure 2.5. Plant survival (A), height (B) and above ground biomass (C) in soil with and without PSDF. Error bars are standard errors. Letters indicate significant differences among PSDF application rates for the same soil. R0 = without PSDF; R30, R45, R60 = PSDF at 30, 45, 60 m^3 ha^{-1}.

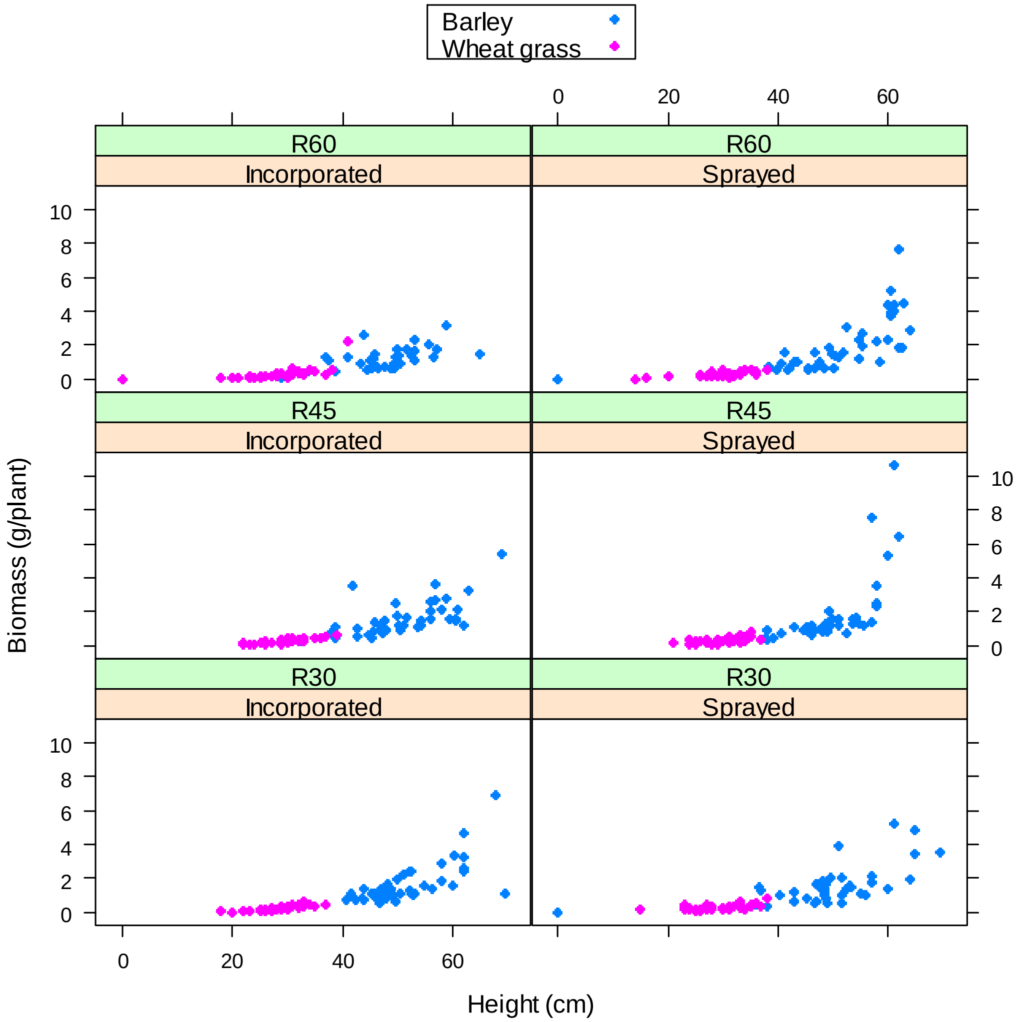

Figure 2.6. Significant interactions among plant species, PSDF application method and rate for plant height (cm) and biomass (g plant^{-1}). R30, R45, R60 = PSDF at 30, 45, 60 m^3 ha^{-1}. Biomass is above ground per plant in the same treatment. Height is the mean of plants in the same treatment.

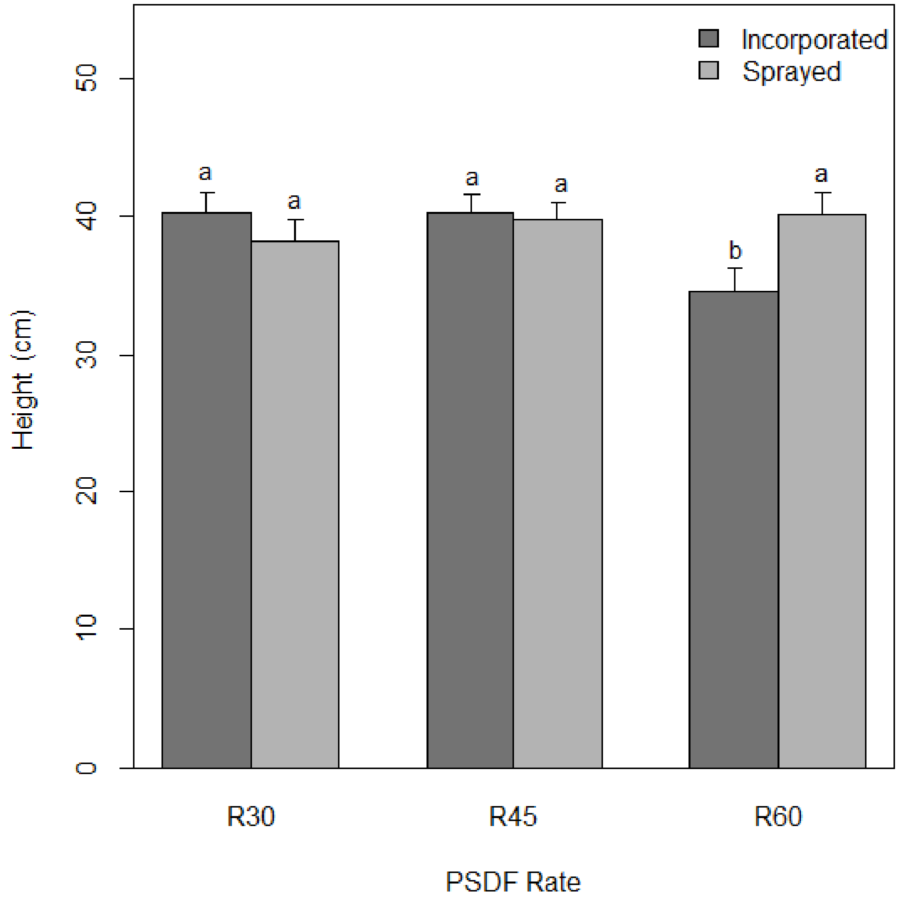

Figure 2.7. Significant interactions for plant height with sprayed and incorporated PSDF at three rates. Height is the mean of plants in the same treatment. R30, R45, R60 = PSDF at 30, 45 and 60 m^3 ha^{-1}. Error bars are standard error. Letters indicate significant differences between incorporated and sprayed PSDF at the same rates.

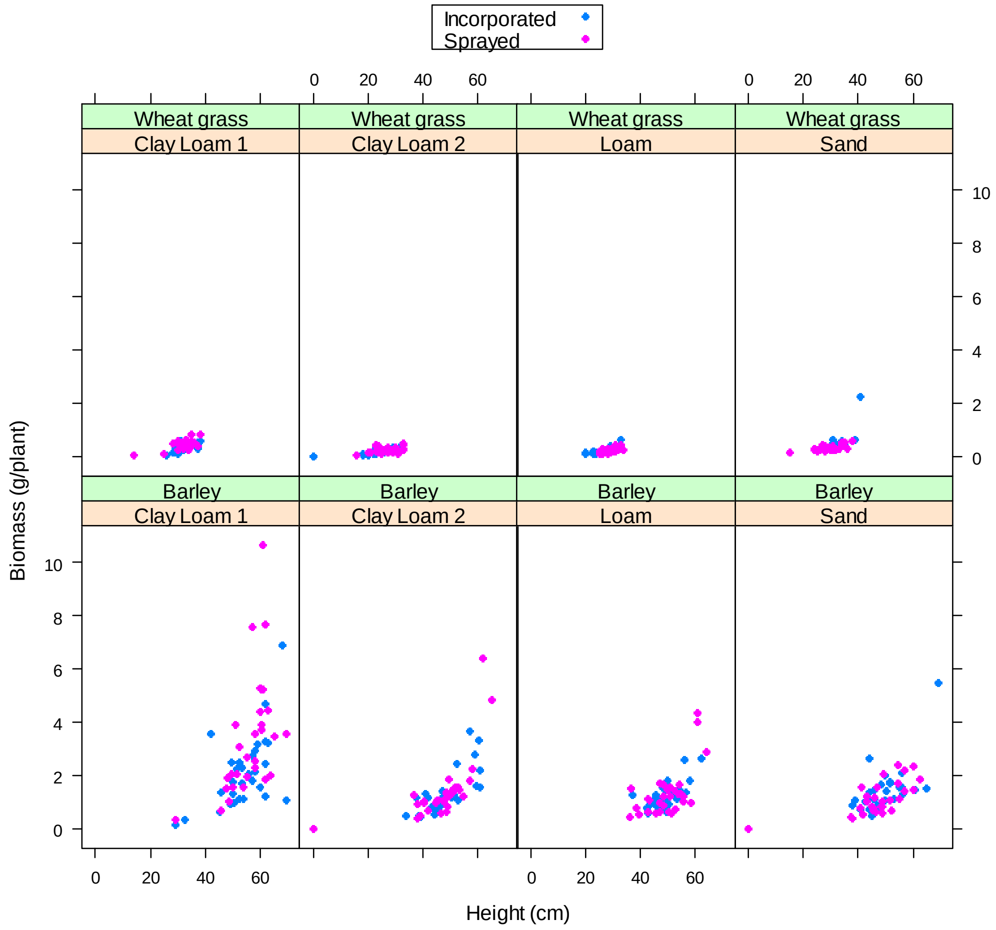

Figure 2.8. Significant interactions among soil type, PSDF application method and plant species for plant biomass. Biomass is above ground per plant in the same treatment. Height is the mean of plants in the same treatment.

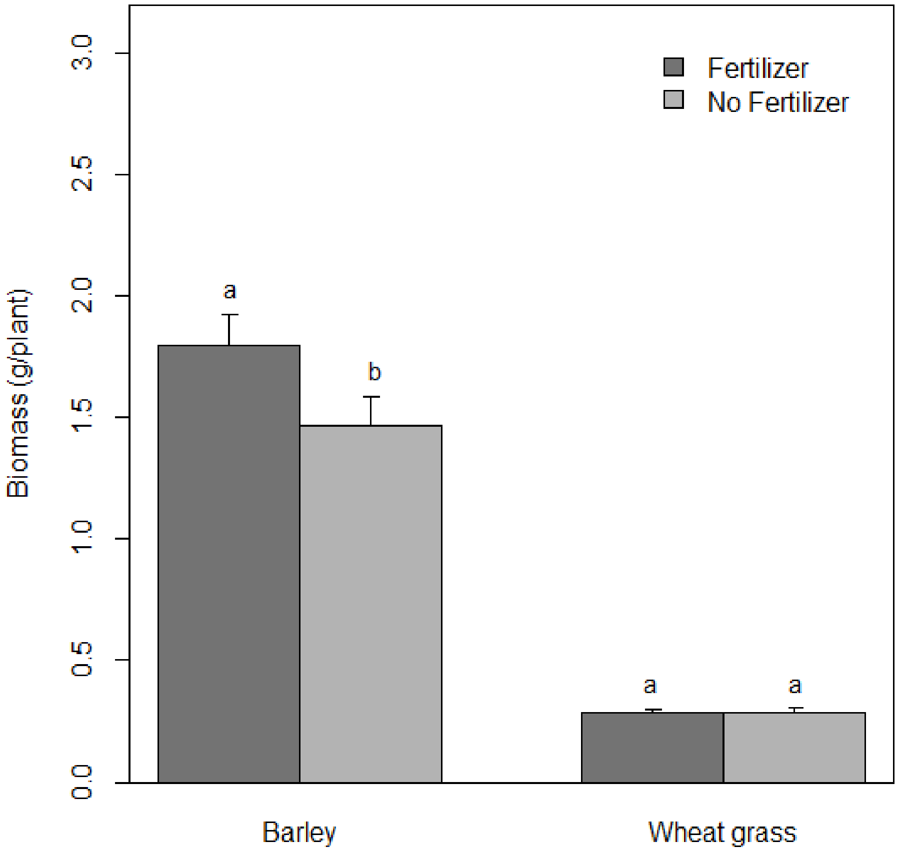

Figure 2.9. Above ground biomass of barley and wheat grass with and without fertilizer. Error bars are standard error. Letters indicate significant differences between fertilizer and no fertilizer for the same species.

CHAPTER 3. SOIL AND PLANT RESPONSES TO RECYCLED POTASSIUM SILICATE DRILLING FLUID IN A GREENHOUSE

1. Introduction

The path to economic and environmental sustainability requires a new way of thinking about oil and gas industry waste streams. In oil and gas operations, drilling fluids, often referred to as drilling muds, are pumped into the bore holes to aid in the drilling process and to transport drill cuttings to the surface. These fluids can be water based, oil based or synthetic based and consist of bentonite, barite, lignite, chrome lignosulphate, sodium hydroxide and various additives mixed with fresh water or hydrocarbons.

In recent years there has been a growing concern over potential environmental effects of land disposal of drilling fluids. Exploration and production in the oil and gas industry generate large volumes of drilling fluids and cuttings with many potential pollutants. This drives research, technology and regulations development with the goal of beneficial reuse and decreased footprints. Few studies have addressed the impact of disposal of spent drilling fluids on soil-plant-water systems. Some researchers found high soluble salts, heavy metals and petroleum residue content of used drilling fluids were detrimental to soil quality and plant growth (Zvomuya et al. 2011, Wojtanowicz 2008, Zvomuya et al. 2008, McFarland et al. 1994, McFarland et al. 1992, Nelson et al. 1984, Miller et al. 1980). Other researchers found positive or no impacts from drilling fluids applied at low rates in coarse textured soils in arid regions due to pH increase, potential micronutrient addition and/or improved soil physical properties (Bauder et al. 2005, Bauder et al. 1999, Lesky et al. 1989, Tucker 1985, Moseley Jr. 1983, Miller and Pesaran 1980). Differences among studies primarily resulted from drilling fluid properties, disposal method and disposal location.

Regulators have recognized that land application and beneficial reuse could be a potential means to reduce and dispose of drilling wastes. In western Canada, land spraying while drilling is a common disposal approach with criteria for maximum disposal rate and maximum acceptable increases in electrical conductivity, sodium adsorption ratio, sodium loading and nitrogen loading for water based drilling waste (Alberta Energy Resources Conservation Board 2012, Saskatchewan Ministry of Energy and Resources 2011, British Columbia Oil and Gas Commission 2006). Spent drilling fluids could be sprayed then incorporated on cultivated land or spread on vegetated land. Due to the shortage of available information, advanced gel chemical muds (e.g. potassium silicate, potassium sulphate), a kind of water based drilling mud system, were not included in the Alberta drilling waste regulatory framework for land spraying while drilling before 2012, and the Energy Resources Conservation Board required site specific approval prior to disposal (Energy Resources Conservation Board 2007).

Environmentally friendly drilling mud systems can minimize waste and decrease disposal cost. Potassium silicate drilling fluid (PSDF), an advanced gel drilling fluid with high performance, has been recently used in the oil and gas industry. PSDF is considered an environmentally acceptable alternative to traditional drilling fluids as it replaces hydrocarbons with silicate polymeric ions and lignite and sodium salts with potassium salts. PSDF has high concentrations of potassium, which industry and regulators see as a potential problem with salt accumulation and soil structure. Few studies addressed this concern. Processes that control potassium in soils from PSDF land disposal are complex and poorly understood, such as rate of potassium release, leaching and long term plant bioavailability. Potassium is required in many biochemical functions and increases plant tolerance to drought, low temperatures or salinity (Whitehead 2000, Tisdale et al. 1999). PSDF land application may result in potassium availability that can be in excess of plant requirements. Hypothetically, potassium in PSDF could serve as a nutrient amendment for reclamation, thus not only would land disposal be practical, but also provide benefits for reclamation.

In a recent greenhouse experiment (Chapter 2), use of raw PSDF had no detrimental effects on soil and plant properties at rates ≤ 45 m^3 ha^{-1}. Whether spent PSDF has the same impacts on soils and plants as raw PSDF is unknown as it varies with soils, drilling locations and recycling times. In this greenhouse experiment changes in properties of different textured soils and responses of a common agricultural crop species were studied to determine effects of raw versus spent PSDF applied at different rates, and with application by surface spraying versus incorporating PSDF.

2. Materials And Methods

2.1 Experimental Design

A greenhouse experiment was set up in a randomized complete block design with treatments replicated 5 times. Treatments were designed to represent reclamation application scenarios in cultivated land. Three soil textures were sand, loam and clay loam, covering the range of soils with potential for reclamation using drilling fluids. Three types of potassium silicate drilling fluids were raw, spent once and spent twice. Two application treatments were spraying over soil and spraying over soil followed by incorporation, approximating likely field application methods. Six potassium silicate drilling fluid application rates were developed around the current Energy Resources Conservation Board (2007) summer maximum loading rate of 40 m^3 ha^{-1}; they were 10, 20, 30, 40, 60 and 120 m^3 ha^{-1}. A common agricultural crop species, common barley (*Hordeum vulgare* L.) was used. Amount of fertilizer was based on optimum macro nutrients generally required by agronomic species so nutrient deficiencies would not be interpreted as plant response to PSDF application. Potassium fertilizer was not applied due to high concentrations in drilling fluid. Treatments were fertilized with 0.06 g pot^{-1} (34 kg ha^{-1}) ammonium nitrate and 0.07 g pot^{-1} (45 kg ha^{-1}) triple super phosphate.

2.2 Potassium Silicate Drilling Fluid and Soil Collection and Analyses

Raw potassium silicate drilling fluid was manufactured by Marquis Alliance Ltd. Spent once and spent twice PSDF were obtained from drilling sites that used the same raw PSDF. Spent twice drilling fluid was collected from well sites that recycled spent once PSDF. Soils were collected from 20 locations per site from the upper 20 cm at three locations in Alberta where drilling was active. Soil and PSDF properties were determined by Exova Laboratory Group. Three random soil samples from each treatment were stored in a refrigerator until analyzed by Exova Laboratory Group.

Soil samples were sieved to remove large particles, ground to < 2 mm and the following analyses conducted. Available nitrate (NO_3^-) and ammonium (NH_4^+) were determined by extraction with 2.0 M potassium chloride (KCl) (Carter and

Gregorich 2008); available phosphorus and potassium by modified Kelowna extraction (Ashworth and Mrazek 1995); and available sulphate by extraction with 0.1 M calcium chloride ($CaCl_2$) (McKeague 1978). Cation exchange capacity was determined by exchange with ammonium acetate (NH_4OA_C) at pH 7 (McKeague 1978). Total nitrogen was determined by Kjeldahl digestion distillation (Bremner 1996). Total carbon was determined by dry combustion (Nelson and Sommers 1996) and total organic carbon by the Walkley-Black wet dichromate oxidation method (Nelson and Sommers 1996). Water soluble cations (sodium, calcium, potassium, magnesium), pH, sodium adsorption ratio and electrical conductivity were determined from saturated paste extracts (Carter and Gregorich 2008). Routine ions (sulphate, chloride) were determined by ion chromatography with chemical suppression (Clesceri et al. 1992). Hydrocarbon fractions (F1, F2, F3, F4) were analyzed by photoionization and electrolytic conductivity detectors (Environmental Protection Agency 1996). Trace metals restricted in concentration by the Canadian Council of Ministers of the Environment (silver, arsenic, barium, beryllium, cadmium, cobalt, chromium, copper, mercury, molybdenum, nickel, lead, antimony, selenium, tin, thallium, uranium, vanadium, zinc) and boron were measured using a nitric acid hotplate digestion method (Environmental Protection Agency 2008). Sand, silt and clay were determined by hydrometer after treatment with calgon (Carter and Gregorich 2008).

2.3 Greenhouse Methods and Plant Measurements

Soil was placed in 15 cm diameter pots. PSDF was applied at rates of 10, 20, 30, 40, 60 and 120 m^3 ha^{-1}. Untreated control pots were included for each soil as controls. PSDF was evenly applied by hand on the soil surface by pouring from beakers. PSDF was incorporated with shovels for incorporation treatments. Pots were allowed to settle for one week before seeding.

Pots were seeded with 20 seeds of common barley (*Hordeum vulgare* L.), then randomly placed on a greenhouse bench blocked by replication. Pots were watered regularly twice per day for the first 4 weeks and once per day thereafter, with sufficient water to keep the pots at field capacity by gravimetric methods throughout the four month experiment. Greenhouse temperature was maintained at 21 °C during the day and 15 °C at night, with a 16 hour photoperiod.

Number of plants emerging and their death in each pot was recorded weekly. After 2 weeks, plants were thinned to the five most vigorous plants per pot. At the seed production stage, above ground biomass from each pot was clipped at ground level. Roots in sandy soil were hand separated from soil; large gravel particles in loam and clay loam soils were removed by hand, then pots were soaked in water for 12 hours in a tray. Roots were cleaned by washing with tap water. All biomass was oven dried at 80 °C for 48 hours to constant weight.

2.4 Statistical Analyses

Analyses were conducted with R software (R Development Core Team 2012). Soil and plants analyses were conducted separately with four way analysis of variance (ANOVA). Data were checked for violations of normality assumption with Shapiro-Wilk test and equal variances assumption with Bartlett's test. Where significant treatment effects were determined an $LSD_{0.05}$ was calculated to provide a method of comparing means and measure of precision. Only when the F test was significant were statistical differences among means determined. To investigate the responses of plants (emergence, death, above ground biomass, below ground biomass) to PSDF application and soil properties, redundancy analysis (RDA) was performed.

3. Results

3.1 Soil Responses to PSDF

PSDF had a density of 1137 kg m^{-3}, equivalent to a dry bulk density of 220 kg m^{-3}, as determined by the manufacturer. Raw PSDF components and properties are presented in Tables 3.1 and 3.2; most notably, raw PSDF had high pH and electrical conductivity and did not contain hydrocarbons. Unamended soil properties are presented in Table 3.3.

Electrical conductivity and sodium adsorption ratio were greater with PSDF than without (Table 3.4). Soil, PSDF type and rate and their interactions were significant for pH, electrical conductivity, sodium adsorption ratio and saturation, except PSDF type for pH (Tables 3.4, 3.5). Electrical conductivity and sodium adsorption ratio were higher in clay loam with spent PSDF than in loam and sand soils (Table 3.6). Except for pH in loam soil, where spent once PSDF had significantly higher values of these properties than spent twice. Electrical conductivity, pH, sodium adsorption ratio and saturation were significantly affected by PSDF rate (Table 3.5). PSDF significantly raised pH on slightly acidic loam soil at 120 m^3 ha^{-1} and basic sand soil at ≥ 60 m^3 ha^{-1} but not on basic clay loam soil (Figure 3.1 A). Electrical conductivity and sodium adsorption ratio increased with increasing PSDF rate with significantly greatest values at 120 m^3 ha^{-1} (Figure 3.1 B, C). Saturation percentage increased in clay loam and sand soil with increasing PSDF rate, and was highest at a rate of 120 m^3 ha^{-1}, whereas in loam soil greatest saturation was with lowest PSDF (Figure 3.1 D).

Available macronutrients varied slightly in soils with and without PSDF (Table 3.7). Soil, PSDF type and rate and their interactions with application method were significant for available nitrogen, phosphorus, potassium and sulphur (Table 3.8). These macronutrients increased significantly with spent once PSDF relative to spent twice PSDF; loam soil with PSDF had highest available nitrogen, phosphorus and potassium (Table 3.9). Clay loam soil with sprayed PSDF had significantly more available nitrogen than incorporated PSDF, with the opposite in sand soil (Figure 3.2). Sprayed PSDF at ≥ 60 m^3 ha^{-1} significantly decreased available nitrogen (Figure 3.3). Available phosphorus was significantly lower in clay loam than sand and loam soil (Table 3.7). Available potassium increased significantly with PSDF rate in all soils (Figure 3.4), with both once and spent twice PSDF (Figure 3.5).

Soil type and its interactions with PSDF type, application method and rate had significant effects on cation exchange capacity, total organic carbon, total carbon and total nitrogen (Tables 3.10, 3.11). Sand soil with spent PSDF had significantly lower values than loam or clay loam soils (Table 3.12). Spent once PSDF had significantly greater cation exchange capacity than spent twice PSDF, with opposite results for total carbon and organic carbon. There were no significant differences within the same application method with increasing rate of PSDF (Table 3.13). Only in loam soil was application method significant for cation exchange capacity; incorporated PSDF had significantly greater values than sprayed (Figure 3.6).

Soluble potassium and sodium increased significantly with increasing PSDF (Figure 3.7 A, B), with sodium significantly highest in clay loam soil (Figure 3.7 B). Soluble magnesium was significantly highest in loam soil (Figure 3.7 C). Only at 120 m^3 ha^{-1} was there a significant decrease in soluble magnesium in loam and sand soil, and a decrease in soluble calcium in sand soil (Figure 3.7 C, D). When the potassium and sodium ratio increased, sodium adsorption ratio decreased (Figure 3.8).

With PSDF, all metals except barium were lower than regulation limits (Table 3.14) (Alberta Environment and Sustainable Resource Development 2010). Barium was highest in soil with spent once PSDF than with no PSDF and 10 times higher than with spent twice PSDF. Relative to the control, chromium increased in clay loam soil, cadmium and lead increased in loam, and lead increased in sand soil.

3.2 Barley Responses to PSDF

Soil, PSDF type, application method and rate and their interactions were significant for barley emergence (Tables 3.15, 3.16). With spent twice relative to raw PSDF emergence dropped significantly in each soil (Table 3.17). Emergence was significantly lowest with PSDF in clay loam than other soils. Emergence decreased significantly with increasing PSDF, especially in sand and loam soils; and was significantly lower with 120 m^3 ha^{-1} PSDF in clay loam and sand soils relative to the control (Table 3.18).

Interactions among soil and PSDF type, or soil and PSDF application method were significant for barley death (Table 3.16). Loam soil had significantly fewer deaths than sand and clay loam except with spent twice PSDF (Table 3.17). Effects of application method on death varied with soil type, with incorporated PSDF in sand having fewest deaths (Figure 3.9). PSDF application rate effects were significant only for clay loam soil which had higher deaths at all application rates than without PSDF (Table 3.18). As deaths averaged < 0.5 plants per pot, soil and PSDF effects were not biologically significant (Table 3.16).

Soil and PSDF type and their interactions with PSDF application rate were significant for above and below ground biomass (Table 3.16). Significantly highest above ground biomass was in loam soil with spent twice PSDF (Table 3.17). Raw PSDF in loam soil had highest below ground biomass. Relative to controls, above and below ground biomass were significantly higher with rates ≥ 20 m^3 ha^{-1} PSDF in loam soil and significantly lower in clay loam soil at 120 m^3 ha^{-1} (Table 3.18).

In the RDA plot, 67 % of the change was explained by axis 1, 21 % by axis 2 and 88 % by the cumulative proportion (Figure 3.10). With PSDF plant emergence was greater on sand and loam soils than clay loam. Emergence was positively affected by available phosphorus and magnesium and significantly negatively affected by sodium adsorption ratio, electrical conductivity and sodium, sulphate and chloride concentrations. Above ground biomass was greater with spent twice PSDF in loam soils, and significantly positively correlated with available nitrogen and potassium, total carbon and nitrogen, total organic carbon, cation exchange capacity and saturation. Below ground biomass was correlated with emergence and above ground biomass, and enhanced by PSDF in loam soil. Soil pH was negatively correlated with biomass.

4. Discussion

4.1 Soil Responses to PSDF

PSDF significantly increased soluble and exchangeable potassium. Since most potassium in PSDF is immediately available, high potassium PSDF rates (6,900 to 21,700 mg L^{-1}) can consistently and rapidly increase solution potassium. For example, potassium rich effluent on rye grass *(Lolium perenne* L. cv. Manhatan 2) significantly increased available and extractable potassium, surpassing that of mineral potassium fertilizer (Gallardo-Lara et al. 1995). Loam soils with high clay content and slight acidity will more likely benefit from potassium in PSDF, explaining the significantly greater biomass in loam soil than other soils.

High potassium PSDF could be used to alleviate soil sodicity. Although sodium adsorption ratio increased with increased PSDF, it dramatically decreased when the potassium and sodium ratio increased. The effect of high concentrations of potassium in drilling fluid on selectivity of exchange sites for potassium in relation to sodium, magnesium and calcium was not clear in previous studies. Some studies showed calcium and magnesium were selectively adsorbed relative to potassium (Levy and Torrento 1995), while others reported soil preference for potassium over calcium, magnesium and sodium (Lieffering and Mclay 1995). Potassium exchange reactions with sodium, calcium and magnesium on clay minerals and soils have been related to mineral composition, salinity and water content of the soil. Robbins (1984) found an increase in the potassium to sodium ratio from 1:20 to 1:1 in soil solution dramatically decreased soil preference for sodium. Exchangeable potassium was more tightly held on exchange sites than sodium, calcium and magnesium, thus soluble salts were leached, reducing high sodium effects. However, high potassium concentrations may decrease hydraulic conductive and permeability and increase soil erodibility (Hao and Chang 2003).

Sand soil with PSDF had an immediate effect on cation exchange capacity. Although cation exchange capacity was lowest in sand soil due to lower total organic carbon and clay content, it was more sensitive to PSDF, significantly increasing. As more clay will remain in drilling fluid when transporting drill cuttings to the surface during drilling operations, material with starch and xanthan gum will increase the ability of sandy soils to attract, retain and exchange cations. This explains the positive impacts of drilling fluids in coarse textured soils (Moseley Jr. 1983). Materials such as starches, gum and tannin in drilling fluids can also stimulate plant growth (Miller et al. 1980).

PSDF can either increase soil pH or have no effect on it, dependent on PSDF pH, initial soil pH and pH buffering capacity of the soil. Increased pH with PSDF on loam and sand soils but not clay loam is expected, given the relatively high pH of PSDF, the slightly acidic loam soil and lower buffering capacity of sandy soil. Other researchers found similar pH increases in sandy, acidic soils following drilling fluid application (Bauder et al. 2005, Miller and Pesaran 1980).

Although fresh PSDF with high potassium and sodium increased soil electrical conductivity and sodium adsorption ratio, all our soils with PSDF were classified as good quality for revegetation relative to disturbance and reclamation for unrestricted land use except clay loam (electrical conductivity < 2, sodium adsorption ratio < 4) (Alberta Environmental Sciences Division 2001). Sodium adsorption ratio of approximately 10 in clay loam before PSDF application led to low quality classification.

Increased barium with PSDF was due to barite (barium sulphate) to increase drilling fluid density and was attenuated with increased use. Barium is insoluble, inert and nontoxic (Monaghan 1980). Miller and Pesaran (1980) found barite drilling fluids mixed with soil had no effect on green beans (*Phaseolus vulgaris* L.). Increased chromium, cadmium and lead in some treatments may be due to components of PSDF, such as xanthate and bentonite clay as sorbents for heavy metals (Bailey et al. 1999).

4.2 Plant Responses to PSDF

There was no evidence of detrimental effects on plant establishment and development at PSDF application rates ≤ 60 m^3 ha^{-1}, which is higher than the maximum disposal rate (40 m^3 ha^{-1}) in regulations for western Canada (Alberta Energy Resources Conservation Board 2012, Saskatchewan Ministry of Energy and Resources 2011, British Columbia Oil and Gas Commission 2006).

The significant positive response between biomass production and soil available potassium indicated PSDF application could alter potassium which may benefit plant growth. Soluble potassium plays a critical role in salt tolerance in barley, which is highly correlated with physiology (Chen et al. 2007). High cocnentrations of potassium could improve enzyme activation, charge balance and osmoregulation, and help plants exclude sodium (Taiz and Zeiger 1998). This partly explained a light acidic loam soil with greater biomass with PSDF than without PSDF.

A significant increase in sodium, electrical conductivity and sodium adsorption ratio with PSDF ≥ 60 m^3 ha^{-1} potentially limited plant growth, but was not detrimental to biomass. Sodium and potassium from PSDF increased osmotic pressure of the soil solution, reducing plant water uptake and growth. PSDF applied to basic clay loam or sand soil will raise pH which may inhibit plants. Miller and Pesaran (1980) studied effects of seven drilling fluids on green beans (*Phaseolus vulgaris* L.) and sweet corn (*Zea mays* L.) in a greenhouse and attributed reduced plant growth to high soluble salts or exchangeable sodium.

Nitrogen and phosphorus fertilizer with PSDF enhanced plant performance. Although inorganic ammonium nitrate fertilizer was loaded at 60 mg pot^{-1}, soil available nitrogen was 2.0 to 3.6 mg kg^{-1}. This was not surprising, considering nitrate is easily transported with percolating water. Zvomuya et al. (2011) found nitrogen concentration in above ground plant tissue increased within 45 days of drilling fluid application, but differences disappeared 1 year after treatment. Since organic matter plays an important role in nitrate immobilization (Willms and Jefferson 1993), treatments in sandy soil with low organic content resulted in significantly lower available nitrate than that of other soils in our study. RDA showed a strong relationship between available nitrogen and above ground biomass. Similar nitrogen fertilizer effects on increasing biomass were reported in other studies following nitrogen application (Jacobsen et al. 1996, Power 1985, Black and Wight 1979). The lower total carbon and positive relationship with biomass in our study suggest organics (Adedokun and Ataga 2007, Ogboghodo et al. 2004, Callaham et al. 2002) in PSDF may enhance development of microbiological processes in soil and thereby improve carbon nitrogen ratio.

Plant emergence and below ground biomass were significantly correlated with available phosphorus, even though it is a slowly available element. Significant phosphorus effects in plant tissue were only observed 3 years after drilling mud disposal by (Zvomuya et al. 2011), although relative to phosphorus carried by drilling mud, inorganic phosphate from fertilizer was immediately available. In our study, since initial phosphorus varied with soil, it was strongly adsorbed to soil, explaining the significantly lower phosphorus in clay loam soil.

Sulphur in PSDF, did not affect emergence or biomass, which could be partly explained by clay loam soils being initially high in available sulphur (108 mg kg^{-1}). High concentrations of exchangeable sulphate in spent PSDF were 2437 to 3016 mg kg^{-1}, above critical levels of 384 mg kg^{-1} for corn and 480 mg kg^{-1} for alfalfa (Fox et al. 1964). There was no consistent response when sulphur fertilizer was applied to soil with high available sulphur (Mnkeni and Mackenzie 1981).

Effect of PSDF recycling times on plants was inconsistent. The significantly lower electrical conductivity and sodium adsorption ratio in spent twice PSDF relative to spent once could benefit plants. However, available potassium, phosphate and sulphate and base saturation decreased with PSDF recycling times as additives to increase drilling fluid density and corrosion inhibition of oil and gas pipes are attenuated during drilling. It is possible that such adverse, and confounding effects may counteract plant benefits from decreasing soil salinity and sodicity.

Concerns were raised that spraying PSDF on soil would inhibit plant establishment and development. An earlier field study found sprayed water based drilling fluid impacted plant health and productivity due to impeded near saturated hydraulic conductivity (Zvomuya et al. 2009), and altered albedo and soil heat flux at the soil surface (Zvomuya et al. 2008). Drilling fluid clogged conductive macropores; water failing to infiltrate remains on the soil surface and is subject to runoff or evaporation loss, and therefore not available for plant uptake or soil storage. Seeding in our study, after a low rate of drilling fluid was applied then left to settle for 1 week, led to cracked PSDF on the soil surface which may have led to less reduction of water infiltration rate. Thus spraying and incorporating PSDF gave similar plant response.

5. Conclusions

PSDF can be used at ≤ 60 m^3 ha^{-1} rates to increase barley production under greenhouse conditions. PSDF significantly increased available potassium, ratio of soluble potassium to sodium and cation exchange capacity, and alleviated soil sodicity. Effect of raw versus spent PSDF or application method on plant properties was insignificant. Soil texture had a significant effect on plants. PSDF on slightly acidic and medium textured soil will have maximum benefits from high potassium in PSDF to soil properties and plants growth.

The main contribution of this research was to show that PSDF at high concentrations can have significant positive effects on soils and plants. This has important implications for management of advanced gel chemical mud systems such as PSDF.

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Table 3.1. PSDF and traditional water based drilling fluid components and their functions.

Function	Traditional Water Based Mud	Potassium Silicate Drilling Fluid
Control viscosity	Bentonite	Pregelatinized starch Xanthan gum
Thin the mud	Lignite or lignosulfonate	Potassium pyrophosphate
Disperse solid particles	Sodium hydroxide (NaOH)	Potassium hydroxide (KOH)
Increase density	Barite ($BaSO_4$) or hematite (Fe_2O_3)	
Control fluid loss		Polyanionic cellulose Pregelatinized starch
Stabilize the formation		Raw silicate Anionic water soluble polymer

Source: Marquis Alliance Ltd. (Ma 2008).

Table 3.2. Selected PSDF properties.

	Raw	Spent Once	Spent Twice
Density (kg m^{-3})	1.29	1.13	0.90
Hydrogen ion activity (pH)	11.4	10.8	10.7
Electrical conductivity (dS m^{-1})	13.9	61.8	44.3
Sodium adsorption ratio	7.1	866.7	436.0
Calcium (mg kg^{-1})	82.1	15.7	31.8
Magnesium (mg kg^{-1})	< 17.6	7.0	4.3
Sodium (mg kg^{-1})	255.1	11633.3	9406.7
Potassium (mg kg^{-1})	6033.4	19200.0	9646.7
Chloride (mg kg^{-1})	91.8	1416.7	2116.7
Sulphate (mg kg^{-1})	< 85.3	3016.7	2436.7
Nitrate and nitrite (mg kg^{-1})	0.4	7.2	< 5.6
Benzene (mg kg^{-1})	ND	2.0	0.4
Toluene (mg kg^{-1})	ND	5.8	1.3
Ethylbenzene (mg kg^{-1})	ND	1.6	0.5
Total Xylenes (mg kg^{-1})	ND	11.3	3.5
F1 C_6 - C_{10} (mg kg^{-1})	ND	90.3	116.3
F2 C_{10} - C_{16} (mg kg^{-1})	ND	642.0	1470.0
F3 C_{16} - C_{34} (mg kg^{-1})	ND	1456.7	1810.0
F4 C_{34} - C_{50} (mg kg^{-1})	ND	578.7	548.0
Boron (mg kg^{-1})	0.1	5.1	9.6
Mercury (mg kg^{-1})	< 0.0002	0.1	0.1
Antimony (mg kg^{-1})	< 0.004	0.0	0.0
Arsenic (mg kg^{-1})	0.0	11.5	11.8
Barium (mg kg^{-1})	0.3	9430.0	4803.3
Beryllium (mg kg^{-1})	< 0.002	0.6	1.0
Cadmium (mg kg^{-1})	< 0.0002	0.2	0.2
Chromium (mg kg^{-1})	0.1	31.6	46.7
Cobalt (mg kg^{-1})	0.004	8.2	11.8
Copper (mg kg^{-1})	0.1	47.7	39.0
Lead (mg kg^{-1})	0.04	42.4	21.7
Molybdenum (mg kg^{-1})	< 0.02	4.0	4.0
Nickel (mg kg^{-1})	0.01	28.3	38.2
Selenium (mg kg^{-1})	< 0.004	1.03	1.03
Silver (mg kg^{-1})	0.02	0.6	0.5
Thallium (mg kg^{-1})	< 0.001	0.2	0.3
Tin (mg kg^{-1})	< 0.02	1.0	< 1.00
Uranium (mg kg^{-1})	0.01	0.9	1.3
Vanadium (mg kg^{-1})	0.1	37.4	60.9
Zinc (mg kg^{-1})	0.1	68.0	74.0

ND = not determined.

Table 3.3. Selected soil properties before PSDF application.

	Clay loam	Loam	Sand
Available nitrogen (mg kg^{-1})	< 2.0	84.3	1.0
Available phosphorus (mg kg^{-1})	< 5.0	34.0	14.0
Available potassium (mg kg^{-1})	312.7	350.7	43.3
Available sulphur (mg kg^{-1})	108.0	7.0	< 1.0
Cation exchange capacity (meq 100 g $^{-1}$)	23.3	24.0	2.0
Total carbon (%)	2.2	2.3	0.1
Total organic carbon (%)	1.7	2.0	0.1
Total nitrogen (%)	0.0	0.2	< 0.02
Hydrogen ion activity (pH)	7.9	4.8	6.5
Electrical conductivity (dS m^{-1})	2.0	1.4	0.1
Sodium adsorption ratio	9.9	0.1	0.3
Soluble sodium (mg kg^{-1})	207.7	3.3	1.0
Soluble calcium (mg kg^{-1})	46.4	82.4	2.2
Soluble magnesium (mg kg^{-1})	7.2	24.4	0.5
Soluble potassium (mg kg^{-1})	10.0	12.3	0.3
Soluble chloride (mg kg^{-1})	3.7	3.0	1.0
Soluble sulphate (mg kg^{-1})	127.0	5.1	0.5
Soluble nitrate (mg kg^{-1})	2.9	83.2	1.6
Total C6-C10 (mg kg^{-1})	< 1.0	< 1.0	< 1.0
Total C11-C40+ (mg kg^{-1})	460.0	590.0	13.3
Sand (%)	38.9	33.1	93.8
Silt (%)	28.2	41.3	1.7
Clay (%)	32.9	25.6	4.5

Table 3.4. Soil detailed salinity in soils with PSDF after 4 months.

		Hydrogen Ion Activity (pH)	Electrical Conductivity (dS/m)	Sodium Adsorption Ratio	Saturation (%)
Soil	Clay Loam	8.0 ± 0.0	1.8 ± 0.1	7.3 ± 0.2	56.1 ± 0.8
	Loam	7.0 ± 0.1	1.0 ± 0.0	1.4 ± 0.1	57.0 ± 0.6
	Sand	8.1 ± 0.0	0.7 ± 0.0	0.9 ± 0.1	29.9 ± 0.2
PSDF	PSDF 1	7.7 ± 0.0	1.4 ± 0.1	3.8 ± 0.3	50.7 ± 1.4
	PSDF 2	7.7 ± 0.1	1.0 ± 0.1	2.6 ± 0.3	44.6 ± 1.1
Application Method	Incorporated	7.7 ± 0.1	1.2 ± 0.1	3.0 ± 0.3	47.8 ± 1.3
	Sprayed	7.7 ± 0.1	1.2 ± 0.1	3.4 ± 0.3	47.6 ± 1.3
Rate (m^3 ha^{-1})	0	7.6 ± 0.1	0.9 ± 0.1	2.4 ± 0.4	48.6 ± 2.9
	10	7.6 ± 0.1	1.0 ± 0.1	2.6 ± 0.5	47.9 ± 2.4
	20	7.6 ± 0.1	1.0 ± 0.1	2.7 ± 0.5	47.7 ± 2.5
	30	7.6 ± 0.1	1.1 ± 0.1	2.9 ± 0.5	45.9 ± 2.1
	40	7.7 ± 0.1	1.2 ± 0.1	3.0 ± 0.5	47.2 ± 2.2
	60	7.7 ± 0.1	1.3 ± 0.1	3.5 ± 0.6	47.7 ± 2.2
	120	7.8 ± 0.1	1.5 ± 0.1	4.6 ± 0.6	49.6 ± 2.3

PSDF types = spent once (1), spent twice (2).
Means within a column within treatments of same variable followed by standard errors.

Table 3.5. P value for detailed salinity in soils with PSDF after 4 months.

	Hydrogen Ion Activity (pH)	Electrical Conductivity (dS/m)	Sodium Adsorption Ratio	Saturation (%)
Soil (S)	< 0.001	< 0.001	< 0.001	< 0.001
PSDF (P)	0.541	< 0.001	< 0.001	< 0.001
Application Method (M)	0.653	0.542	< 0.001	0.641
Rate (R, m^3 ha^{-1})	< 0.001	< 0.001	< 0.001	0.001
S × P	< 0.001	0.031	< 0.001	< 0.001
S × M	0.002	0.617	< 0.001	0.044
P × M	0.057	0.461	0.604	0.792
S × R	0.003	0.153	< 0.001	< 0.001
P × R	< 0.001	0.150	0.236	0.253
M × R	0.010	0.774	0.026	0.626
S × P × M	0.002	0.092	0.013	0.097
S × P × R	< 0.001	0.890	0.740	0.186
S × M × R	< 0.001	0.142	0.287	0.632
P × M × R	0.019	0.871	< 0.001	0.149
S × P × M × R	< 0.001	0.007	0.996	0.225

Table 3.6. Electrical conductivity, pH, sodium adsorption ratio and saturation in soil with PSDF types.

	Clay Loam	Loam	Sand
Hydrogen Ion Activity (pH)			
PSDF 1	7.8 (0.02)	7.6 (0.03)	7.8 (0.04)
PSDF 2	8.1 (0.01)	6.6 (0.05)	8.3 (0.02)
Electrical Conductivity (dS/m)			
PSDF 1	2.0 (0.09)	1.3 (0.05)	0.8 (0.03)
PSDF 2	1.6 (0.08)	0.8 (0.04)	0.5 (0.04)
Sodium Adsorption Ratio			
PSDF 1	8.6 (0.2)	1.5 (0.1)	1.1 (0.2)
PSDF 2	6.0 (0.3)	1.2 (0.1)	0.7 (0.1)
Saturation (%)			
PSDF 1	61.8 (0.7)	59.5 (0.9)	30.9 (0.3)
PSDF 2	50.4 (0.7)	54.6 (0.7)	28.9 (0.2)

Means within treatments of same soil and PSDF type followed by standard errors.

Table 3.7. Available nutrients in soils with PSDF after 4 months.

		Nitrogen (mg kg^{-1})	Phosphorus (mg kg^{-1})	Potassium (mg kg^{-1})	Sulphur (mg kg^{-1})
Soil	Clay Loam	2.4 ± 0.1	5.0 ± 0.0	368.0 ± 10.9	119.9 ± 7.8
	Loam	3.6 ± 0.2	34.9 ± 1.3	559.3 ± 23.5	53.3 ± 3.1
	Sand	2.0 ± 0.0	22.5 ± 0.6	152.8 ± 13.0	15.7 ± 1.0
PSDF	PSDF 1	2.9 ± 0.1	22.2 ± 1.3	407.2 ± 23.8	81.9 ± 6.8
	PSDF 2	2.4 ± 0.1	19.4 ± 1.4	312.9 ± 16.8	44.0 ± 3.7
Application Method	Incorporated	2.5 ± 0.1	21.2 ± 1.5	355.9 ± 20.4	66.8 ± 6.3
	Sprayed	2.8 ± 0.2	20.4 ± 1.3	364.2 ± 21.7	59.1 ± 5.2
Rate (m^3 ha^{-1})	0	3.0 ± 0.2	17.4 ± 1.6	242.7 ± 25.2	56.8 ± 9.4
	10	2.6 ± 0.2	20.3 ± 2.1	242.7 ± 25.2	56.8 ± 9.4
	20	2.6 ± 0.2	19.8 ± 2.0	285.0 ± 28.1	56.0 ± 7.8
	30	3.1 ± 0.3	21.4 ± 2.3	309.3 ± 27.4	60.6 ± 10.6
	40	2.7 ± 0.3	20.5 ± 2.1	338.3 ± 30.7	63.9 ± 9.2
	60	2.4 ± 0.2	20.4 ± 2.2	401.4 ± 33.5	62.1 ± 9.2
	120	2.5 ± 0.2	22.3 ± 3.3	583.6 ± 42.2	78.5 ± 13.0

PSDF types = spent once (1), spent twice (2).
Means within a column within treatments of same variable followed by standard errors.

Table 3.8. P values for available nutrients in soils with PSDF after 4 months.

	Nitrogen (mg kg^{-1})	Phosphorus (mg kg^{-1})	Potassium (mg kg^{-1})	Sulphur (mg kg^{-1})
Soil (S)	< 0.001	< 0.001	< 0.001	< 0.001
PSDF (P)	< 0.001	< 0.001	< 0.001	< 0.001
Application Method (M)	0.015	0.227	0.204	0.086
Rate (R, m^3 ha^{-1})	0.079	0.262	< 0.001	0.051
S × P	0.100	0.013	< 0.001	< 0.001
S × M	0.005	0.212	0.551	0.141
P × M	0.574	0.187	0.792	0.285
S × R	0.129	0.001	< 0.001	0.474
P × R	0.230	0.169	0.002	0.993
M × R	< 0.001	< 0.001	0.591	0.178
S × P × M	< 0.001	< 0.001	0.277	0.019
S × P × R	0.421	0.045	0.019	0.938
S × M × R	0.034	< 0.001	< 0.001	0.020
P × M × R	0.196	< 0.001	0.086	0.203
S × P × M × R	0.671	< 0.001	0.132	0.09

Table 3.9. Available nitrogen, phosphorus, potassium and sulphur in soils with PSDF types.

	Clay Loam	Loam	Sand
Nitrogen (mg kg^{-1})			
PSDF 1	2.8 (0.2)	3.8 (0.3)	2.1 (0.1)
PSDF 2	1.9 (0.0)	3.4 (0.2)	1.9 (0.0)
Phosphorus (mg kg^{-1})			
PSDF 1	5.1 (0.1)	37.4 (0.5)	24.2 (0.4)
PSDF 2	4.9 (0.0)	31.4 (1.7)	20.8 (1.1)
Potassium (mg kg^{-1})			
PSDF 1	393.7 (15.5)	661.7 (33.4)	166.3 (17.7)
PSDF 2	342.3 (14.2)	456.9 (23.0)	139.3 (19.1)
Sulphur (mg kg^{-1})			
PSDF 1	154.5 (11.9)	71.8 (4.1)	19.5 (1.3)
PSDF 2	85.4 (6.1)	34.9 (2.0)	11.8 (1.4)

Means within treatments of the same soil and PSDF type followed by standard errors.

Table 3.10. Cation exchange capacity, total carbon, total nitrogen and total organic carbon in soils with PSDF after 4 months.

		Cation Exchange Capacity (meq 100g^{-1})	Total Carbon (%)	Total Nitrogen (%)	Total Organic Carbon (%)
Soil	Clay Loam	23.8 ± 0.3	2.3 ± 0.0	0.1 ± 0.0	1.8 ± 0.0
	Loam	24.4 ± 0.2	2.6 ± 0.0	0.2 ± 0.0	2.5 ± 0.0
	Sand	4.5 ± 0.1	0.1 ± 0.0	0.0 ± 0.0	0.1 ± 0.0
PSDF	PSDF 1	18.4 ± 0.9	1.6 ± 0.1	0.1 ± 0.0	1.4 ± 0.1
	PSDF 2	16.7 ± 0.9	1.7 ± 0.1	0.1 ± 0.0	1.5 ± 0.1
Application Method	Incorporated	17.9 ± 0.9	1.7 ± 0.1	0.1 ± 0.0	1.5 ± 0.1
	Sprayed	17.2 ± 0.9	1.7 ± 0.1	0.1 ± 0.0	1.5 ± 0.1
Rate (m^3 ha^{-1})	0	16.8 ± 1.9	1.6 ± 0.2	0.1 ± 0.0	1.4 ± 0.2
	10	17.6 ± 1.7	1.6 ± 0.2	0.1 ± 0.0	1.4 ± 0.2
	20	17.3 ± 1.6	1.7 ± 0.2	0.1 ± 0.0	1.6 ± 0.2
	30	17.5 ± 1.5	1.7 ± 0.2	0.1 ± 0.0	1.5 ± 0.2
	40	17.5 ± 1.6	1.6 ± 0.2	0.1 ± 0.0	1.4 ± 0.2
	60	18.0 ± 1.7	1.7 ± 0.2	0.1 ± 0.0	1.5 ± 0.2
	120	17.6 ± 1.5	1.7 ± 0.2	0.1 ± 0.0	1.5 ± 0.2

PSDF types = spent once (PSDF 1), spent twice (PSDF 2).
Means within a column within treatments of same variable followed by standard errors.

Table 3.11. P values for cation exchange capacity, total carbon, total nitrogen and total organic carbon in soils with PSDF after 4 months.

	Cation Exchange Capacity (meq 100g^{-1})	Total Carbon (%)	Total Nitrogen (%)	Total Organic Carbon (%)
Soil (S)	< 0.001	< 0.001	< 0.001	< 0.001
PSDF (P)	< 0.001	< 0.001	0.239	< 0.001
Application Method (M)	< 0.001	0.822	0.001	0.328
Rate (R, m^3 ha^{-1})	0.109	0.087	0.015	0.058
S × P	< 0.001	0.002	< 0.001	< 0.001
S × M	< 0.001	0.592	0.467	0.145
P × M	0.792	0.806	0.068	0.841
S × R	0.016	0.096	0.012	0.137
P × R	0.080	0.188	0.234	0.144
M × R	0.014	< 0.001	< 0.001	0.004
S × P × M	0.034	0.780	0.808	0.796
S × P × R	< 0.001	0.011	0.204	0.002
S × M × R	0.091	0.001	< 0.001	0.001
P × M × R	0.065	0.135	< 0.001	0.087
S × P × M × R	0.396	0.116	< 0.001	0.095

Table 3.12. Cation exchange capacity, total carbon, nitrogen and organic carbon in soils with PSDF types.

	Clay Loam	Loam	Sand
Cation exchange capacity (meq $100g^{-1}$)			
PSDF 1	25.4 (0.4)	24.9 (0.2)	4.9 (0.2)
PSDF 2	22.2 (0.1)	23.8 (0.2)	4.1 (0.1)
Total carbon (%)			
PSDF 1	2.2 (0.03)	2.5 (0.02)	0.1 (0.00)
PSDF 2	2.3 (0.08)	2.7 (0.03)	0.1 (0.01)
Total nitrogen (%)			
PSDF 1	0.04 (0.00)	0.24 (0.01)	0.02(0.00)
PSDF 2	0.10 (0.01)	0.18 (0.01)	0.04 (0.00)
Total organic carbon (%)			
PSDF 1	1.8 (0.04)	2.4 (0.03)	0.1 (0.00)
PSDF 2	1.9 (0.08)	2.6 (0.03)	0.1 (0.01)

PSDF types = spent once (PSDF 1), spent twice (PSDF 2).
Means within treatments of same soil and PSDF type followed by standard errors.

Table 3.13. Cation exchange capacity, total carbon, nitrogen and organic carbon in soils with PSDF application methods and rates.

PSDF Rate	10 (m^3 ha^{-1})	20 (m^3 ha^{-1})	30 (m^3 ha^{-1})	40 (m^3 ha^{-1})	60 (m^3 ha^{-1})	120 (m^3 ha^{-1})
Cation exchange capacity (meq $100g^{-1}$)						
Incorporated	18.2 (2.5)	17.6 (2.3)	17.8 (2.3)	18.1 (2.3)	18.2 (2.4)	17.4 (2.1)
Sprayed	16.9 (2.2)	16.9 (2.2)	17.2 (2.1)	16.9 (2.2)	17.8 (2.3)	17.7 (2.2)
Total carbon (%)						
Incorporated	1.6 (0.3)	1.8 (0.3)	1.7 (0.3)	1.6 (0.3)	1.6 (0.3)	1.7 (0.3)
Sprayed	1.7 (0.3)	1.7 (0.3)	1.6 (0.3)	1.7 (0.3)	1.8 (0.3)	1.6 (0.3)
Total nitrogen (%)						
Incorporated	0.1 (0.02)	0.1 (0.02)	0.1 (0.02)	0.1 (0.02)	0.1 (0.02)	0.1 (0.02)
Sprayed	0.1 (0.02)	0.1 (0.02)	0.1 (0.02)	0.1 (0.02)	0.1 (0.03)	0.1 (0.03)
Total organic carbon (%)						
Incorporated	1.4 (0.2)	1.6 (0.3)	1.5 (0.2)	1.4 (0.2)	1.4 (0.2)	1.5 (0.3)
Sprayed	1.5 (0.3)	1.5 (0.3)	1.4 (0.2)	1.5 (0.2)	1.6 (0.3)	1.5 (0.2)

PSDF types = spent once (PSDF 1) and spent twice (PSDF 2).
Means within treatments of same soil and PSDF type followed by standard errors.

Table 3.14. Canadian Council of Ministers of the Environment (CCME) metals in soil with and without PSDF and AENV upper limits.

Element (mg kg^{-1})	Clay Loam			Loam			Sand			AENV Soil Upper Limits	
	Control	Spent Once	Spent Twice	Control	Spent Once	Spent Twice	Control	Spent Once	Spent Twice	Agricultural Land Use	Natural Land Use
Boron	1.0	1.1	0.9	0.5	0.5	0.6	0.0	0.0	0.0	2	2
Mercury	0.1	0.1	0.1	0.0	0.0	0.0	0.0	0.0	0.0	6.6	12
Antimony	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	20	20
Arsenic	6.2	5.5	5.4	6.4	7.2	6.0	1.7	1.7	1.8	17	17
Barium	212	2344	273	220	2980	317	65	1045	124	750	750
Beryllium	0.8	0.8	0.8	0.7	0.7	0.6	0.1	0.2	0.2	5	5
Cadmium	0.3	0.3	0.2	0.4	0.4	0.4	0.1	0.0	0.1	1.4	3.8
Chromium	17.0	18.5	17.8	23.7	24.5	21.3	3.8	3.9	4.1	64	64
Cobalt	11.5	9.0	8.6	9.3	10.0	8.9	2.3	2.3	2.4	20	20
Copper	24.7	27.4	28.5	21.8	21.7	22.2	3.2	3.2	3.8	63	63
Lead	13.2	13.1	11.8	9.9	11.3	9.8	2.4	2.9	2.7	70	70
Molybdenum	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.0	4	4
Nickel	49.9	25.0	24.5	22.0	23.5	17.3	4.4	5.1	4.6	50	50
Selenium	0.6	0.5	0.5	0.8	0.8	0.9	0.5	0.0	0.2	1	1
Silver	0.2	0.2	0.2	0.2	0.2	0.2	0.1	0.0	0.0	20	20
Thallium	0.3	0.3	0.3	0.2	0.3	0.2	0.0	0.1	0.0	1	1
Tin	0.2	0.1	0.0	0.0	0.0	0.1	0.8	1.6	0.8	5	5
Uranium	2.0	2.2	1.9	1.0	1.0	0.9	0.0	0.0	0.0	23	23
Vanadium	25.4	28.4	27.2	42.8	42.7	38.4	8.3	8.2	8.8	130	130
Zinc	53.3	56.1	53.6	65.5	70.3	66.9	15.8	17.1	15.8	200	200

AENV = Alberta Environment and Sustainable Resource Development.
All the ceiling limits are based on the Alberta Tier 1 Soil and Groundwater Remediation Guidelines (2009).

Table 3.15. Plant emergence, death and biomass in soils with PSDF.

		Emergence (plant pot^{-1})	Dead (plant pot^{-1})	Above Ground Biomass (g pot^{-1})	Below Ground Biomass (g pot^{-1})
Soil	Clay Loam	6.9 ± 0.3	0.4 ± 0.1	1.8 ± 0.1	0.3 ± 0.0
	Loam	11.3 ± 0.2	0.2 ± 0.0	3.3 ± 0.1	0.5 ± 0.0
	Sand	11.4 ± 0.2	0.3 ± 0.1	1.5 ± 0.1	0.3 ± 0.0
PSDF	PSDF 0	11.0 ± 0.2	0.3 ± 0.1	1.9 ± 0.1	0.4 ± 0.0
	PSDF 1	10.5 ± 0.3	0.4 ± 0.1	1.9 ± 0.1	0.4 ± 0.0
	PSDF 2	8.1 ± 0.3	0.2 ± 0.0	2.8 ± 0.1	0.3 ± 0.0
Application Method	Incorporated	9.8 ± 0.2	0.3 ± 0.0	2.3 ± 0.1	0.4 ± 0.0
	Sprayed	9.9 ± 0.2	0.3 ± 0.0	2.1 ± 0.1	0.4 ± 0.0
Rate (m^3 ha^{-1})	0	10.7 ± 0.6	0.0 ± 0.0	2.1 ± 0.2	0.3 ± 0.0
	10	10.3 ± 0.4	0.2 ± 0.1	2.1 ± 0.1	0.3 ± 0.0
	20	10.3 ± 0.4	0.2 ± 0.1	2.2 ± 0.1	0.4 ± 0.0
	30	10.2 ± 0.4	0.3 ± 0.1	2.2 ± 0.1	0.4 ± 0.0
	40	10.1 ± 0.4	0.3 ± 0.1	2.3 ± 0.2	0.4 ± 0.0
	60	9.8 ± 0.4	0.3 ± 0.1	2.4 ± 0.2	0.4 ± 0.0
	120	8.5 ± 0.4	0.5 ± 0.1	2.2 ± 0.1	0.3 ± 0.0

PSDF types = raw (0), spent once (1), spent twice (2).
Means within a column within treatments of same variable followed by standard errors.

Table 3.16. P values for plant emergence, death and biomass in soils with PSDF.

	Emergence (plant pot^{-1})	Dead (plant pot^{-1})	Above Ground Biomass (g pot^{-1})	Below Ground Biomass (g pot^{-1})
Soil (S)	< 0.001	0.003	< 0.001	< 0.001
PSDF (P)	< 0.001	0.109	< 0.001	< 0.001
Application Method (M)	0.704	0.550	0.064	0.357
Rate (R, m^3 ha^{-1})	< 0.001	0.158	0.358	0.009
S × P	0.169	0.005	< 0.001	< 0.001
S × M	0.558	0.001	0.626	0.057
P × M	< 0.001	0.541	0.158	0.748
S × R	0.320	0.198	0.017	0.001
P × R	0.325	0.058	0.689	0.082
M × R	0.097	0.142	0.293	0.722
S × P × M	< 0.001	0.699	0.280	< 0.001
S × P × R	0.323	0.459	0.038	< 0.001
S × M × R	0.443	0.455	0.185	0.046
P × M × R	0.644	0.345	0.523	0.650
S × P × M × R	0.041	0.096	0.625	0.385

Table 3.17. Plant emergence, death and biomass in soils with PSDF types.

	Clay Loam	Loam	Sand
Emergence (plant pot^{-1})			
PSDF 0	8.6 (0.4)	12.3 (0.3)	12.2 (0.3)
PSDF 1	7.2 (0.4)	11.9 (0.3)	12.3 (0.4)
PSDF 2	4.9 (0.4)	9.7 (0.4)	9.7 (0.4)
Dead (plant pot^{-1})			
PSDF 0	0.5 (0.1)	0.1 (0.0)	0.2 (0.1)
PSDF 1	0.6 (0.1)	0.2 (0.1)	0.4 (0.1)
PSDF 2	0.2 (0.1)	0.3 (0.1)	0.3 (0.1)
Above ground biomass (g pot^{-1})			
PSDF 0	1.8 (0.1)	2.7 (0.1)	1.2 (0.0)
PSDF 1	1.7 (0.1)	2.9 (0.1)	1.2 (0.1)
PSDF 2	2.0 (0.3)	4.3 (0.2)	2.2 (0.1)
Below ground biomass (g pot^{-1})			
PSDF 0	0.4 (0.0)	0.5 (0.0)	0.1 (0.0)
PSDF 1	0.4 (0.0)	0.4 (0.0)	0.3 (0.0)
PSDF 2	0.2 (0.0)	0.4 (0.0)	0.3 (0.0)

PSDF types = raw (0), spent once (1), spent twice (2).
Means within treatments of same soil and PSDF type followed by standard errors.

Table 3.18. Plant emergence, death and biomass in clay, loam and sand soil with PSDF application rates.

	0 (m^3 ha^{-1})	10 (m^3 ha^{-1})	20 (m^3 ha^{-1})	30 (m^3 ha^{-1})	40 (m^3 ha^{-1})	60 (m^3 ha^{-1})	120 (m^3 ha^{-1})
Emergence (plant pot^{-1})							
Clay Loam	8.3 (1.1)	6.9 (0.5)	7.5 (0.7)	7.0 (0.7)	7.2 (0.7)	6.8 (0.5)	6.0 (0.7)
Loam	11.0 (0.7)	12.1 (0.5)	11.9 (0.4)	11.7 (0.5)	11.0 (0.5)	10.7 (0.5)	10.2 (0.5)
Sand	12.9 (0.5)	11.9 (0.6)	11.5 (0.6)	11.8 (0.6)	12.1 (0.5)	11.9 (0.5)	9.3 (0.5)
Dead (plant pot^{-1})							
Clay Loam	0.0 (0.0)	0.3 (0.1)	0.3 (0.2)	0.5 (0.1)	0.7 (0.2)	0.2 (0.1)	0.7 (0.2)
Loam	0.1 (0.0)	0.2 (0.1)	0.2 (0.1)	0.2 (0.1)	0.1 (0.0)	0.2 (0.1)	0.2 (0.1)
Sand	0.5 (0.3)	0.2 (0.1)	0.1 (0.0)	0.4 (0.2)	0.1 (0.0)	0.4 (0.2)	0.5 (0.2)
Above ground biomass (g pot^{-1})							
Clay Loam	1.9 (0.2)	1.8 (0.1)	1.8 (0.1)	1.9 (0.3)	2.1 (0.4)	1.8 (0.1)	1.5 (0.1)
Loam	2.7 (0.2)	2.9 (0.1)	3.2 (0.3)	3.2 (0.2)	3.1 (0.1)	3.8 (0.4)	3.6 (0.2)
Sand	1.7 (0.4)	1.5 (0.1)	1.6 (0.1)	1.4 (0.1)	1.7 (0.2)	1.5 (0.1)	1.4 (0.1)
Below ground biomass (g pot^{-1})							
Clay Loam	0.3 (0.1)	0.4 (0.0)	0.4 (0.0)	0.4 (0.0)	0.3 (0.0)	0.3 (0.0)	0.2 (0.0)
Loam	0.4 (0.0)	0.4 (0.0)	0.4 (0.0)	0.4 (0.0)	0.5 (0.0)	0.5 (0.0)	0.4 (0.0)
Sand	0.3 (0.0)	0.2 (0.0)	0.3 (0.0)	0.3 (0.0)	0.3 (0.0)	0.3 (0.0)	0.3 (0.0)

Means within treatments of same soil and PSDF type followed by standard errors.

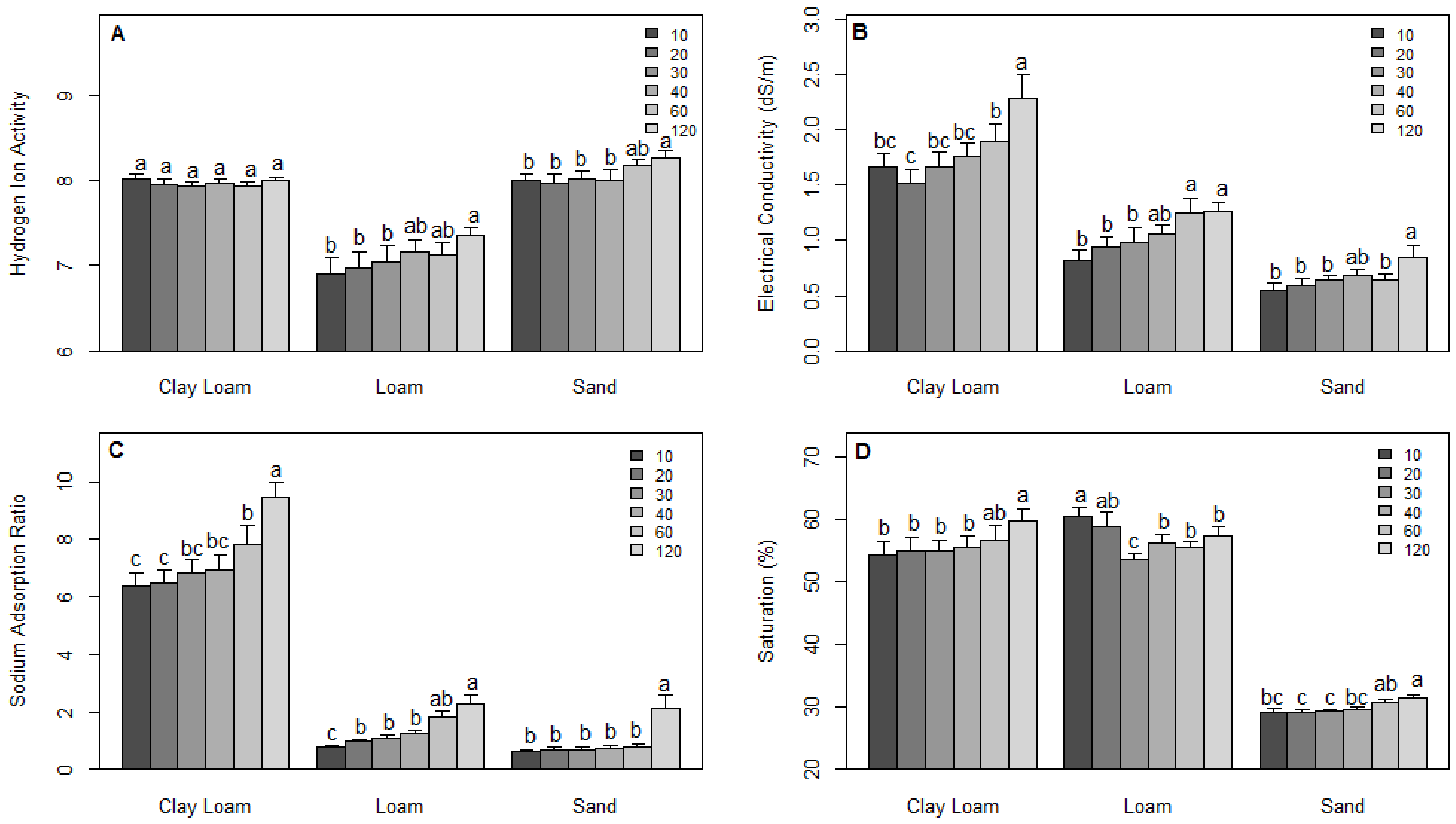

Figure 3.1. Soil pH (A), electrical conductivity (B), sodium adsorption ratio (C) and base saturation (D) in different soil type and PSDF application rates (m^3 ha^{-1}). Error bars represent standard error. Letters indicate significant differences between PSDF application rates in the same soil.

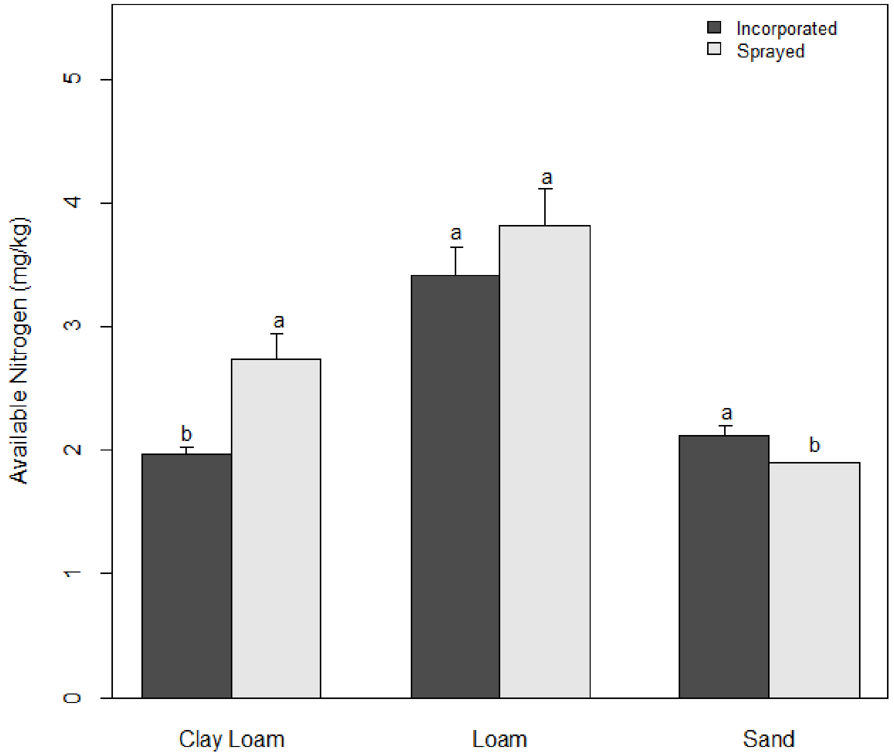

Figure 3.2. Available nitrogen in clay loam, loam and sand soil with incorporated or sprayed PSDF. Error bars represent standard error. Letters indicate significant differences between incorporated and sprayed PSDF within same soil.

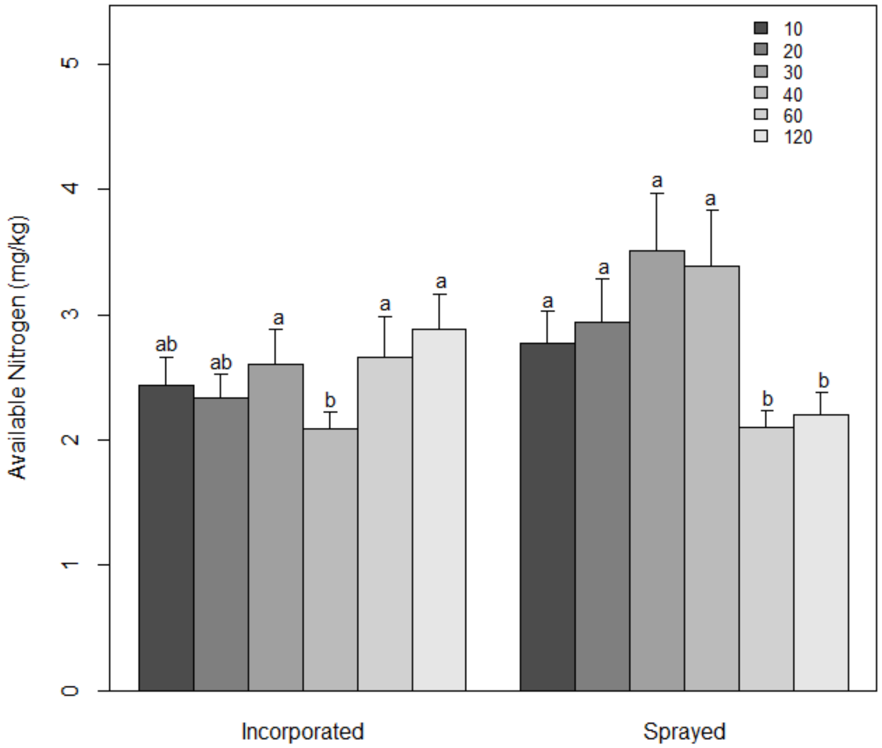

Figure 3.3. Available nitrogen in soil with incorporated or sprayed PSDF at 10, 20, 30, 40, 60 and 120 m^3 ha^{-1}. Error bars represent standard error. Letters indicate significant differences between PSDF rates with the same application method.

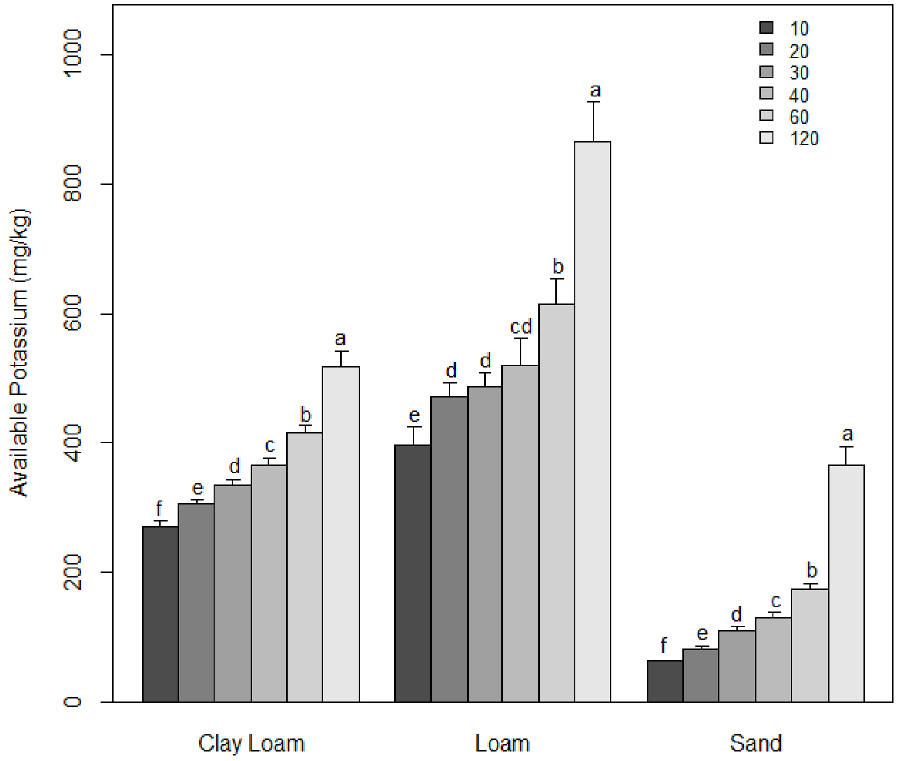

Figure 3.4. Available potassium in clay loam, loam and sand soil with PSDF at 10, 20, 30, 40, 60 and 120 m^3 ha^{-1}. Error bars represent standard error. Letters indicate significant differences between PSDF application rates in the same soil.

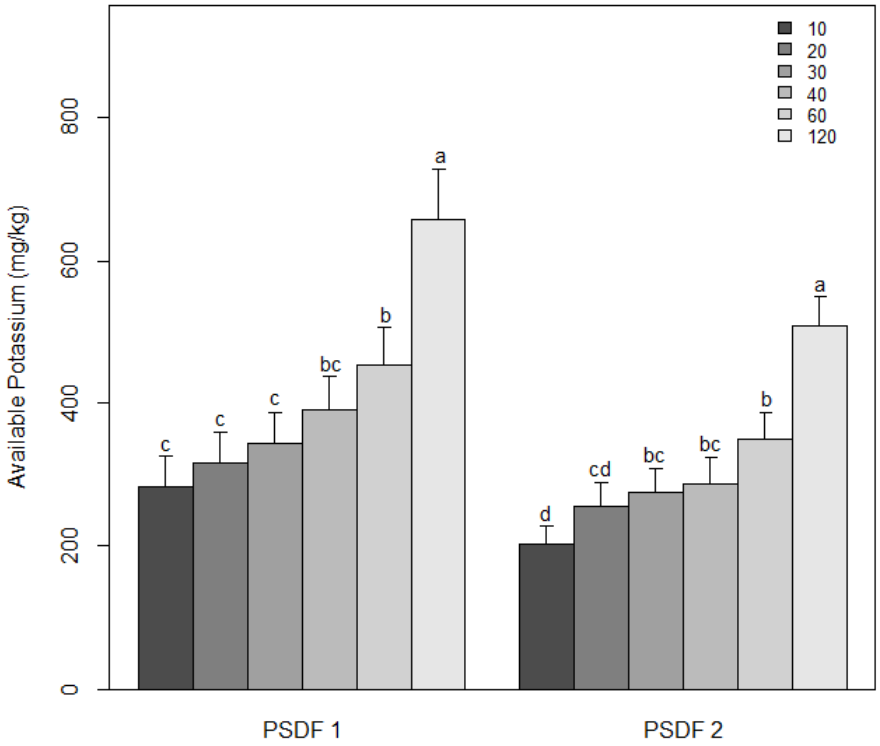

Figure 3.5. Available potassium with PSDF at 10, 20, 30, 40, 60 and 120 m^3 ha^{-1}. PSDF 1 = spent once. PSDF 2 = spent twice. Error bars represent standard error. Letters indicate significant differences between application rates in the same PSDF type.

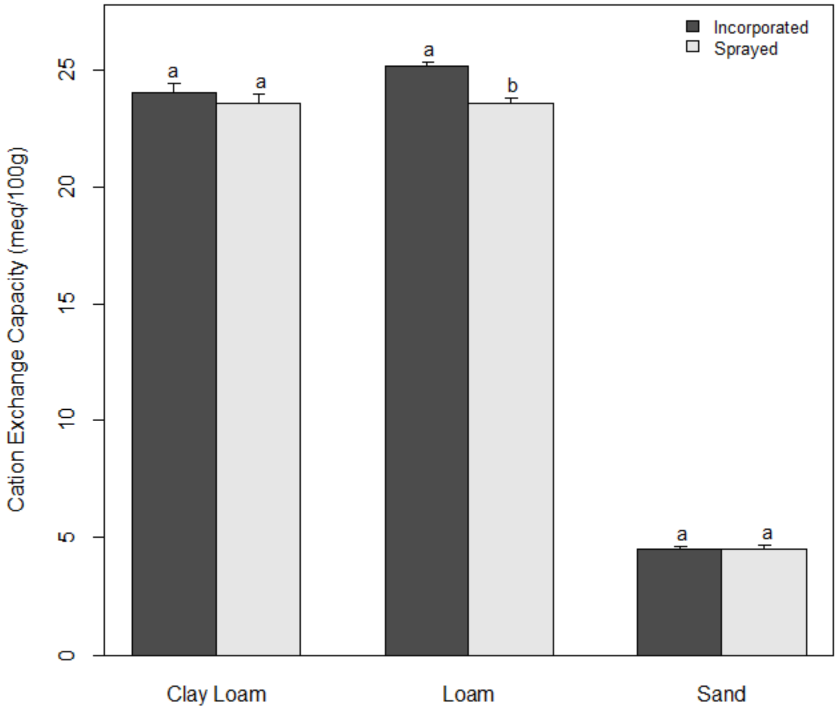

Figure 3.6. Cation exchange capacity with incorporated and sprayed PSDF in clay loam, loam and sand soils. Error bars represent standard error. Letters indicate significant differences between incorporated and sprayed PSDF in the same soil.

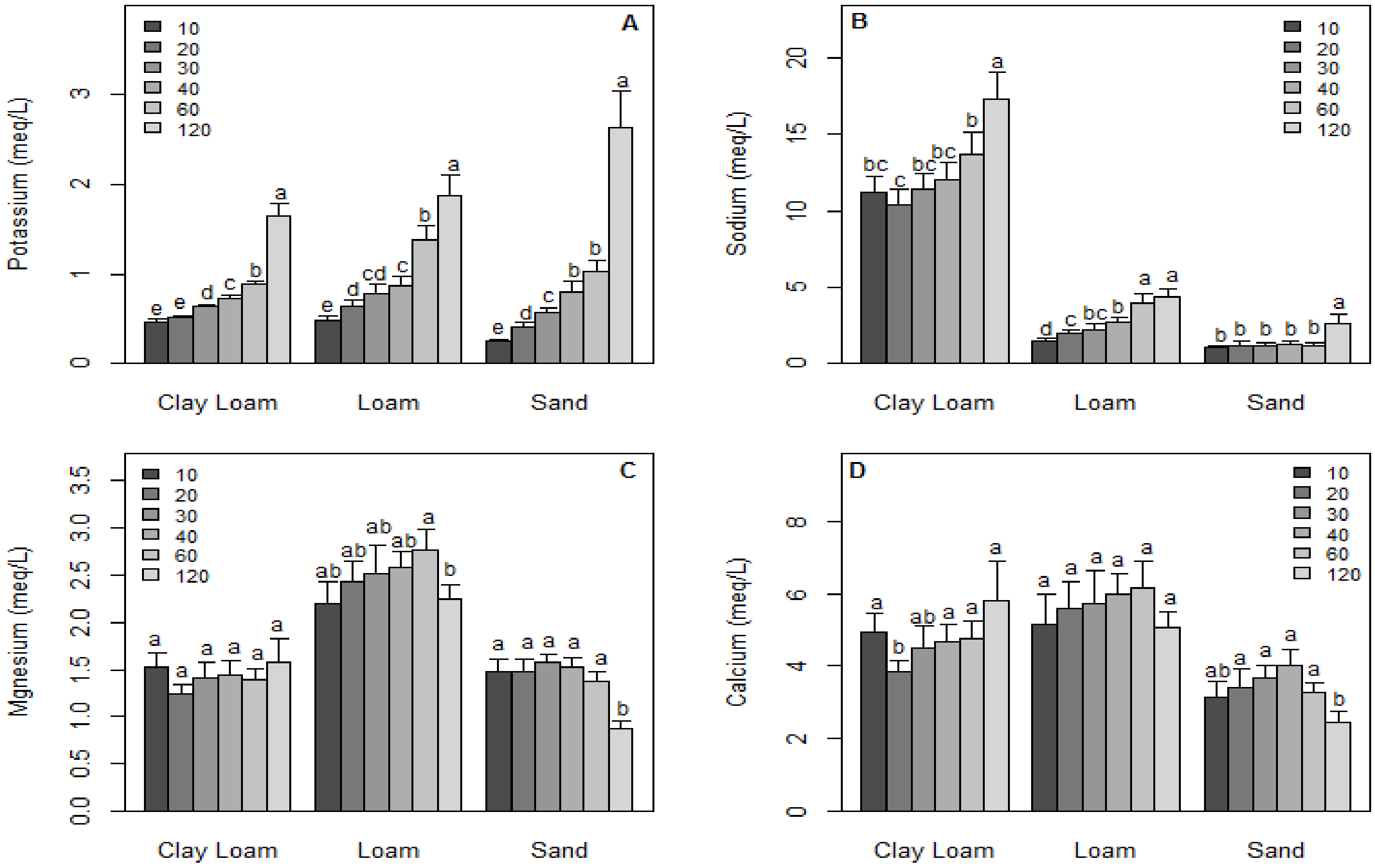

Figure 3.7. Soluble potassium (A), sodium (B), magnesium (C) and calcium (D) with different soil types and PSDF application rates (m^3 ha^{-1}). Error bars are standard error. Letters indicate significant differences with PSDF application rates in same soil.

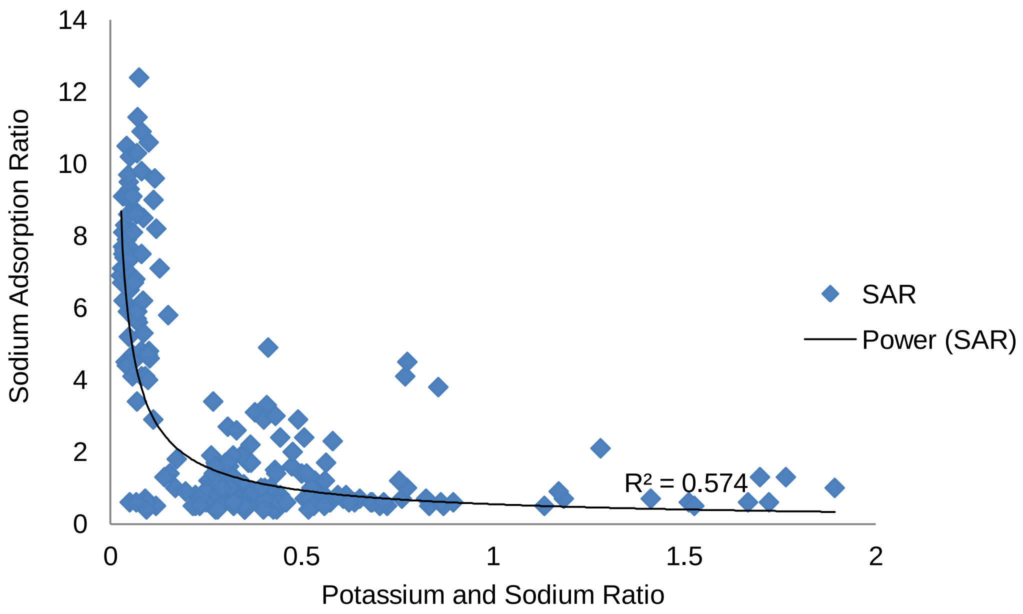

Figure 3.8. Correlation between sodium adsorption ratio and potassium and sodium ratio from treatments with PSDF. The solid line is the correlation trend line.

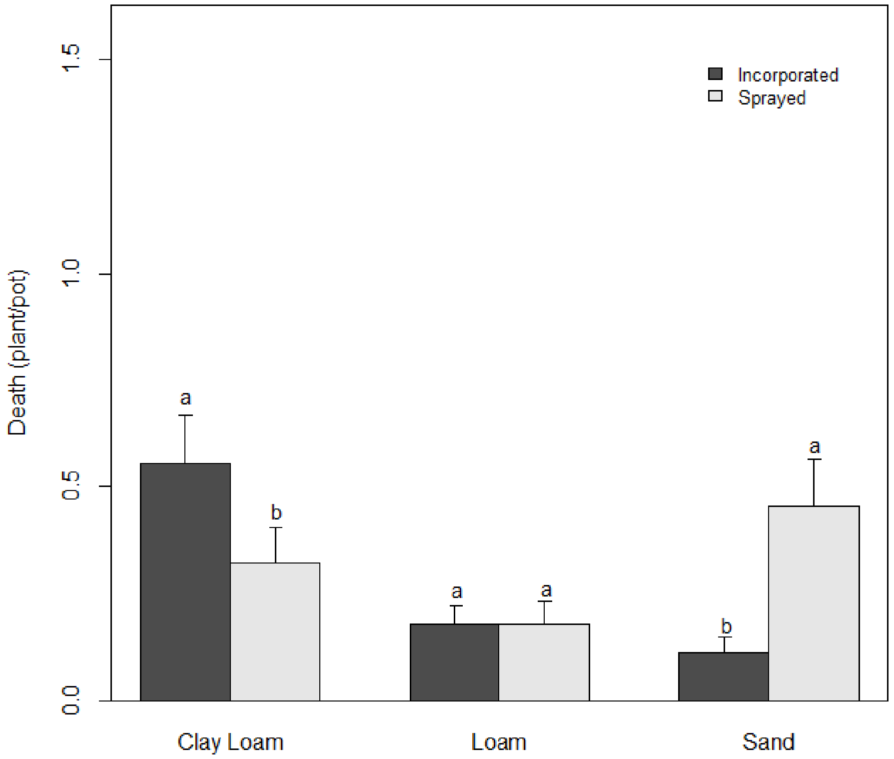

Figure 3.9. Plant death in each soil with incorporated and sprayed PSDF. Error bars represent standard error. Letters indicate significant differences between PSDF application methods in the same soil.

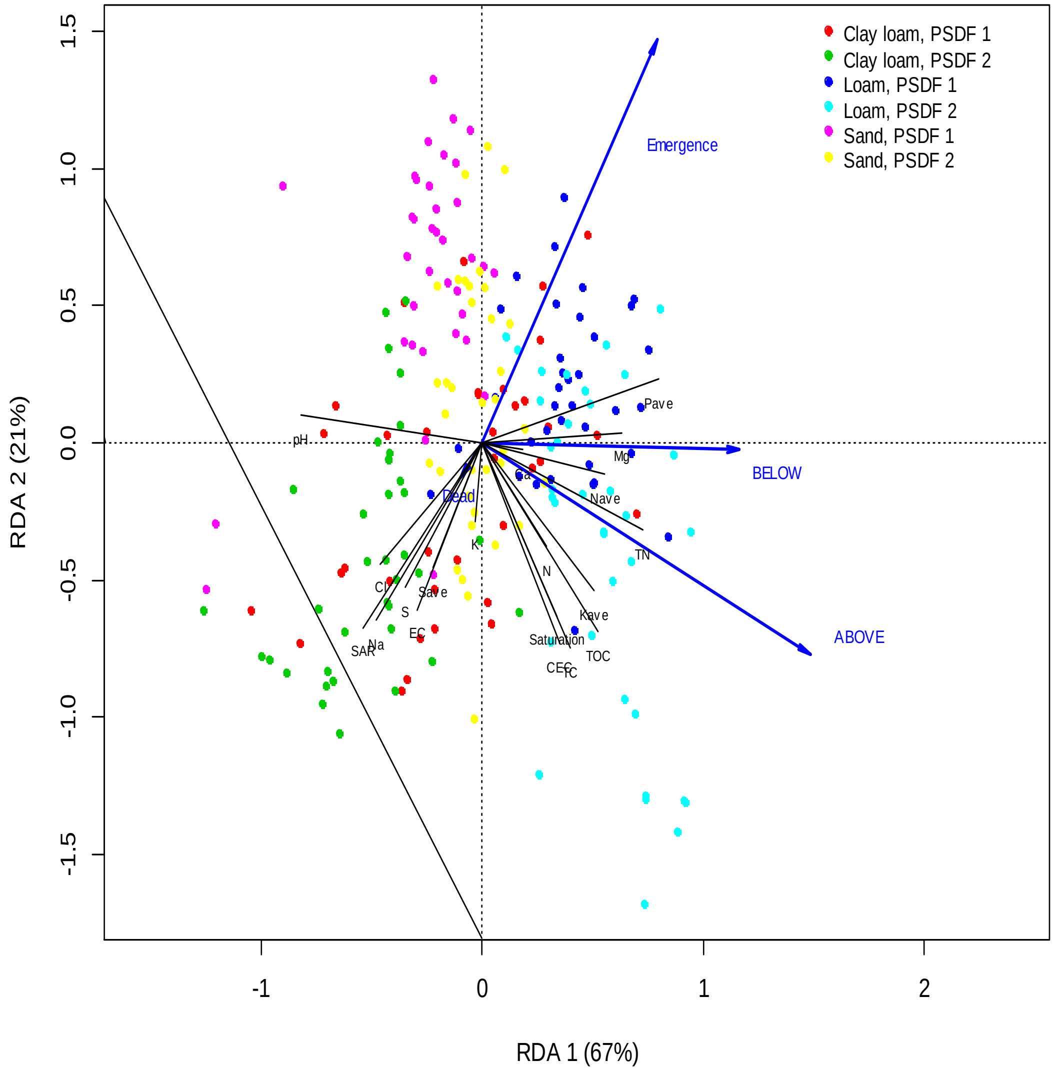

Figure 3.10. Redundancy analysis (RDA) of plants (emergence, death, above ground biomass, below ground biomass) versus soil chemistry (salinity, nutrients, exchangeable ions) of potassium silicate drilling fluid (PSDF) with soil. Types of soil and PSDF are shown in legend. Above = above ground biomass. Below = below ground biomass. Dead = plant death. Nave = available nitrogen. Pave = available phosphorus. Kave = available potassium. Save = available sulphur. TC = total carbon. TOC = total organic carbon. TN = total nitrogen. pH = hydrogen ion activity. EC = electrical conductivity. SAR = sodium adsorption ratio. CEC = cation exchange capacity. K = soluble potassium. Na = soluble sodium. Mg = soluble magnesium. Ca = soluble calcium. Cl = soluble chloride. S = soluble sulphate. N = soluble nitrate.

CHAPTER 4. SOIL PHYSICAL AND CHEMICAL RESPONSES TO POTASSIUM SILICATE DRILLING FLUID IN A LABORATORY COLUMN STUDY

1. Introduction

Drilling fluid is a lubricant used while drilling oil and natural gas wells and in exploration drilling rigs. Water based drilling fluids typically consist of bentonite and various additives mixed with water. During rotary drilling, an increased volume of waste fluids results from disintegration of rock cuttings during transport to the surface and limited efficiency of cuttings removal by the solids control separators (Wojtanowicz 2008). The spent drilling fluid properties vary with additives periodically added to enhance properties that optimize and improve drilling (United States Army Corps of Engineers 2001).

In recent years, land disposal of drilling fluids has been a growing concern due to the potential environmental effects from large volumes of drilling wastes with large amounts of potential pollutants. The volume of drilling wastes to drill a hole is approximately three times the volume of the hole (United States Army Corps of Engineers 2001) and a typical shallow gas well (250-650 m deep) in Alberta will generate around 68 m^3 of spent drilling fluid (Zvomuya et al. 2009). In Alberta, approximately 213,000 well sites were active up to December 2012 (Alberta Environment and Sustainable Resource Development 2012). From these well sites an estimated 14.5 million m^3 of drilling wastes in Alberta will be disposed.

Few studies addressed the impact of spent drilling fluids disposal on soil-plant-water systems. Some found high soluble salts, heavy metals and petroleum residue content of used drilling fluids were detrimental to soil quality and plant growth (Zvomuya et al. 2011, Wojtanowicz 2008, Zvomuya et al. 2008, McFarland et al. 1994, McFarland et al. 1992, Nelson et al. 1984, Miller et al. 1980). Other researchers found positive or no impacts from drilling fluids at low rates in coarse texture soils native to arid regions due to pH increase, potential micronutrient addition or improved soil physical properties (Bauder et al. 2005, Bauder et al. 1999, Macyk et al. 1990, Lesky et al. 1989, Moseley Jr. 1983). Differences resulted from waste properties and disposal method and location.

In western Canada, environmental regulations stringently limit the range of disposal approaches for water based drilling fluids, with criteria for maximum disposal rate and maximum acceptable increases in soil salinity, sodicity, sodium and nitrogen loading (Alberta Energy Resources Conservation Board 2012, Saskatchewan Ministry of Energy and Resources 2011, British Columbia Oil and Gas Commission 2006). Due to lack of information, advanced gel chemical muds (potassium silicate, potassium sulphate), a kind of water based drilling fluid system, were not included in the Alberta drilling waste regulatory framework for land application before 2012, and the Energy Resources Conservation Board required site specific approval prior to disposal (Energy Resources Conservation Board 2007). The Energy Resources Conservation Board is now developing new criteria for disposal of advanced gel chemical muds.

Potassium silicate drilling fluids (PSDF), a recent advanced gel drilling fluid system with high drilling performance, has been developed to promote waste volume minimization and decrease disposal cost. PSDF is considered an environmentally acceptable alternative to traditional drilling fluids with silicate polymeric ions and potassium salts replacing lignite and sodium salts (Table 4.1). PSDF has high levels of potassium. Potassium concentrations in PSDF before drilling operations and after range from 6,900 to 21,700 mg L^{-1}, based on laboratory measurements (Table 4.2). Little is known about potential effects of PSDF on plants and soil. Potassium accumulation in soil is seen by regulators and industries as a potential problem because of the effect it might have on salt accumulation and soil structure. However, results from recent studies in the greenhouse indicated that, even 120 m^3 ha^{-1} in loam soil did not cause significant risks for plant establishment and development (Chapters 2, 3). Mechanisms of these effects are not fully understood in soil and leachate.

Zvomuya et al. (2009) found reduced hydraulic conductivity with drilling fluid at a rate of 80 m^3 ha^{-1}. Available zinc increased but not detrimentally, with drilling fluids incorporated in sandy soil with a volume ratio of 1:8 in columns (Bates 1988). Amending 3 % water based drilling fluid with calcium salts, or cationic organic polymers in sandy loam soil columns, increased leaching of excessive salts without mobilizing chromium and contaminating ground water (Kim 2003, Kim et al. 2002, Kim 1998).

Data on effects of high concentrations of potassium on soil physical properties are limited. Chen et al. (1983) found potassium can cause a reduction in relative hydraulic conductivity from dispersion of clay microaggregates and their rearrangement in situ to form a dense network of particles with smaller pores. Some researchers found the effect of exchangeable potassium was not as detrimental as sodium addition on infiltration. Similar results showed greater soil stability in the presence of potassium relative to sodium (Arienzo et al. 2012).

This research was part of a larger study that examined the effects of PSDF on soil properties and plants establishment and productivity. In this laboratory column experiment, changes in soil and hydrologic properties following PSDF application were studied. Research objectives were as follows.

- To evaluate effects of different rates of PSDF on soil physical and chemical properties of different texture soils.
- To evaluate the current industry potential method for spraying versus incorporating PSDF with topsoil on soil and leachate properties.
- To investigate whether increasing drilling fluid use affected nutrient concentrations, salinity and sodicity.
- To examine levels of retention of PSDF components with depth following irrigation through columns to leachate.

2. Materials And Methods

2.1 Experimental Design

A four month, laboratory column experiment was established in a randomized complete block design, with treatments replicated 5 times. Treatments represented reclamation scenarios under various end land uses, such as cultivation and pastures. Soils and drilling fluids were the same as those used in previous greenhouse experiments in this research program (Chapters 2, 3). Three soil texture treatments were sand, loam and clay loam, covering the wide range of soils with potential for reclamation using drilling fluids in Alberta. Three types of potassium silicate drilling fluids were raw, spent once and spent twice. Two application treatments were spraying PSDF on the soil surface and spraying it on the soil surface followed by incorporation into the soil, approximating potential field application methods. Four PSDF application rates were 20, 40, 60 and 120 $m^3 ha^{-1}$. Total number of columns was 225.

2.2 Laboratory Procedures

Transparent columns were constructed of acrylic; they were 40 cm long with 10 cm diameter and 78.54 cm^2 in area. Plastic screen was secured to the bottom of the column with a cable tie. Before filling with substrate, 2 cm of clean sand was added to the bottom to prevent soil from moving through the plastic screen.

Columns were weighed and masses recorded (Mc). Soils were passed through a 2 mm sieve and air dried to obtain a homogeneous sample. Each column required approximately 2,300 g of sand soil or 1,650 g of loam soil. A flexible plastic hose was taped to the bottom of a large funnel with a 1.5 cm bottom opening. The hose was placed in the column, then the funnel was gently filled with substrate. Once full, the hose was slowly lifted to release soil then moved around to place 20 cm of soil. When columns were filled with the requisite amount of soil, column sides were gently tapped with a rubber mallet, starting at the bottom, until substrate settled to approximately 20 cm depth in the column. Substrate depth was measured (h).

Soils in columns were treated with raw (PSDF 0), spent once (PSDF 1) and spent twice (PSDF 2) PSDF at four application rates by spraying evenly on the soil surface or incorporating into the upper 10 cm of soil with a fork. Columns with PSDF were left to settle for 48 hours. Filled columns were weighed and masses were recorded (Mc+s+p). Calculations for bulk density (ρbs) and porosity (fs) of soils with PSDF were completed as follows. Bulk Density (ρbs) = mass of dry soil (Mc+s+p – Mc) / volume of soil ($\pi r^2 h$). Porosity (fs) = 1 – ($\rho bs\ \rho s^{-1}$) (assume ρs = 2.65 $Mg\ m^{-3}$).

Columns were placed vertically in a tray of distilled water and wet for 48 hours by allowing water to rise through capillary action to saturate the soil. At saturation the soil surface was glistening. Columns were weighed with saturated soil and masses recorded (Mc+wsp). Volume of water held in the soil pore space at saturation based on measured porosity was calculated. Treatments with clay loam soil were discarded after several unsuccessful attempts to bring the soil in the columns to saturation. The soil would not transmit water, even after 1 month of wetting, from both the top and the bottom of the column.

Large ring clamps were attached to metal stands and columns clamped in place on the stand. Saturated columns rested on large ring clamps, their length was recorded as length of soil. Hydraulic conductivity was determined by the falling head method (Reynolds 2008). Water was slowly added to columns to 10 cm height (H_0). Water dripped from the columns through the funnel into graduated cylinders and the events were timed starting from time zero (t_0). Time (t_1) for each 2 cm height of ponded water was recorded and height of water calculated, over the soil surface (H_1). From this point throughout the experiment, calculating stopped if $H_1 < 0$. Hydraulic conductivity K was calculated as follows. $K = [L/(t_1 – t_0)] \bullet \ln [(L+ H_0) /(L+ H_1)]$ where: t_0 = beginning time (s), t_1 = recording time (s), L = sample thickness, H_0 = height of water over soil sample at time t_0 (cm), H_1 = height of the water over soil sample at time t_1 (cm).

Leachate was collected immediately from the bottom of each column. At the end of the experiment, columns were weighed (W_1) after 2 days to determine field capacity. The soil cores were divided into two equal 10 cm fractions. Leachate and soil fraction samples were kept cool in a refrigerator until delivered to Exova Laboratory Group in Edmonton for analyses.

2.3 PSDF, Soil and Leachate Samples Analyses

Raw PSDF was made in the laboratory of the supplier, Marquis Alliance Ltd. Spent drilling fluids were collected from drilling sites using the same PSDF as the raw samples for the experiment. Soils were collected from three locations in Alberta where drilling was active. Prior to experimentation, PSDF was stored in the refrigerator.

At the end of the experiment, three soil samples were randomly taken from each treatment. Due to budget limitations, chemical analyses of soils and leachate with raw PSDF were not determined. Some of the raw PSDF data were available from earlier experiments. Samples were kept cool in a refrigerator until delivered to the laboratory for analyses. Soil samples were sieved to remove large gravel particles then ground to < 2 mm.

Available nitrate (NO_3^-) and ammonium (NH_4^+) were determined by extraction with 2.0 M potassium chloride (KCl) (Carter and Gregorich 2008). Available phosphorus and potassium were determined by modified Kelowna extraction (Ashworth and Mrazek 1995) and available sulphate by extraction with 0.1 M calcium chloride ($CaCl_2$) (McKeague 1978). Cation exchange capacity was determined by exchange with ammonium acetate (NH_4OA_C) at pH 7 (McKeague 1978). Water soluble cations (sodium, calcium, potassium, magnesium), pH, sodium adsorption ratio and electrical conductivity were determined from saturated paste extracts (Carter and Gregorich 2008). Sulphate and chloride were determined by ion chromatography with chemical suppression (Clesceri et al. 1992). Hydrocarbon fractions (F1, F2, F3, F4) were determined by photoionization and electrolytic conductivity detectors (Environmental Protection Agency 1996). Trace metals restricted in concentration by the Canadian Council of Ministers of the Environment (silver, arsenic, barium, beryllium, cadmium, cobalt, chromium, copper, mercury, molybdenum, nickel, lead, antimony, selenium, tin, thallium, uranium, vanadium, zinc) and boron were determined with a nitric acid hotplate digestion method (Environmental Protection Agency 2008). Sand, silt and clay were determined by hydrometer after treatment with calgon (Carter and Gregorich 2008).

Three leachate samples were randomly collected from each treatment and refrigerated until delivered to the laboratory for analyses. Organic carbon was determined by high temperature combustion (American Public Health Association 2005). Total phosphorus, orthophosphate and Kjeldahl nitrogen were determined by automated ascorbic acid reduction (American Public Health Association 2005). Dissolved silicon and trace metals were determined by inductively coupled plasma (American Public Health Association 2005). Leachate pH was determined electrometrically; electrical conductivity and sodium adsorption ratio by conductivity (American Public Health Association 2005). Water soluble cations (sodium, calcium, potassium, magnesium) were determined from saturated paste extracts (Carter and Gregorich 2008). Soluble sulphate was determined by ion chromatography with chemical suppression and chloride was determined by the automated ferricyanide method (American Public Health Association 2005). Hydrocarbon fractions (F1, F2, F3, F4) were determined by photoionization and electrolytic conductivity detectors (Environmental Protection Agency 1996).

2.4 Statistical Analyses

Effects of soil, PSDF types, PSDF application methods and rates were considered fixed. Soil and leachate properties were analyzed with four way factorial analysis of variance (ANOVA) with R software (R Development Core Team 2012). A five way ANOVA with the R software was conducted to analyse soil chemical properties.

Data were checked for violations of normality assumption with Shapiro-Wilk test and equal variances assumption with Bartlett's test. Where significant treatment effects were determined an $LSD_{0.05}$ was calculated to provide a method of comparing means and measures of precision. Statistical differences among means were determined only when the F test was significant.

3. Results

3.1 PSDF and Baseline Soil Properties

PSDF components and properties are presented in Tables 4.1 and 4.2. Selected baseline soil properties prior to PSDF addition are presented in Table 4.3.

3.2 PSDF Impacts on Soil Physical Properties

PSDF had little impact on loam soil texture. PSDF with sand increased clay content to 3.7 % clay by weight from 1.7 % clay without PSDF (data not shown).

Soil, PSDF type and application method were significant for bulk density (Table 4.4). With PSDF, bulk density of loam soil was significantly lower than bulk density of sand soil (Table 4.5). Bulk density was highest with spent once PSDF. Incorporating PSDF resulted in a higher bulk density than when spraying it. The rate of PSDF application had no significant effect on bulk density.

Soil, PSDF type, application method and rate were significant for total porosity (Table 4.4). With PSDF, porosity in loam soil was significantly greater than that of sand soil (Table 4.5). Porosity was slightly higher with raw PSDF than with spent PSDF, and was not affected by spraying or incorporating PSDF. PSDF at all rates significantly decreased porosity relative to no PSDF (Table 4.6).

Soil, PSDF application method and rate significantly affected field capacity (Table 4.4). Loam soil with PSDF had significantly higher field capacity than sand soil (Table 4.5). Field capacity significantly decreased with increasing application rate (Table 4.6). The interaction between application methods and rate was significant (Table 4.4). Incorporating PSDF significantly decreased field capacity with lowest values at rates of 120 m^3 ha^{-1}, but there were no differences among rates with sprayed PSDF (Figure 4.1). Field capacity was positively correlated with total porosity (Figure 4.2).

Soil, PSDF application method, rate and their respective interactions with PSDF type were significant for saturated hydraulic conductivity (Table 4.4). Loam soil with and without spent PSDF had significantly lower hydraulic conductivity than sand soil; these significant differences did not occur with ≥ 40 m^3 ha^{-1} PSDF (Tables 4.5, 4.6). Sand soil with sprayed PSDF had significantly higher hydraulic conductivity than with incorporated PSDF (Figure 4.3). Rate effects were significant with PSDF (Table 4.4). Hydraulic conductivity decreased dramatically in loam and sand soil with PSDF, being highest on sand without PSDF and lowest on loam with 120 m^3 ha^{-1} PSDF (Table 4.6). Hydraulic conductivity in sand with the highest rate of PSDF was 60 % lower than without PSDF (Table 4.6).

3.3 PSDF Impacts on Soil Chemical Properties

Soil depth, type and their interactions with PSDF type or application method were significant for available nitrogen (Table 4.7). Concentration in sand soil was too low to be affected by PSDF (Table 4.8). Loam soil with PSDF had significantly more available nitrogen than sand soil, with more in the lower than upper 10 cm (Table 4.9). Only incorporating PSDF with soil significantly affected available nitrogen at each depth; most was transported from the top of the column to the bottom (Figure 4.4). At the end of the study, available nitrogen in loam soil significantly decreased with increasing PSDF rate, with highest values in the control (Table 4.10).

Soil type and its interactions with soil depth or PSDF application method were significant for available phosphorus (Table 4.7). Loam soil with PSDF had significantly more phosphorus than sand soil (Table 4.8). There were no significant differences in upper and lower fractions of soil with PSDF (Table 4.9).

Soil depth, soil type, PSDF application method and rate and their interactions were significant for available potassium (Table 4.7). With PSDF available potassium was significantly lower in sand than loam soil (Table 4.8). Soil with PSDF had significantly more potassium in the upper than lower 10 cm (Table 4.9). Available potassium significantly increased with increasing PSDF rate, being significantly higher in sand soil with than without PSDF (Table 4.10).

Soil type and depth and PSDF type and rate and their interactions with PSDF application method were significant for available sulphur (Table 4.7). With PSDF loam soil had significantly higher available sulphur than sand (Table 4.8). PSDF resulted in significantly more sulphur in the upper than lower 10 cm (Table 4.9). Type of PSDF was only significant in the upper 10 cm. Spent twice PSDF had more sulphur than spent once (Figure 4.5). PSDF application rate significantly affected available sulphur, being highest at the highest PSDF rate (Table 4.10).

Soil depth and type and their interactions with PSDF application method and rate significantly influenced pH (Table 4.11). Soil pH was greater in sand than loam soil (Table 4.12) and in upper than lower 10 cm (Table 4.13). Increasing PSDF rate elevated pH in both soils and depths (Table 4.14). Both soils had significantly higher pH with PSDF than without (Table 4.14).

Soil type and depth and PSDF application method and their interactions were significant for electrical conductivity (Table 4.11). Electrical conductivity in loam soil with PSDF was highest in the lower 10 cm, and in sand was highest in the upper 10 cm (Table 4.13). Electrical conductivity in sand significantly increased with increasing PSDF rate. Electrical conductivity decreased with PSDF in loam soil relative to the control (Table 4.14), and was greater with sprayed PSDF in the upper 10 cm (Table 4.15).

Soil type and depth and PSDF rate and their interactions were significant for sodium adsorption ratio (Table 4.11). With PSDF in sand sodium adsorption ratio was 3 times higher than in loam (Table 4.12) and was significantly higher in upper than lower depths (Table 4.13). Sodium adsorption ratio increased significantly with increased PSDF, being lowest in controls (Table 4.14).

Cation exchange capacity and saturation were significantly affected by soil depth and type (Table 4.11). Loam soil had significantly greater cation exchange capacity and saturation than sand soil (Table 4.12). The highest cation exchange capacity was in loam soil with PSDF in the upper 10 cm (Table 4.13).

PSDF did not significantly affect heavy metals and hydrocarbon and all were below regulated limits (Tables 4.16, 4.17). With spent once PSDF barium was higher than with spent twice PSDF (Table 4.16). Chromium was slightly elevated with PSDF; copper increased in loam soil (Table 4.16). Total petroleum hydrocarbon and nickel increased in sand soil with PSDF (Table 4.17). All heavy metals and hydrocarbon with PSDF were much lower than the criteria limits (Alberta Environment and Sustainable Resource Development 2010).

3.4 PSDF Impacts on Leachate Quality

Leachate pH was significantly affected by soil type (Table 4.18). Spent PSDF in loam soil resulted in lowest pH (Table 4.19). Soil type, PSDF type, application method and rate and their interactions significantly affected electrical conductivity and sodium adsorption ratio (Table 4.18). Electrical conductivity was significantly higher in loam than sand soil. Sodium adsorption ratio with spent twice PSDF was highest in sand soil, and increased with increasing PSDF rate and incorporation versus spraying (Table 4.19).

PSDF did not significantly affect phosphorus in leachate (Table 4.20). Soil type and PSDF application method were significant for orthophosphate, which was significantly higher in loam than sand soil (Table 4.21). Incorporating PSDF elevated orthophosphate relative to spraying (Table 4.21). Soil type, PSDF type, application method and rate and their interactions significantly affected organic carbon (Table 4.20). Loam soils with PSDF had more organic carbon than sand soils (Table 4.21). Organic carbon in leachate with spent twice PSDF was significantly higher relative to spent once PSDF; incorporating PSDF significantly increased organic carbon in leachate relative to spraying (Figure 4.6). Organic carbon increased with PSDF rate, with highest values at the highest rate of incorporated PSDF (Figure 4.7). Dissolved silicon was significantly highest with incorporated PSDF in loam soil (Table 4.21).

Most heavy metals were not significantly impacted by PSDF application and had lower values than provincial water quality guidelines (Table 4.22). Manganese and iron were the exceptions (Alberta Environment and Sustainable Resource Development 2010).

Hydrocarbons in leachate were not significantly affected by PSDF (Table 4.23). Sand with spent twice PSDF had small amounts of toluene, total xylenes and F3 hydrocarbons, but much lower than provincial ground water quality requirements (Alberta Environment and Sustainable Resource Development 2010).

By the end of the experiment, calcium and magnesium had been transported from the upper 10 cm to the lower 10 cm of the column, then leached (Figure 4.8). For example, only 16 % (0.9 meq L^{-1}) total calcium remained in the upper soil fraction, while 56 % was in leachate. Small amounts of extractable sodium, potassium, chloride and sulphate were transported through the columns. Most of nitrate and nitrite were transported and accumulated in the lower 10 cm with and without PSDF. Total dissolved calcium, magnesium, nitrate and nitrite, in soil and leachate, decreased with PSDF; sodium and sulphate increased.

4. Discussion

4.1 PSDF Impacts on Soil Physical Properties

The significant decrease in hydraulic conductivity with increasing PSDF application rate is likely due to similar sized PSDF components in the pore spaces in the sand soil matrix and clay particle dispersion by potassium and sodium in loam soil. Zvomuya et al. (2009) also found reduced hydraulic conductivity at high drilling fluid rates. Increased bulk density and decreased total porosity with increasing PSDF indicated clogging of soil pores could reduce hydraulic conductivity (Daniel and Bouma 1974). High sodicity of PSDF likely induced structural forces and diffuse double layer forces which increased aggregation and stability between particles (Quirk and Murray 1991). High exchangeable potassium in PSDF could also reduce soil structure stability associated with reduced hydraulic conductivity. Potassium can cause lower relative hydraulic conductivity by dispersion of clay micro aggregates and their rearrangement to form a dense network of particles with smaller pores, rather than massive clay migration and formation of an impeded layer (Chen et al. 1983). Effects of soluble sodium and potassium on soil structure stability are not clearly defined in the literature. Some researchers showed greater soil stability with potassium relative to sodium (Arienzo et al. 2012). Thus high concentrations of potassium in PSDF may alleviate negative effects on soil structure and reduce hydraulic conductivity, relative to the large amount of sodium in traditional drilling fluid. Although PSDF at high rates (> 60 m^3 ha^{-1}) adversely affected hydraulic conductivity, application to medium to coarse texture soil could potentially benefit plant emergence and growth due to water availability, at least in the short term.

Changing soil function and clogged pores also explains why incorporating PSDF with soil lowered hydraulic conductivity relative to spraying. Zvomuya et al. (2009) found sprayed drilling fluids at 80 m^3 ha^{-1} impeded near saturated hydraulic conductivity and ponded water on the soil surface. In greenhouse (Chapters 2, 3) and laboratory, as PSDF cracked and shrank with lost water content and texture changes, water infiltrated quickly into cracks.

Soil organic matter can markedly increase field capacity (Bouyoucos 1939) and available water capacity of mineral soils (Hudson 1994). Organic matter significantly increased with increasing PSDF rate in leachate, likely the reason for decreasing field capacity with PSDF. High pH and salinity with PSDF affected organic matter depletion and increased leaching. Ondrasek et al. (2012) found lower dissolved organic carbon with increasing sodium chloride in a 34 day greenhouse experiment. The significant positive correlation between field capacity and total porosity indicated organic matter leached with PSDF, reducing micro porosity, which significantly affected soil water retention.

The significantly decreased hydraulic conductivity suggests addition of soluble calcium to improve salinity and sodicity with PSDF is difficult to achieve, at least initially, because of the leaching. However, field capacity and organic matter correlation indicates adding some organic matter with PSDF, such as polyacrylamide (Wallace et al. 1986), could be an effective way to alleviate PSDF salinity effects on soil aggregate stability and water retention for plant growth.

4.2 PSDF Impacts on Soil Chemical Properties

Available nitrogen decreasing with increasing PSDF rate is not unexpected as nitrate and nitrite are easily transported with percolating water. This was evidenced by the significantly higher available nitrogen and soluble nitrate in the upper 10 cm. Significantly lower available nitrogen with PSDF may be due to lower rates of organic components, such as starches and gum in PSDF, which could stimulate denitrification in saturated soil columns (Ball et al. 1996).

Since phosphorus is slowly available and strongly adsorbed by soil constituents, there was no significant response during the experiment. An earlier field experiment found phosphorus was slowly available for plant uptake and significant phosphorus effects in plant tissue were only observed 3 years after drilling fluids disposal (Zvomuya et al. 2011).

Available potassium and sulphur significantly increased when PSDF rate due to constituents of potassium pyrophosphate and potassium hydroxide in PSDF. Xie et al. (2011) found spreading up to 20 cm of drilling fluids on sandy soil increased available potassium and phosphorus. With PSDF potassium accumulated in the upper column soil since it has low leachability and was absorbed by soil particles. Almost all exchangeable and non-exchangeable soil sites would have to be potassium saturated before there was leaching (Johnston and Goulding 1992).

Soil pH with PSDF could have increased as a result of the higher pH drilling fluid, and resulting precipitation of calcium and magnesium, by cation adsorption competition in soil with high potassium and sodium in PSDF (Smiles 2006). Although electrical conductivity and sodium adsorption ratio significantly increased with increasing PSDF, values at the end of the study were lower than provincial and federal criteria for problem soils and would still be considered in the good soil quality category (electrical conductivity < 2, sodium adsorption ratio < 4) (Alberta Environmental Sciences Division 2001).

Some researchers found a significant increase in sodium, electrical conductivity and sodium adsorption ratio with drilling fluid land disposal, which appeared limiting to plant growth, but not detrimental to decrease crop production significantly (Bauder et al. 2005, Macyk et al. 1990, Lesky et al. 1989). The effect of high concentrations of potassium in PSDF on selectivity of exchange sites for potassium in relation to sodium, magnesium and calcium was observed in a previous study (Chapter 3). Some studies reported soil preference for potassium over calcium, magnesium and sodium (Lieffering and Mclay 1995). Robbins (1984) found an increase in the ratio of potassium to sodium from 1:20 to 1:1 in soil solution dramatically decreased soil preference for sodium, suggesting high applications of PSDF increased the potassium to sodium ratio and has potential to reduce sodium adsorption ratio.

4.3 PSDF Impacts on Leachate Quality

Elevated sodium adsorption ratio in leachate with increasing PSDF rate was caused by sodium in drilling fluid. The high pH could cause calcium and magnesium to precipitate in soil. The large amount of exchangeable potassium was more tightly held on soil exchange sites than were sodium, calcium and magnesium, thus these soluble salts were leached, reducing sodium effects in soil. Organic carbon in leachate significantly increased without PSDF, likely due to high pH and salinity in PSDF depleting and leaching organic matter (Ondrasek et al. 2012). The significantly higher sodium adsorption ratio and organic matter in leachate with incorporated PSDF indicated application method could change soluble ion movement in soil and affect leachate.

High potassium in PSDF potentially increases the risk of potassium in ground water. Most water regulations do not establish a threshold concentration for potassium. However, a maximum admissible concentration for potassium in water for human consumption of 12 mg L^{-1} was established by the European community (European Economic Community 2000). Potassium in our leachate was 5.68 to 6.40 mg L^{-1}, well below this limit.

Iron and manganese were slightly higher than Alberta guidelines (Alberta Environment and Sustainable Resource Development 2010). Other studies also found drilling fluid waste disposal was unlikely to impact release of arsenic, barium, chromium and lead (Deeley and Canter 1986). With loam soil, iron was 0.33 to 0.45 mg L^{-1} in leachate compared to the Alberta criteria of 0.30 mg L^{-1} in ground water. A number of studies support a proposed guideline in the range suggested for aquatic life. Milam and Farris (1998) suggested a no effect level of 0.4 mg L^{-1} (Fe^{2+}) would be appropriate. Wang (1986) suggested 0.37 mg L^{-1} was appropriate to protect aquatic plants. Thus, the concentration of iron in leachate with PSDF in loam soil was slightly above this guideline, but should not be an issue for agriculture and industry water use. Increased available iron with PSDF may benefit plant growth as a greenhouse study found plant biomass increased as drilling fluid rates increased, attributed to increased zinc and iron from drilling fluid when applying 5 to 60 g dry spent drilling fluid kg^{-1} on sorghum (*Sorghum bicolour* L. Moench) and corn (*Zea mays* L.) growth (Bauder et al. 1999).

As manganese is only slightly to moderately toxic to aquatic organisms in excessive amounts, the Alberta Government recommended 0.05 mg L^{-1} manganese in ground water (Alberta Environment and Sustainable Resource Development 2010), seems too strict to protect manganese toxicity in drinking and recreational water (Reimer 1988).

5. Conclusions

No detrimental effects on soil physical and chemical properties and leachate quality were detected when applying PSDF at rates ≤ 60 m^3 ha^{-1}, which was higher than the rate currently allowed in western Canada (40 m^3 ha^{-1}) for general water based drilling fluids. Hydraulic conductivity and field capacity were reduced and available potassium increased with PSDF. Loam soil with PSDF had better physical and chemical properties than those of sand soils. Spent PSDF did not significantly impact soil and leachate properties. Incorporated PSDF in the upper 10 cm of soil accelerated PSDF element transport through soil columns to leachate. This suggests incorporating PSDF with elevated soil macronutrients and hydraulic conductivity may be beneficial when applied at rates ≤ 60 m^3 ha^{-1} to well drained soils with deep rooted plant species.

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Table 4.1. Drilling fluid additive list supplied by drilling fluid supplier.

Function	Traditional Water Based Mud	Potassium Silicate Drilling Fuid
Control viscosity	Bentonite	Pregelatinized starch Xanthan gum
Thin the mud	Lignite or lignosulfonate	Potassium pyrophosphate
Disperse solid particles	Sodium hydroxide (NaOH)	Potassium hydroxide (KOH)
Increase density	Barite ($BaSO_4$) or hematite (Fe_2O_3)	
Control fluid loss		Polyanionic cellulose Pregelatinized starch
Stabilize the formation		Raw silicate Anionic water soluble polymer

Source: Marquis Alliance Ltd. (Ma 2008).

Table 4.2. Selected properties of raw, spent once and spent twice PSDF.

	Raw	Spent Once	Spent Twice
Density (Mg m^{-3})	1.29	1.13	0.90
Hydrogen ion activity (pH)	11.4	10.8	10.7
Electrical conductivity (dS m^{-1})	13.9	61.8	44.3
Sodium adsorption ratio	7.1	866.7	436.0
Calcium (mg kg^{-1})	82.1	15.7	31.8
Magnesium (mg kg^{-1})	< 17.6	7.0	4.3
Sodium (mg kg^{-1})	255.1	11633.3	9406.7
Potassium (mg kg^{-1})	6033.4	19200.0	9646.7
Chloride (mg kg^{-1})	91.8	1416.7	2116.7
Sulphate (mg kg^{-1})	< 85.3	3016.7	2436.7
Nitrate and nitrite (mg kg^{-1})	0.4	7.2	< 5.6
Benzene (mg kg^{-1})	ND	2.0	0.4
Toluene (mg kg^{-1})	ND	5.8	1.3
Ethylbenzene (mg kg^{-1})	ND	1.6	0.5
Total Xylenes (mg kg^{-1})	ND	11.3	3.5
F1 C_6 - C_{10} (mg kg^{-1})	ND	90.3	116.3
F2 C_{10} - C_{16} (mg kg^{-1})	ND	642.0	1470.0
F3 C_{16} - C_{34} (mg kg^{-1})	ND	1456.7	1810.0
F4 C_{34} - C_{50} (mg kg^{-1})	ND	578.7	548.0
Boron (mg kg^{-1})	0.1	5.1	9.6
Mercury (mg kg^{-1})	< 0.0002	0.1	0.1
Antimony (mg kg^{-1})	< 0.004	0.0	0.0
Arsenic (mg kg^{-1})	0.0	11.5	11.8
Barium (mg kg^{-1})	0.3	9430.0	4803.3
Beryllium (mg kg^{-1})	< 0.002	0.6	1.0
Cadmium (mg kg^{-1})	< 0.0002	0.2	0.2
Chromium (mg kg^{-1})	0.1	31.6	46.7
Cobalt (mg kg^{-1})	0.004	8.2	11.8
Copper (mg kg^{-1})	0.1	47.7	39.0
Lead (mg kg^{-1})	0.04	42.4	21.7
Molybdenum (mg kg^{-1})	< 0.02	4.0	4.0
Nickel (mg kg^{-1})	0.01	28.3	38.2
Selenium (mg kg^{-1})	< 0.004	1.03	1.03
Silver (mg kg^{-1})	0.02	0.6	0.5
Thallium (mg kg^{-1})	< 0.001	0.2	0.3
Tin (mg kg^{-1})	< 0.02	1.0	< 1.00
Uranium (mg kg^{-1})	0.01	0.9	1.3
Vanadium (mg kg^{-1})	0.1	37.4	60.9
Zinc (mg kg^{-1})	0.1	68.0	74.0

ND = not determined.

Table 4.3. Selected soil properties before PSDF application.

	Clay loam	Loam	Sand
Available nitrogen (mg kg^{-1})	< 2.0	84.3	1.0
Available phosphorus (mg kg^{-1})	< 5.0	34.0	14.0
Available potassium (mg kg^{-1})	312.7	350.7	43.3
Available sulphur (mg kg^{-1})	108.0	7.0	< 1.0
Cation exchange capacity (meq 100 g $^{-1}$)	23.3	24.0	2.0
Total carbon (%)	2.2	2.3	0.1
Total organic carbon (%)	1.7	2.0	0.1
Total nitrogen (%)	0.0	0.2	< 0.02
Hydrogen ion activity (pH)	7.9	4.8	6.5
Electrical conductivity (dS m^{-1})	2.0	1.4	0.1
Sodium adsorption ratio	9.9	0.1	0.3
Soluble sodium (mg kg^{-1})	207.7	3.3	1.0
Soluble calcium (mg kg^{-1})	46.4	82.4	2.2
Soluble magnesium (mg kg^{-1})	7.2	24.4	0.5
Soluble potassium (mg kg^{-1})	10.0	12.3	0.3
Soluble chloride (mg kg^{-1})	3.7	3.0	1.0
Soluble sulphate (mg kg^{-1})	127.0	5.1	0.5
Soluble nitrate (mg kg^{-1})	2.9	83.2	1.6
Total C6-C10 (mg kg^{-1})	< 1.0	< 1.0	< 1.0
Total C11-C40+ (mg kg^{-1})	460.0	590.0	13.3
Sand (%)	38.9	33.1	93.8
Silt (%)	28.2	41.3	1.7
Clay (%)	32.9	25.6	4.5

Table 4.4. P values of soil physical properties in soil with PSDF.

	Hydraulic Conductivity (10^{-3} cm $second^{-1}$)	Bulk Density (Mg m^{-3})	Total Porosity (%)	Field Capacity (%)
Soil (S)	0.002	< 0.001	< 0.001	< 0.001
PSDF (P)	0.318	0.012	0.016	0.458
Application Method (M)	< 0.001	< 0.001	< 0.001	0.029
Rate (R, m^3 ha^{-1})	< 0.001	0.053	0.029	< 0.001
S × P	0.196	0.697	0.622	0.686
S × M	< 0.001	0.454	0.48	0.055
P × M	0.231	0.186	0.173	0.697
S × R	0.401	0.296	0.261	0.747
P × R	0.152	0.167	0.156	0.638
M × R	0.912	0.365	0.357	0.003
S × P × M	< 0.001	0.277	0.253	0.749
S × P × R	0.561	0.536	0.466	0.836
S × M × R	0.939	0.776	0.797	0.355
P × M × R	0.107	0.841	0.824	0.892
S × P × M × R	0.500	0.549	0.570	0.872

Table 4.5. Soil physical properties in soil with PSDF.

		Hydraulic Conductivity (10^{-3} cm $second^{-1}$)	Bulk Density (Mg m^{-3})	Total Porosity (%)	Field Capacity (%)
Soil	Loam	6.9 ± 0.4	1.1 ± 0.01	58.6 ± 0.2	40.6 ± 0.4
	Sand	8.2 ± 0.3	1.6 ± 0.01	40.5 ± 0.2	22.4 ± 0.3
PSDF	PSDF 0	7.1 ± 0.6	1.3 ± 0.04	50.1 ± 1.3	31.6 ± 1.4
	PSDF 1	7.8 ± 0.5	1.4 ± 0.04	49.2 ± 1.4	31.7 ± 1.4
	PSDF 2	7.8 ± 0.4	1.3 ± 0.04	49.4 ± 1.3	31.1 ± 1.4
Application Method	Incorporated	6.6 ± 0.3	1.4 ± 0.03	49.0 ± 1.1	31.0 ± 1.2
	Sprayed	8.5 ± 0.4	1.3 ± 0.03	50.2 ± 1.1	31.9 ± 1.1
Rate (m^3 ha^{-1})	0	10.6 ± 1.8	1.3 ± 0.11	52.2 ± 4.2	36.4 ± 4.4
	20	9.3 ± 0.6	1.3 ± 0.04	50.0 ± 1.6	33.3 ± 1.6
	40	8.0 ± 0.5	1.3 ± 0.04	49.9 ± 1.5	32.4 ± 1.6
	60	7.7 ± 0.5	1.3 ± 0.04	49.5 ± 1.5	31.2 ± 1.6
	120	5.4 ± 0.5	1.4 ± 0.04	49.0 ± 1.6	29.0 ± 1.6

PSDF types = raw (PSDF 0), spent once (PSDF 1), spent twice (PSDF 2).
Means within a column, within treatments of the same variable, followed by standard errors.

Table 4.6. Hydraulic conductivity, bulk density, total porosity and field capacity for loam and sand soil with PSDF rate.

	0 (m^3 ha^{-1})	20 (m^3 ha^{-1})	40 (m^3 ha^{-1})	60 (m^3 ha^{-1})	120 (m^3 ha^{-1})
Hydraulic conductivity (10^{-3} cm second^{-1})					
Loam	7.2 (1.9)	8.0 (0.9)	7.3 (0.8)	7.3 (0.8)	5.1 (0.6)
Sand	14.1 (0.4)	10.5 (0.5)	8.6 (0.6)	8.2 (0.6)	5.6 (0.7)
Bulk density (Mg m^{-3})					
Loam	1.0 (0.0)	1.1 (0.0)	1.1 (0.0)	1.1 (0.0)	1.1 (0.0)
Sand	1.5 (0.0)	1.6 (0.0)	1.6 (0.0)	1.6 (0.0)	1.6 (0.0)
Total porosity (%)					
Loam	61.6 (0.6)	59.2 (0.3)	58.5 (0.3)	58.4 (0.5)	58.4 (0.4)
Sand	42.8 (1.0)	40.9 (0.5)	41.2 (0.5)	40.5 (0.4)	39.6 (0.3)
Field capacity (%)					
Loam	46.2 (0.4)	42.7 (0.6)	41.5 (0.7)	40.3 (0.5)	37.7 (1.0)
Sand	26.6 (0.1)	23.9 (0.4)	23.3 (0.4)	22.0 (0.5)	20.3 (0.4)

Means within treatments of the same variable, followed by standard errors.

Table 4.7. P values for available nutrients in soil with PSDF.

	Nitrogen (mg kg^{-1})	Phosphorus (mg kg^{-1})	Potassium (mg kg^{-1})	Sulphur (mg kg^{-1})
Depth (D)	< 0.001	0.629	< 0.001	< 0.001
Soil (S)	< 0.001	< 0.001	< 0.001	< 0.001
PSDF (P)	0.107	0.322	0.454	0.011
Application Method (M)	0.593	0.214	0.003	0.084
Rate (R, m^3 ha^{-1})	0.050	0.930	< 0.001	< 0.001
D × S	< 0.001	0.046	< 0.001	0.101
D × P	0.065	0.525	0.561	0.008
S × P	0.107	0.741	0.507	1.000
D × M	< 0.001	0.298	0.355	0.019
S × M	0.593	< 0.001	0.002	0.145
P × M	0.283	0.980	0.246	0.145
D × R	0.245	0.988	< 0.001	< 0.001
S × R	0.050	0.747	0.282	0.509
P × R	0.919	0.993	0.980	0.125
M × R	0.185	0.915	0.403	0.596
D × S × P	0.065	0.741	0.808	0.465
D × S × M	< 0.001	0.461	0.814	0.007
D × P × M	0.863	0.859	0.812	0.236
S × P × M	0.283	0.347	0.589	0.927
D × S × R	0.245	0.877	0.560	0.397
D × P × R	0.672	0.990	0.945	0.599
S × P × R	0.919	0.957	0.989	0.358
D × M × R	0.111	0.931	0.601	0.456
S × M × R	0.185	0.793	0.923	0.884
P × M × R	0.413	0.944	0.991	0.714
D × S × P × M	0.863	0.779	0.948	0.315
D × S × P × R	0.672	0.982	0.951	0.446
D × S × M × R	0.111	0.841	0.981	0.373
D × P × M × R	0.902	0.937	0.988	0.669
S × P × M × R	0.413	0.880	0.973	0.931
D × S × P × M × R	0.902	0.961	0.949	0.906

Table 4.8. Available nutrients in soils with PSDF.

		Nitrogen (mg kg^{-1})	Phosphorus (mg kg^{-1})	Potassium (mg kg^{-1})	Sulphur (mg kg^{-1})
Depth	Lower 10 cm	14.1 ± 1.9	23.3 ± 0.9	188.1 ± 16.3	5.5 ± 0.3
	Upper 10 cm	6.8 ± 1.2	23.1 ± 0.9	328.5 ± 22.2	9.1 ± 0.7
Soil	Loam	19.0 ± 2.0	31.3 ± 0.4	424.3 ± 14.6	10.3 ± 0.5
	Sand	2.0 ± 0.0	15.1 ± 0.2	92.3 ± 8.4	4.3 ± 0.4
PSDF	PSDF 1	11.6 ± 1.7	23.4 ± 0.9	262.1 ± 21.0	6.7 ± 0.5
	PSDF 2	9.4 ± 1.6	23.0 ± 0.9	254.6 ± 20.5	7.9 ± 0.6
Application Method	Incorporated	10.8 ± 1.9	23.5 ± 1.0	243.2 ± 19.3	6.9 ± 0.5
	Sprayed	10.1 ± 1.4	23.0 ± 0.8	273.4 ± 22.1	7.7 ± 0.6
Rate (m^3 ha^{-1})	0	30.2 ± 10.1	24.9 ± 3.2	207.9 ± 52.8	3.9 ± 0.8
	20	12.9 ± 2.7	23.1 ± 1.3	210.4 ± 24.6	5.0 ± 0.5
	40	10.8 ± 2.4	23.2 ± 1.2	231.4 ± 26.1	6.1 ± 0.5
	60	10.7 ± 2.4	23.4 ± 1.3	257.7 ± 27.9	7.1 ± 0.8
	120	7.5 ± 1.6	23.2 ± 1.2	333.8 ± 35.3	11.0 ± 1.0

PSDF types = spent once (PSDF 1), spent twice (PSDF 2).
Means within a column within treatments of the same variable, followed by standard errors.

Table 4.9. Soil nutrients in column upper and lower fractions with PSDF.

	Upper 10 cm	Lower 10 cm
Nitrogen (mg kg^{-1})		
Loam	11.7 (2.2)	26.2 (2.9)
Sand	2.0 (0.0)	2.0 (0.0)
Phosphorus (mg kg^{-1})		
Loam	30.8 (0.6)	31.8 (0.6)
Sand	15.5 (0.2)	14.8 (0.2)
Potassium (mg kg^{-1})		
Loam	514.5 (18.7)	334.1 (13.0)
Sand	142.5 (13.3)	42.1 (1.8)
Sulphur (mg kg^{-1})		
Loam	12.5 (0.9)	8.2 (0.3)
Sand	5.6 (0.7)	2.9 (0.2)

Means within the same variable, followed by standard errors.

Table 4.10. Available nutrients in soils with PSDF application rates.

	0 (m^3 ha^{-1})	20 (m^3 ha^{-1})	40 (m^3 ha^{-1})	60 (m^3 ha^{-1})	120 (m^3 ha^{-1})
Nitrogen (mg kg^{-1})					
Loam	58.3 (11.5)	23.9 (4.5)	19.5 (4.1)	19.5 (4.1)	12.9 (2.8)
Sand	2.0 (0.0)	2.0 (0.0)	2.0 (0.0)	2.0 (0.0)	2.0 (0.0)
Phosphorus (mg kg^{-1})					
Loam	35.0 (0.4)	31.2 (0.9)	31.0 (0.9)	31.9 (0.8)	31.1 (0.9)
Sand	14.8 (1.8)	15.0 (0.3)	15.3 (0.3)	15.0 (0.3)	15.3 (0.3)
Potassium (mg kg^{-1})					
Loam	374.3 (34.2)	365.2 (19.5)	392.8 (21.9)	423.9 (24.4)	515.4 (39.1)
Sand	41.3 (2.0)	55.5 (4.1)	70.1 (7.0)	91.4 (13.2)	152.3 (26.4)
Sulphur (mg kg^{-1})					
Loam	6.5 (0.6)	7.7 (0.5)	8.9 (0.6)	10.2 (0.9)	14.6 (1.3)
Sand	1.3 (0.2)	2.3 (0.2)	3.3 (0.4)	4.0 (0.8)	7.4 (1.1)

Means within the same variable, followed by standard errors.

Table 4.11. P values for detailed salinity in soil with PSDF.

	Hydrogen Ion Activity (pH)	EC (dS m^{-1})	SAR	CEC (meq $100g^{-1}$)	Saturation (%)
Depth (D)	< 0.001	0.563	< 0.001	0.028	0.042
Soil (S)	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
PSDF (P)	0.015	0.957	< 0.001	0.772	0.052
Application Method (M)	0.882	0.918	0.669	< 0.001	0.110
Rate (R, m^3 ha^{-1})	< 0.001	0.003	< 0.001	0.228	0.081
D × S	0.613	< 0.001	< 0.001	0.017	0.195
D × P	0.929	0.004	0.233	0.923	0.878
S × P	0.388	0.122	0.087	0.923	0.552
D × M	< 0.001	< 0.001	0.114	0.923	0.142
S × M	0.001	0.866	0.956	< 0.001	0.708
P × M	0.081	0.066	0.532	0.385	0.282
D × R	< 0.001	< 0.001	< 0.001	0.659	0.802
S × R	0.034	0.004	< 0.001	0.243	0.763
P × R	0.572	0.599	0.190	0.981	0.797
M × R	0.155	0.290	0.304	0.533	0.586
D × S × P	0.655	0.224	0.685	0.772	0.552
D × S × M	0.357	< 0.001	0.669	0.772	0.775
D × P × M	0.976	0.931	0.375	0.923	0.708
S × P × M	0.388	0.149	0.309	0.499	0.552
D × S × R	0.926	0.676	0.896	0.800	0.996
D × P × R	0.869	0.719	0.996	0.358	0.649
S × P × R	0.400	0.668	0.703	0.968	0.483
D × M × R	0.715	0.467	0.985	0.426	0.363
S × M × R	0.506	0.216	0.917	0.610	0.776
P × M × R	0.886	0.509	0.913	0.402	0.786
D × S × P × M	0.699	0.997	0.869	0.923	0.947
D × S × P × R	0.576	0.933	0.905	0.518	0.809
D × S × M × R	0.669	0.540	0.424	0.274	0.739
D × P × M × R	0.851	0.994	0.922	0.426	0.614
S × P × M × R	0.484	0.734	0.791	0.504	0.691
D × S × P × M × R	0.896	0.983	0.534	0.533	0.906

EC = electrical conductivity.
SAR = sodium adsorption ratio.
CEC = cation exchange capacity.

Table 4.12. Detailed salinity in soils with PSDF.

		Hydrogen Ion Activity (pH)	Electrical Conductivity (dS m^{-1})	Sodium Adsorption Ratio	Cation Exchange Capacity (meq 100 g^{-1})	Saturation (%)
Depth	Lower 10 cm	6.3 ± 0.1	0.4 ± 0.03	1.4 ± 0.1	13.1 ± 0.9	39.0 ± 0.9
	Upper 10 cm	6.8 ± 0.1	0.4 ± 0.02	3.1 ± 0.2	13.4 ± 1.0	40.0 ± 1.0
Soil	Loam	5.5 ± 0.0	0.5 ± 0.03	1.1 ± 0.1	22.5 ± 0.1	47.8 ± 0.4
	Sand	7.6 ± 0.1	0.2 ± 0.02	3.5 ± 0.2	4.0 ± 0.0	31.1 ± 0.2
PSDF	PSDF 1	6.5 ± 0.1	0.4 ± 0.03	2.1 ± 0.2	13.2 ± 0.9	39.0 ± 0.9
	PSDF 2	6.6 ± 0.1	0.4 ± 0.03	2.5 ± 0.2	13.3 ± 1.0	39.9 ± 0.9
Application Method	Incorporated	6.5 ± 0.1	0.4 ± 0.03	2.3 ± 0.2	13.5 ± 1.0	39.8 ± 0.9
	Sprayed	6.5 ± 0.1	0.4 ± 0.03	2.3 ± 0.2	13.0 ± 0.9	39.1 ± 0.9
Rate (m^3 ha^{-1})	0	5.9 ± 0.3	0.6 ± 0.18	0.2 ± 0.0	13.8 ± 2.6	38.6 ± 2.7
	20	6.3 ± 0.2	0.3 ± 0.04	1.2 ± 0.1	13.0 ± 1.3	38.6 ± 1.2
	40	6.4 ± 0.2	0.3 ± 0.04	1.8 ± 0.2	13.3 ± 1.4	39.4 ± 1.3
	60	6.5 ± 0.2	0.4 ± 0.04	2.3 ± 0.2	13.3 ± 1.4	39.5 ± 1.3
	120	6.9 ± 0.2	0.5 ± 0.03	3.8 ± 0.4	13.3 ± 1.4	40.4 ± 1.3

PSDF types = spent once (PSDF 1), spent twice (PSDF 2).
Means within a column, within treatments of the same variable, followed by standard errors.

Table 4.13. Electrical conductivity, pH, sodium adsorption ratio and cation exchange capacity in column upper and lower fractions with PSDF.

	Upper 10 cm	Lower 10 cm
Hydrogen ion activity (pH)		
Loam	5.7 (0.1)	5.2 (0.0)
Sand	7.9 (0.1)	7.3 (0.0)
Electrical conductivity (dS m^{-1})		
Loam	0.4 (0.0)	0.6 (0.0)
Sand	0.3 (0.0)	0.1 (0.0)
Sodium adsorption ratio		
Loam	1.7 (0.1)	0.4 (0.1)
Sand	4.5 (0.3)	2.5 (0.2)
Cation exchange capacity (meq 100 g^{-1})		
Loam	22.7 (0.2)	22.2 (0.2)
Sand	4.0 (0.0)	4.0 (0.0)

Means within the same variable, followed by standard errors.

Table 4.14. Electrical conductivity, pH, sodium adsorption ratio and cation exchange capacity in soils with PSDF application rates.

	0 (m^3 ha^{-1})	20 (m^3 ha^{-1})	40 (m^3 ha^{-1})	60 (m^3 ha^{-1})	120 (m^3 ha^{-1})
Hydrogen ion activity (pH)					
Loam	4.9 (0.0)	5.3 (0.1)	5.4 (0.1)	5.5 (0.1)	5.7 (0.1)
Sand	6.9 (0.1)	7.3 (0.1)	7.4 (0.1)	7.6 (0.1)	8.0 (0.1)
Electrical conductivity (dS m^{-1})					
Loam	1.1 (0.2)	0.5 (0.1)	0.5 (0.1)	0.5 (0.1)	0.5 (0.0)
Sand	0.1 (0.0)	0.1 (0.0)	0.2 (0.0)	0.2 (0.0)	0.4 (0.0)
Sodium adsorption ratio					
Loam	0.2 (0.1)	0.5 (0.1)	0.8 (0.1)	1.1 (0.1)	1.9 (0.3)
Sand	0.3 (0.0)	2.0 (0.2)	2.8 (0.2)	3.5 (0.3)	5.8 (0.4)
Cation exchange capacity (meq 100 g^{-1})					
Loam	22.3 (0.2)	22.1 (0.2)	22.5 (0.2)	22.6 (0.2)	22.6 (0.2)
Sand	5.2 (0.8)	4.0 (0.0)	4.0 (0.0)	4.0 (0.0)	4.0 (0.0)

Means within the same variable, followed by standard errors.

Table 4.15. Electrical conductivity, pH, sodium adsorption ratio and cation exchange capacity in soils with PSDF application methods.

	Upper 10 cm	Lower 10 cm
Hydrogen ion activity (pH)		
Incorporated	6.9 (0.2)	6.2 (0.2)
Sprayed	6.7 (0.2)	6.4 (0.1)
Electrical conductivity (dS m^{-1})		
Incorporated	0.3 (0.0)	0.4 (0.1)
Sprayed	0.5 (0.0)	0.3 (0.0)
Sodium adsorption ratio		
Incorporated	3.1 (0.3)	1.5 (0.2)
Sprayed	3.2 (0.3)	1.3 (0.2)
Cation exchange capacity (meq $100g^{-1}$)		
Incorporated	13.6 (1.4)	13.4 (1.4)
Sprayed	13.1 (1.3)	12.9 (1.3)

Means within the same variable, followed by standard errors.

Table 4.16. Canadian council of Ministers of the Environment (CCME) metals in soil with and without PSDF and AENV upper limits.

	Loam			Sand			AENV Soil Endpoint Limits	
Element (mg kg^{-1})	Control	Spent Once	Spent Twice	Control	Spent Once	Spent Twice	Agricultural Land Use	Natural Land Use
Boron	0.6	0.6	0.6	0.0	0.0	0.0	2	2
Mercury	0.0	0.0	0.0	0.0	0.0	0.0	6.6	12
Antimony	0.0	0.0	0.0	0.0	0.0	0.0	20	20
Arsenic	6.7	5.8	5.7	1.7	1.8	1.8	17	17
Barium	225	670	227	65	302	65	750	750
Beryllium	0.6	0.6	0.6	0.2	0.2	0.2	5	5
Cadmium	0.4	0.4	0.4	0.1	0.0	0.0	1.4	3.8
Chromium	20.0	21.5	20.4	4.0	4.1	4.2	64	64
Cobalt	9.9	9.1	8.9	2.5	2.3	2.3	20	20
Copper	17.5	19.1	18.9	3.0	2.3	2.6	63	63
Lead	10.3	9.9	9.9	2.6	2.5	2.4	70	70
Molybdenum	0.0	0.1	0.0	0.0	0.0	0.0	4	4
Nickel	18.3	25.0	16.9	3.8	4.8	5.0	50	50
Selenium	1.0	0.9	0.9	0.0	0.0	0.0	1	1
Silver	0.2	0.2	0.1	0.0	0.0	0.0	20	20
Thallium	0.2	0.2	0.2	0.1	0.0	0.0	1	1
Tin	1.0	0.0	0.0	0.0	0.0	0.1	5	5
Uranium	1.0	0.9	0.9	0.0	0.0	0.0	23	23
Vanadium	38.2	36.4	35.5	8.8	8.1	8.5	130	130
Zinc	69.0	61.3	61.6	15.0	14.9	15.2	200	200

AENV = Alberta Environment and Sustainable Resource Development.

All upper limits are based on the Alberta Tier 1 Soil and Groundwater Remediation Guidelines (2010).

Table 4.17. Hydrocarbon in soil with and without PSDF.

	Loam			Sand			AENV Soil Endpoints Limits Agricultural Use	
	Control	Spent Once	Spent Twice	Control	Spent Once	Spent Twice	Fine Soil (mg kg^{-1})	Coarse Soil (mg kg^{-1})
Benzene	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1
Toluene	0.0	0.0	0.0	0.0	0.0	0.0	0.5	0.5
Ethylbenzene	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.2
Total Xylenes (m,p,o)	0.0	0.0	0.0	0.0	0.0	0.0	15.0	12.0
Total hydrocarbon (C_6-C_{10})	0.0	0.2	0.6	0.0	0.3	0.3	210.0	24.0
Total hydrocarbon (>C_{11}-C_{40+})	425.0	160.6	160.0	0.0	1.9	0.0	150 - 5600	130 - 2800

AENV = Alberta Environment and Sustainable Resource Development.
All upper limits are based on Alberta Tier 1 Soil and Groundwater Remediation Guidelines (2010).

Table 4.18. P values for leachate detailed salinity in soils with PSDF.

	Hydrogen Ion Concentration (pH)	Electrical Conductivity (dS m^{-1})	Sodium Adsorption Ratio
Soil (S)	< 0.001	< 0.001	< 0.001
PSDF (P)	0.367	0.013	0.001
Application Method (M)	0.683	0.019	< 0.001
Rate (R, m^3 ha^{-1})	0.290	0.022	< 0.001
S × P	0.625	0.092	0.001
S × M	0.772	0.358	0.368
P × M	0.964	0.074	0.195
S × R	0.444	0.205	0.001
P × R	0.889	0.064	0.178
M × R	0.769	0.553	0.040
S × P × M	0.477	0.440	0.081
S × P × R	0.917	0.737	0.044
S × M × R	0.718	0.635	0.911
P × M × R	0.271	0.476	0.553
S × P × M × R	0.284	0.940	0.251

Table 4.19. Leachate detailed salinity in soils with PSDF.

		Hydrogen Ion Concentration (pH)	Electrical Conductivity (dS m^{-1})	Sodium Adsorption Ratio
Soil	Loam	7.3 ± 0.0	0.66 ± 0.02	0.17 ± 0.02
	Sand	7.8 ± 0.0	0.24 ± 0.01	0.34 ± 0.04
PSDF	PSDF 1	7.5 ± 0.1	0.42 ± 0.04	0.21 ± 0.03
	PSDF 2	7.5 ± 0.1	0.48 ± 0.03	0.30 ± 0.04
Application Method	Incorporated	7.5 ± 0.0	0.47 ± 0.04	0.35 ± 0.04
	Sprayed	7.5 ± 0.1	0.43 ± 0.03	0.16 ± 0.02
Rate (m^3 ha^{-1})	0	7.4 ± 0.2	0.75 ± 0.28	0.18 ± 0.05
	20	7.6 ± 0.1	0.40 ± 0.05	0.15 ± 0.02
	40	7.4 ± 0.1	0.45 ± 0.05	0.22 ± 0.03
	60	7.5 ± 0.1	0.45 ± 0.05	0.27 ± 0.04
	120	7.5 ± 0.1	0.50 ± 0.05	0.39 ± 0.07

PSDF types = spent once (PSDF 1), spent twice (PSDF 2).
Means within a column within treatments of the same variable, followed by standard errors.

Table 4.20. P values for selected leachate chemical properties in soils with PSDF.

	Phosphorus (mg L^{-1})	Orthophosphate (mg L^{-1})	Organic Carbon (mg L^{-1})	Silicon (mg L^{-1})
Soil (S)	0.898	< 0.001	< 0.001	< 0.001
PSDF (P)	0.389	0.518	< 0.001	0.754
Application Method (M)	0.840	< 0.001	< 0.001	< 0.001
Rate (R, m^3 ha^{-1})	0.160	0.755	< 0.001	0.633
S × P	0.177	0.557	0.001	0.835
S × M	0.659	0.242	0.041	0.109
P × M	0.883	0.680	0.001	0.403
S × R	0.922	0.993	0.132	0.987
P × R	0.808	0.895	< 0.001	0.864
M × R	0.518	0.963	< 0.001	0.929
S × P × M	0.927	0.638	0.304	0.987
S × P × R	0.872	0.991	0.013	0.754
S × M × R	0.967	0.615	0.617	0514
P × M × R	0.616	0.963	0.077	0.922
S × P × M × R	0.960	0.975	0.366	0.991

Table 4.21. Selected leachate chemical properties in soils with PSDF.

		Phosphorus (mg L^{-1})	Orthophosphate (mg L^{-1})	Organic Carbon (mg L^{-1})	Silicon (mg L^{-1})
Soil	Loam	0.32 ± 0.01	0.18 ± 0.01	55.9 ± 3.5	17.7 ± 0.7
	Sand	0.32 ± 0.02	0.10 ± 0.00	39.0 ± 5.4	9.9 ± 0.5
PSDF	PSDF 1	0.31 ± 0.01	0.14 ± 0.01	37.9 ± 3.2	13.9 ± 0.8
	PSDF 2	0.33 ± 0.02	0.14 ± 0.01	57.1 ± 5.5	13.7 ± 0.8
Application Method	Incorporated	0.32 ± 0.01	0.15 ± 0.01	62.0 ± 5.3	15.8 ± 0.6
	Sprayed	0.31 ± 0.02	0.12 ± 0.01	32.9 ± 2.7	11.7 ± 0.9
Rate (m^3 ha^{-1})	0	0.22 ± 0.02	0.15 ± 0.02	23.5 ± 8.5	13.4 ± 2.5
	20	0.28 ± 0.01	0.13 ± 0.01	28.8 ± 3.0	13.0 ± 1.1
	40	0.31 ± 0.01	0.14 ± 0.01	40.9 ± 4.1	14.2 ± 1.2
	60	0.33 ± 0.03	0.14 ± 0.01	48.4 ± 4.8	13.8 ± 1.2
	120	0.35 ± 0.02	0.14 ± 0.01	71.8 ± 9.4	14.1 ± 1.2

PSDF types = spent once (PSDF 1), spent twice (PSDF 2).
Means within a column within treatments of the same variable, followed by standard errors.

Table 4.22. CCME metals in leachate with and without PSDF and AENV upper limits.

Element (mg kg^{-1})	Loam			Sand			Alberta Groundwater Quality Limits
	Control	Spent Once	Spent Twice	Control	Spent Once	Spent Twice	
Hardness	186.0	237.0	251.4	50.8	102.8	127.3	500.0
Manganese	0.74	0.64	0.88	0.00	0.11	0.17	0.050
Iron	0.02	0.33	0.45	0.04	0.18	0.13	0.300
Chromium - III	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	NGR
Chromium - VI	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	NGR
Aluminum	0.040	0.637	0.401	0.092	0.364	0.143	NGR
Mercury	0.0000	0.0001	0.0000	0.0000	0.0000	0.0000	0.0001
Antimony	0.000	0.000	0.000	0.000	0.001	0.001	0.006
Arsenic	0.003	0.003	0.003	0.001	0.001	0.001	0.005 - 0.010
Barium	0.560	0.605	0.667	0.096	0.264	0.294	1.000
Boron	0.058	0.075	0.075	0.027	0.031	0.042	0.500 - 1.500
Cadmium	0.001	0.005	0.008	0.000	0.001	0.001	NGR
Copper	0.008	0.040	0.025	0.003	0.020	0.026	NGR
Lead	0.000	0.001	0.000	0.000	0.000	0.001	NGR
Nickel	0.005	0.022	0.016	0.001	0.005	0.007	NGR
Selenium	0.001	0.003	0.003	0.000	0.000	0.001	0.001 - 0.010
Silver	0.0000	0.0001	0.0000	0.0000	0.0000	0.0000	NGR
Uranium	0.000	0.000	0.000	0.000	0.000	0.000	0.010 - 0.020
Zinc	0.005	0.015	0.010	0.000	0.007	0.014	0.030 - 5.000

CCME = Canadian council of Ministers of the Environment.
NGR = no guideline required.
All upper the ceiling limits are based on the Alberta Tier 1 Soil and Groundwater Remediation Guidelines (2009).

Table 4.23. Hydrocarbons in leachate with and without PSDF.

	Loam			Sand			AENV Groundwater Quality Limits
	Control	Spent Once	Spent Twice	Control	Spent Once	Spent Twice	
Benzene	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	0.005
Toluene	< 0.001	<0.001	<0.001	<0.001	< 0.001	0.001	0.024
Ethylbenzene	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	0.002
Total Xylenes (m,p,o)	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	0.001	0.300
F1 (BTEX)	< 0.200	< 0.200	< 0.200	< 0.200	< 0.200	< 0.200	NGR
F1 (C_6-C_{10})	< 0.200	< 0.200	< 0.200	< 0.200	< 0.200	< 0.200	2.200
F2 (>C_{10}-C_{16})	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100	1.100
F3 (>C_{16}-C_{34})	< 0.100	0.013	< 0.100	< 0.100	< 0.100	0.013	NGR
F4 (>C_{34})	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100	NGR

AENV = Alberta Environment and Sustainable Resource Development.
NGR = no guideline required.
BTEX = benzene, toluene, ethylbenzene, xylenes.
All upper limits are based on Alberta Tier 1 Soil and Groundwater Remediation Guidelines (2010).

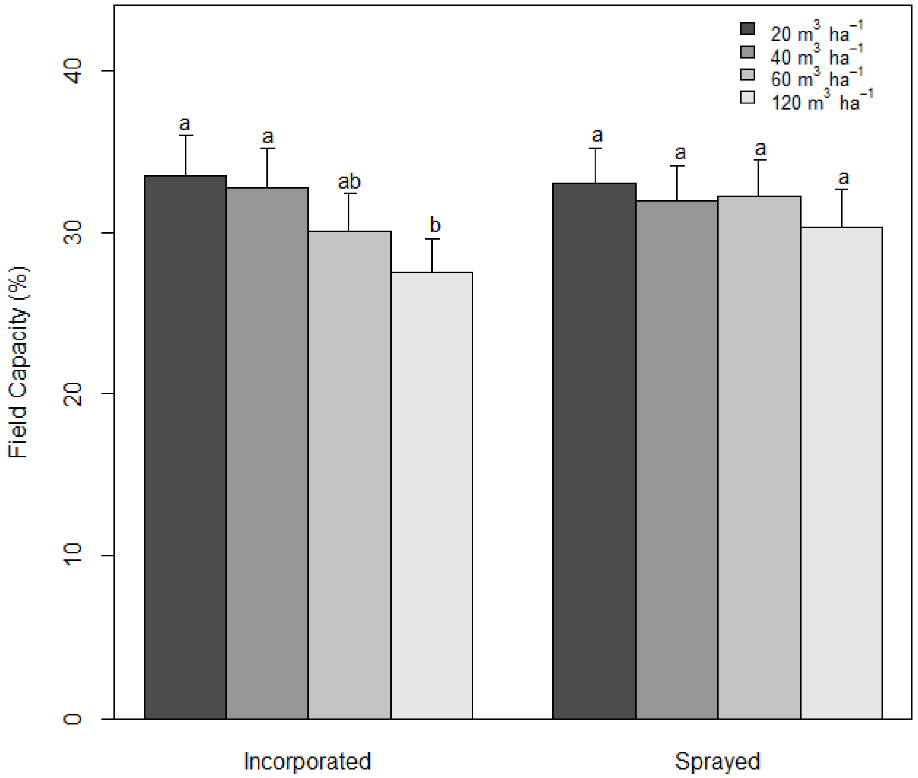

Figure 4.1. Field capacity with PSDF application method and rate. Error bars are standard error. Letters indicate significant differences between PSDF rates within PSDF incorporated and sprayed.

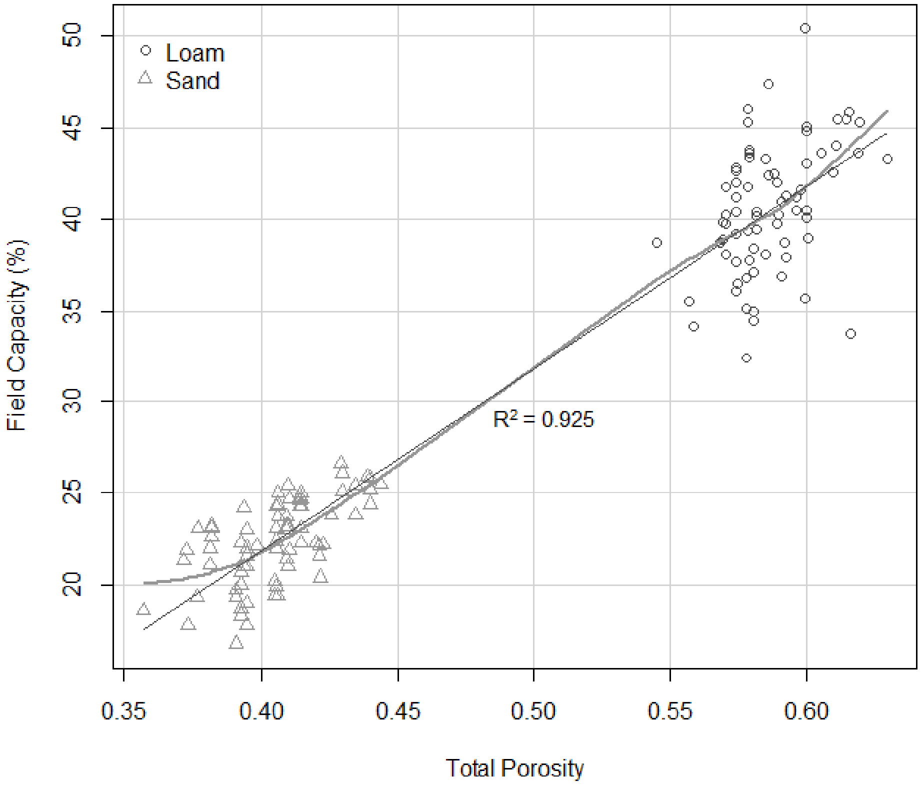

Figure 4.2. Correlation between field capacity and total porosity with PSDF in loam and sand soil.

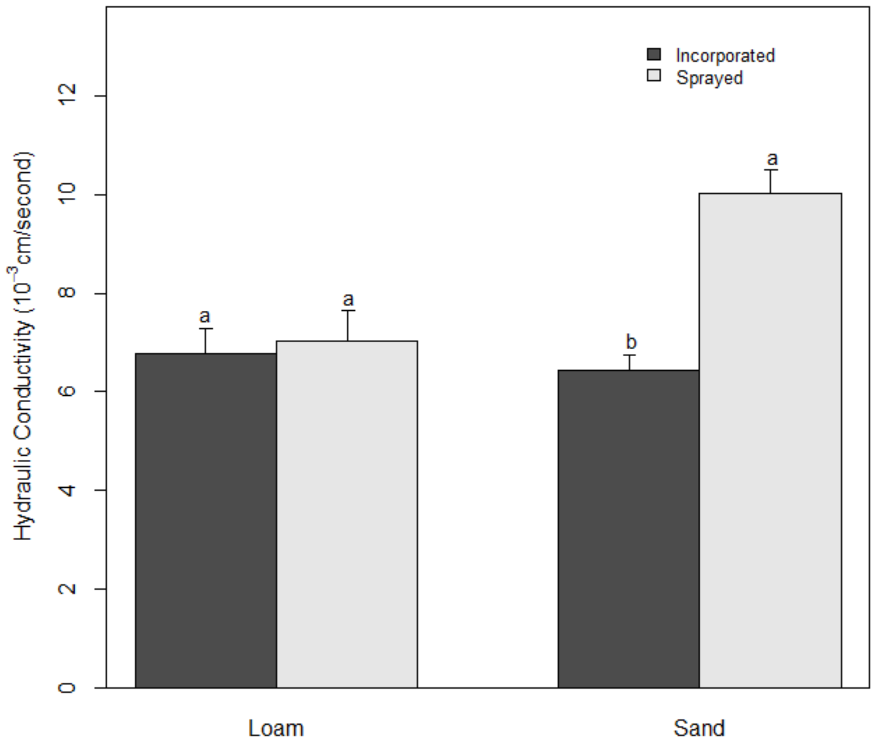

Figure 4.3. Hydraulic conductivity (10^{-3} cm second^{-1}) with PSDF application method and soil type. Error bars are standard error. Letters indicate significant differences between incorporated and sprayed PSDF within a soil type.

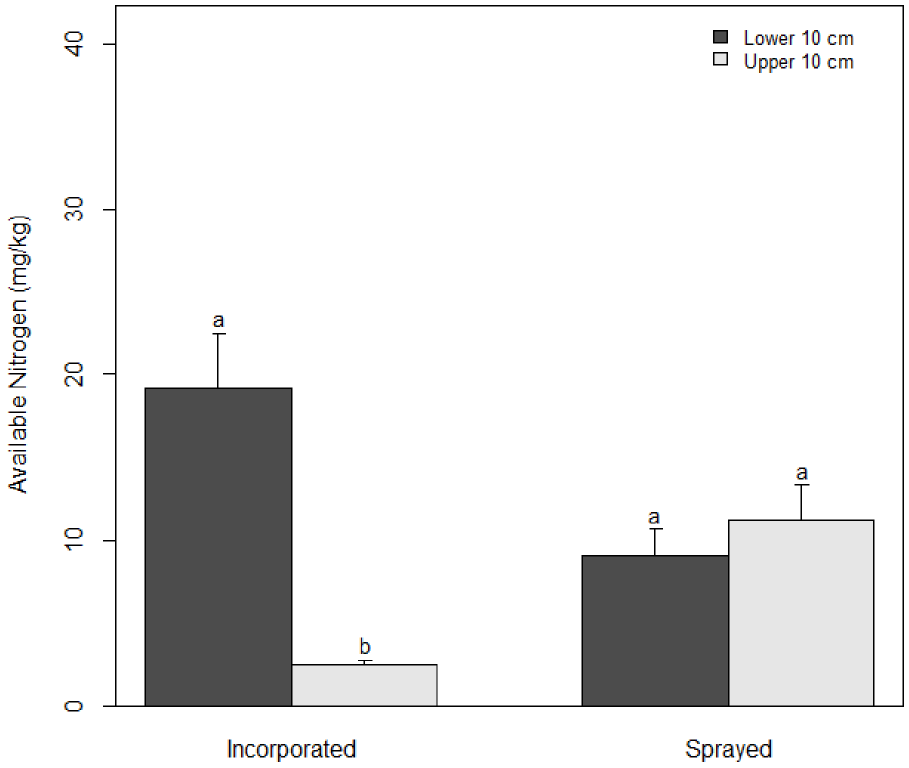

Figure 4.4. Available nitrogen in upper and lower 10 cm of soil with incorporated and sprayed PSDF. Error bars are standard error. Letters indicate significant differences between soil fractions within incorporated and sprayed PSDF.

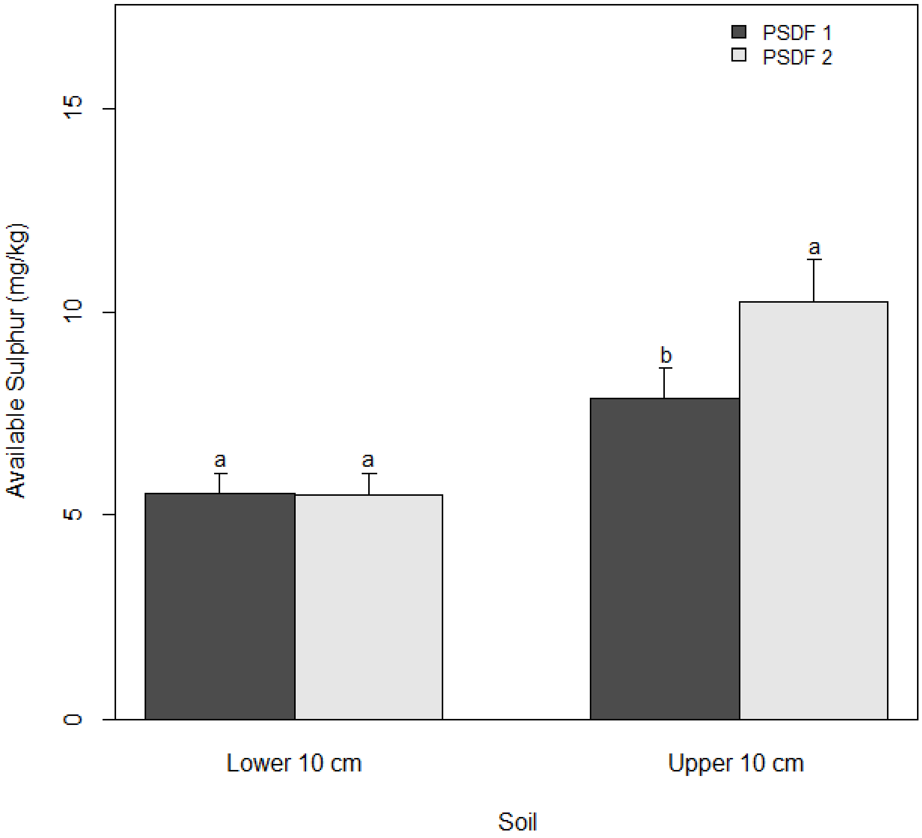

Figure 4.5. Available sulphur in upper and lower 10 cm of soil with spent once (PSDF 1) and spent twice (PSDF 2) PSDF. Error bars are standard error. Letters indicate significant differences between PSDF recycling times within soil depth increments.

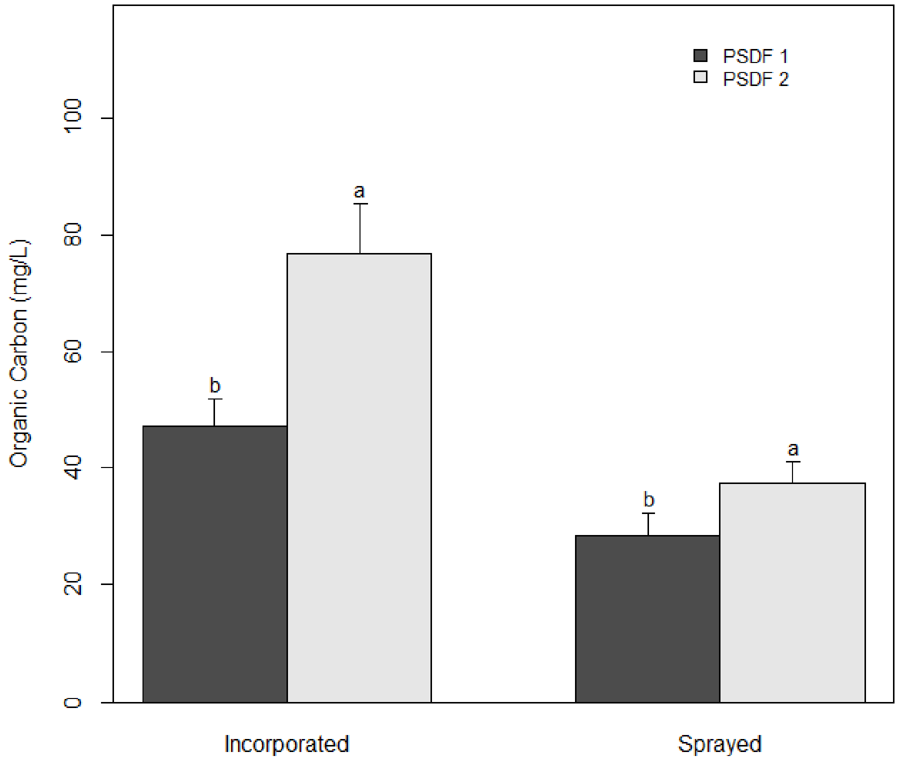

Figure 4.6. Organic carbon in leachate from soil with spent once (PSDF 1) and spent twice (PSDF 2) PSDF sprayed and incorporated. Error bars are standard error. Letters indicate significant differences between PSDF recycling times within PSDF application method.

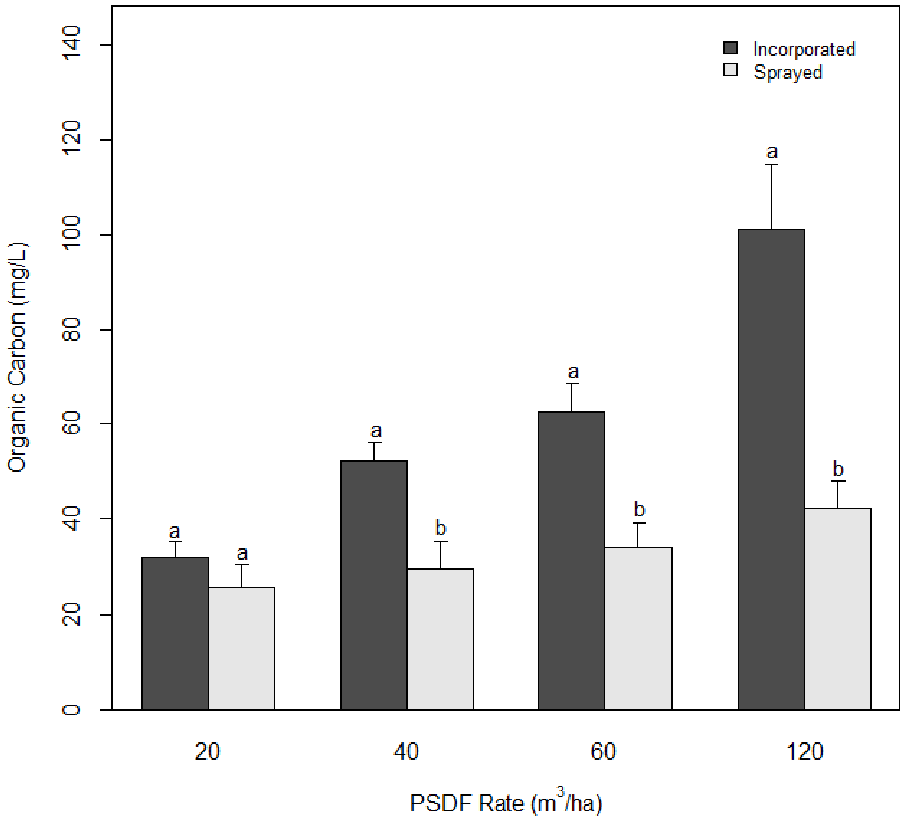

Figure 4.7. Organic carbon in leachate from soil with sprayed and incorporated PSDF rates. Error bars are standard error. Letters indicate significant differences between PSDF rates within PSDF application method.

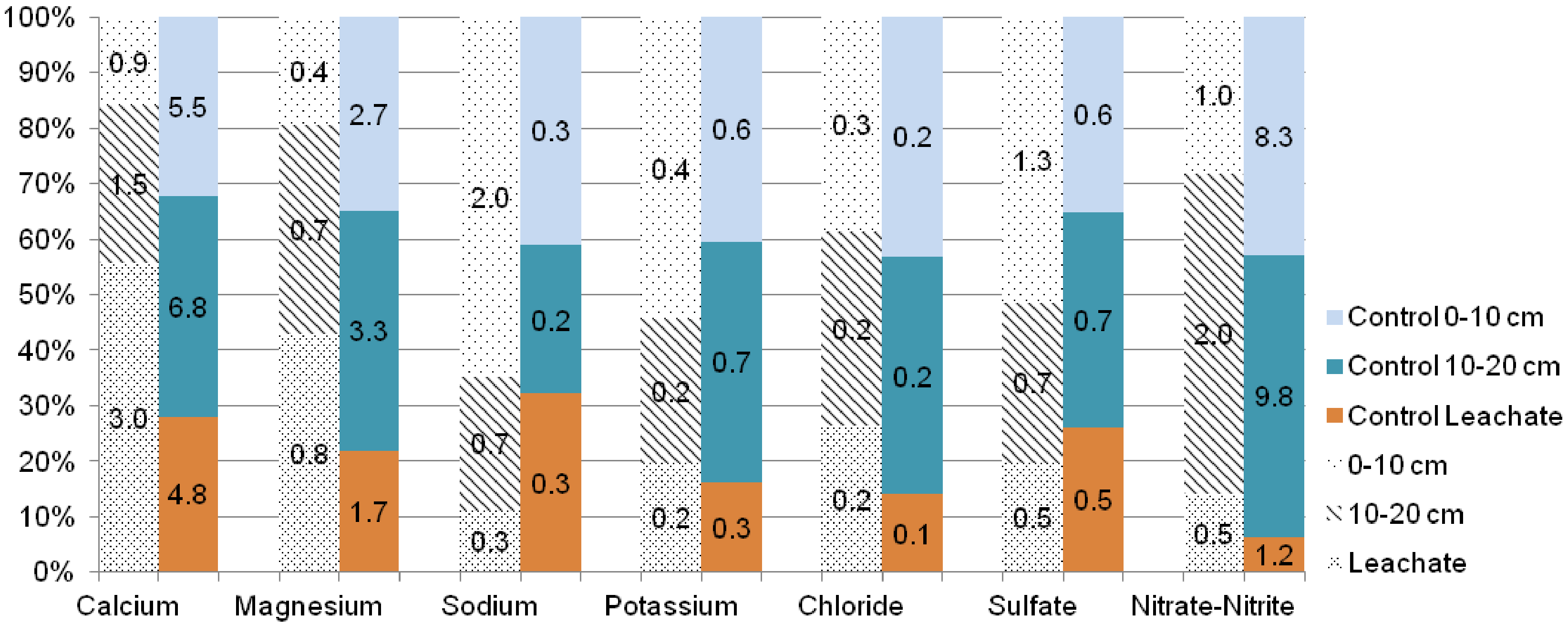

Figure 4.8. Distribution of extractable salts in soil and leachate with and without PSDF. Each pattern or color in the bars shows the percentage of soluble ions concentration in 0-10 cm, 10-20 cm depth of soil or collected leachate. Numbers in the bar indicate average ion concentrations (meq L^{-1}) in each treatment.

CHAPTER 5. INFLUENCE OF POTASSIUM SILICATE DRILLING FLUID DISPOSAL ON THE SOIL MICROBIAL COMMUNITY

1. Introduction

Drilling fluids are pumped into the bore holes to aid in the drilling process and transport drill cuttings to the soil surface in oil and gas operations. The drilling fluid systems typically consist of bentonite, barite, lignite, chrome lignosulphate, sodium hydroxide and various additives that are mixed with fresh water or hydrocarbons. Disposal of drilling fluid is a growing concern to the oil and gas industry and the regulators as it can require extensive waste management and result in significant land disturbance.

The path to economic and environmental sustainability requires a new way of thinking about oil and gas industry waste streams. Potassium silicate drilling fluid (PSDF), a relatively new mud system with high potassium and low sodium components, is a major waste generated in the oil and gas industry with potential as a reclamation amendment. In previous research, positive and negative effects of PSDF application on soil physical and chemical properties and vegetation were reported (Chapters 2, 3, 4). However, the measured parameters of vegetation and soil did not address soil microbial community dynamics. Since microorganisms are important components in linking whole ecosystems to plant and soil, research on the response of microorganisms to PSDF will help determine the overall impacts to the recipient soil and its biotic constituents.

Other researchers found that some compounds in drilling fluids can serve as carbon and energy sources for microorganisms. Hydrocarbons and surfactants in spent drilling fluid significantly impacted hydrocarbon utilizing microorganisms (Roling et al. 2003, James and Burns 1984, Atlas 1981). Sulphate reducing bacteria were the first microorganisms recovered from the deeply buried waters from oil bearing horizons of oil fields (Bastin et al. 1926). Addition of organic polymers, barite and lignosulfonates to drilling fluids stimulated sulphide producing bacteria communities (Struchtemeyer et al. 2011, Pereyra et al. 2010, Lie et al. 1999, Baldi et al. 1996). Various nitrate reducing microorganisms have been isolated, including autotrophic (Myhr and Torsvik 2000), heterotrophic

(Nazina et al. 1995) and chemolithotrophic (Gevertz et al. 2000). Some oil field isolates include ferric iron as an electron acceptor, but it is unclear how prevalent iron is in oil reservoirs (Greene et al. 1997).

Numerous factors affect composition and abundance of the soil microbial community, such as salinity (Okpokwasili and Odokuma 1990), electron donors (Chukwuma et al. 2010, Achtnich et al. 1995), electron acceptors (Lovley et al. 1996) and fertilizer (Jobson et al. 1974). In spite of nitrogen fertilizer deficiency and high potassium availability in soil with PSDF, increased salinity, sodicity and pH have been observed in previous studies (Chapters 2, 3, 4). Investigation of these effects on soil microbial communities is essential to understand the full impact of PSDF as a soil amendment in land reclamation.

Recycling drilling fluid is an important sustainability strategy to reduce waste volume and disposal cost. However, volume of waste fluids is increased by disintegration of rock cuttings during transport to the surface and limited efficiency of cuttings removal by the solids during rotary drilling (Wojtanowicz 2008). Those drilling cuttings have potential to change components of spent drilling fluid. Research to date has provided very little information on the influence of recycling times of drilling fluids on microorganisms.

Research has been conducted on distribution and diversity of microorganisms in oil based drilling fluid contaminated soil, but not on PSDF. The research objectives of this study were to determine whether the soil microbial community (microbial diversity and abundance) was impacted by PSDF, by recycling of PSDF and by different rates of PSDF application.

2. Material And Methods

2.1 Experimental Design

A soil microbiology experiment was established using a completely randomized design. Treatments represented reclamation application scenarios under various end land uses. The same soil and drilling fluid were used as in previous greenhouse and laboratory experiments. Soils were collected from three different locations in Alberta where drilling was active. Prior to experimentation, the soils were air dried and passed through a 2 mm sieve to obtain a homogeneous sample. Raw PSDF was made in the laboratory of the supplier, Marquis Alliance Ltd. Spent drilling fluids were collected from active well sites, all using the same PSDF. Prior to experimentation, fluids were refrigerated until used. All soil and drilling fluid properties were determined by Exova Laboratory Group.

Soil samples were sieved to remove large particles, ground to < 2 mm and the following analyses conducted. Available nitrate (NO_3^-) and ammonium (NH_4^+) were determined by extraction with 2.0 M potassium chloride (KCl) (Carter and Gregorich 2008); available phosphorus and potassium by modified kelowna extraction (Ashworth and Mrazek 1995); and available sulphate by extraction with 0.1 M calcium chloride ($CaCl_2$) (McKeague 1978). Cation exchange capacity was determined by exchange with ammonium acetate (NH_4OA_C) at pH 7 (McKeague 1978). Total nitrogen was determined by kjeldahl digestion distillation (Bremner 1996). Total carbon was determined by dry combustion (Nelson and Sommers 1996) and total organic carbon by the Walkley-Black wet dichromate oxidation method (Nelson and Sommers 1996). Water soluble cations (sodium, calcium, potassium, magnesium), pH, sodium adsorption ratio and electrical conductivity were determined from saturation paste extracts (Carter and Gregorich 2008). Routine ions (sulphate, chloride) were determined by ion chromatography with chemical suppression (Clesceri et al. 1992). Hydrocarbon fractions (F1, F2, F3, F4) were analyzed by photoionization and electrolytic conductivity detectors (Environmental Protection Agency 1996). Trace metals restricted in concentration by the Canadian Council of Ministers of the Environment (silver, arsenic, barium, beryllium, cadmium, cobalt, chromium, copper, mercury, molybdenum, nickel, lead, antimony, selenium, tin, thallium, uranium, vanadium, zinc) and boron were determined by nitric acid hotplate digestion (Environmental Protection Agency 2008). Sand, silt and clay were determined by hydrometer after treatment with calgon (Carter and Gregorich 2008).

Three soil texture treatments were sand, loam and clay loam, covering the wide range of soils with potential for reclamation using drilling fluids in Alberta. Three types of potassium silicate drilling fluids were raw, spent once and spent twice. Soil and PSDF were mixed with PSDF rates of 0, 40, 120 $m^3 ha^{-1}$, equivalent to 0, 15.1 and 45.3 mL PSDF per kg of dry soil, respectively. Pot loading rate was based on soil in greenhouse pots, 15 cm in diameter and 10 cm deep; assuming a particle density as 2.65 g cm^{-3}. The filled pots were stored in polypropylene bags and exposed to regular temperature (25 to 27 °C) for 2 weeks to allow time for soil microorganisms to respond to the PSDF. The three types of PSDF (raw, spent once, spent twice) without soil were also analyzed.

2.2 Enrichment and Quantification of Sulphate Reducing, Denitrifying and Iron Reducing Bacteria

Numbers of culturable sulphate reducing, denitrifying and iron reducing bacteria in PSDF treatments were determined using most probable number (MPN) dilutions (Cochran 1950). All MPN experiments were performed with five serial dilutions at 10^{-1}, 10^{-2}, 10^{-3}, 10^{-4} and 10^{-5} by using glass tubes and serially diluting 1.0 ml of sample into 9.0 ml of the appropriate medium.

Sulphate reducing bacteria were enumerated using a medium of 1 L deionized water with 0.5 g of dipotassium phosphate (K_2HPO_4), less than 1 g of ammonium (NH_4), 2.0 g of sodium sulphate (Na_2SO_4), 1.5 ml of sodium lactate (60%) and 1.0 g of yeast extract with pH 7.1 to 7.2 (Butlin et al. 1949). Denitrifying bacteria cultural media was prepared by dissolving 5 g of potassium nitrate (KNO_3) and peptone into 1 L deionized water with pH 7, excluding the agar in the original formula (Aaronson 1970). Iron reducing bacteria growth media was prepared by dissolving 0.5 g of ammonium sulphate (NH_4SO_4), 0.5 g of sodium sulphate (Na_2SO_4), 0.1 g of dipotassium phosphate (K_2HPO_4), 1.0 g of magnesium sulphate heptahydrate ($MgSO_4.7H_2O$), 5 g of ferric ammonium phosphate and 5 g of nutrient broth into 1 L deionized water (Aaronson 1970).

Five replicate tubes of each dilution were incubated at 21 °C for 7 days before assessment of bacteria. Sulphate reducing bacteria, denitrifying bacteria and iron reducing bacteria were enumerated in their respective selective culture media. Individual treatment dilution tubes were scored positive if they turned black for sulphate reducers as a result of production of ferrous sulphide, if they produced visible gas in Hungate tubes for denitrifying bacteria as they produced nitrous oxide gas, and if they displayed a visible colour change for iron reducing bacteria during ferric iron reduction and precipitation. Observations were made every 7 days for 21 days.

2.3 Enrichment and Quantification of Bacteria, Actinomycete and Fungi

Changes in aerobic heterotrophic microbial numbers were monitored by using a spread plate count technique. Serial dilution plate counts were used for enumeration of total heterotrophic aerobic bacteria and actinomycete on plate count agar and fungi on rose bengal malt extract agar (Ottow and Glathe 1968). Plate count agar was prepared by suspending 23.5 g powder of plate count agar (Difco) in 1 L of deionized water in a 2 L flask and mixing thoroughly (Zimbro et al. 2009). Rose bengal malt extract agar was prepared by dissolving 39 g of potato dextrose agar, 20 g of malt extract and 0.5 g of dipotassium phosphate (K_2HPO_4) in 1 L of tap water and adding 3 ml of micronutrients solution (100 ppm of each of ferric iron, molybdenum, copper and cobalt) and 8 ml of rose bengal solution. Rose bengal solution was prepared by dissolving 3 g of rose bengal in 500 ml deionized water, equivalent to 0.006 g ml^{-1} rose bengal (Ottow and Glathe 1968). All agar was sterilized in an autoclave at 121 °C for 30 minutes before dispensing into petri dishes to solidify.

Soil inocula were prepared by adding 10 g of each soil, previously sieved in a flame sterilized sieve, into a 90 ml phosphate buffer solution blank (10^{-1} dilution) and shaken 40 minutes in a laboratory shaker. Shaking time was based on a previous test with soil samples being shaken for increasing 10 minute periods up to an hour, after which serial dilutions were completed and 0.1 ml samples were plated, and colony forming units of bacteria were counted to verify that numbers were no longer increasing or decreasing due to shaking times. After this step, 10 ml of soil solution were added to another 90 ml phosphate buffer blank (10^{-2} dilution) and then shaken by hand for 15 seconds. These 10^{-2} diluted aliquots were then in turn serially diluted to 10^{-3}, 10^{-4}, 10^{-5}, 10^{-6} and 10^{-7}, with 15 second shaking periods at each dilution

Four plates for each dilution of a soil sample were prepared. A 100 µL volume of a given dilution was inoculated onto each plate and spread with sterile glass sticks to uniformly distribute the inoculum over the agar surface. Plated dilutions for plate count agar were 10^{-5}, 10^{-6} and 10^{-7}. Plated dilutions for rose bengal malt extract agar were 10^{-3}, 10^{-4} and 10^{-5}. Plates were placed in a plastic bag to avoid evaporation of the medium and incubated in the dark at 21 °C for 6 days before assessment. Colony forming units were cumulatively counted each week over a 4 week period. Colonies were scored by hand using sharpie marks on the reverse side of the plates to keep an accurate count.

A composite subsample from each soil was used to measure soil water content. The soil subsamples were weighed, oven dried at 80 °C for 24 hours and weighed again. A dry weight factor was calculated for each soil and the factor was used to estimate number of colony forming units per gram of dry soil. Mean number of bacteria and fungi colony forming units obtained from each soil sample was multiplied by the dry weight factor for that sample. Morphotypes of fungi, bacteria and actinomycetes on each replicate plate at the counting dilution were described based on shape (regular, irregular, conical, round), colour and texture (glossy, opaque, velvety).

2.4 Identification of Bacteria and Fungi

Microorganisms growing on mineral salt agar plates with each of the PSDF and soil treatments as the sole carbon source were counted, purified and isolated. The isolates were stored on slants of plate count agar and rose bengal malt extract agar for bacteria and fungi, respectively. A small portion of each fungus growth was picked and emulsified by inoculating a needle and placing the inoculant on separate glass slides containing a drop of lactophenol cotton blue (Gilman and Abbott 1957). A cover slip was applied and the slide was examined under 4 x to 40 x power objectives. Fungi were identified to genera based on morphological features and arrangement of spores and fruiting bodies as observed under electron microscopy following standard determinative schemes (Watanabe 2010).

Bacteria which comprised the mixed population were classified to genera on the basis of the following tests. For gram reaction, a dilute bacterial smear on a glass slide was heat fixed then was stained with a dark stain (crystal violet). After a minute of exposure to the staining solution, the slide was washed in water. The smear was treated with few drop of gram's iodine to fix the crystal violet and allowed to act for a minute. After rinsing with water, absolute ethyl alcohol was used to rinse off the slide and remove any excess crystal violet or destain the smear if the bacterial isolate was not gram positive. Ethyl alcohol contact time was never more than 30 seconds. The smear was treated with few drops of counterstain (safranin) for 1 minute and washed in water; excess water was removed using a blotting paper, after which the slide was dried in air and heat fixed before observing under oil immersion with a microscope at 93 x 10 magnification. Bacteria that retained the stain and appeared dark purple under microscopy were called gram positive and those which lost the stain and spores appeared pink or red under microscopy were called gram negative (Gram 1884).

For the gram negative bacteria an oxidase test was conducted to determine whether the isolates were oxidase positive or oxidase negative. Oxidase positive bacteria were then inoculated into Oxi / Ferm strips (BBL II) and oxidase negative into Enterotube (BBL II) strips. After overnight incubation, the color and gas production in strip chambers were observed. These changes indicated a positive or negative result for the presence of particular enzymes. Each positive result was used in generating a five digit number, which identified the bacteria based on the Oxi / Ferm or Enterotube code book (Gilardi 1985, Lennette et al. 1985).

2.5 Statistical Analyses

Distribution of the taxonomic composition of the soil microbial community associated with PSDF treatments and with textured soils was indentified in the ordination using correspondence analysis, a multivariate statistical technique, since taxonomic composition is categorical rather than continuous data (Greenacre 2010). The taxonomic distance among genera was assessed using Hellinger distance calculated from the scores of the first two axes (Legendre and Gallagher 2001). The position of the treatments and genera indicate the relationship between microbial composition and the environmental variables, such as soil and PSDF. All analyses and the graphics were performed with R software (R Development Core Team 2012).

3. Results

3.1 Soil and PSDF

Selected baseline soil properties prior to PSDF application are presented in Table 5.1. Selected PSDF components and properties are presented in Table 5.2

and 5.3. Most notably, raw PSDF had high pH and electrical conductivity and did not contain hydrocarbons.

3.2 Sulphate Reducing, Denitrifying and Iron Reducing Bacteria Most Probable Number

In PSDF, sulphate reducers were not detected except in spent twice PSDF (Table 5.4). There was no response of sulphate reducing bacteria numbers to PSDF application in sand soil and no significant response in clay loam and loam soil, except with spent PSDF at 120 m^3 ha^{-1}.

Addition of PSDF to soil significantly stimulated nitrate reducing bacteria, relative to controls without PSDF (Table 5.4). Low numbers of denitrifying bacteria were detected in spent twice PSDF but not in raw and spent once PSDF without soils. All three soil treatments had a resident denitrifying population; however clay loam and sand soils had denitrifiers in the hundreds per gram of soil whereas the loam had numbers in excess of twenty thousand viable cells per gram of soil.

MPN estimates of iron reducing bacteria decreased in all treatments with PSDF on clay loam soil relative to controls (Table 5.4). Iron reducers were stimulated with PSDF in sand and loam soil, except with spent twice PSDF in loam soil. In treatments with pure PSDF without soils, only spent twice PSDF had detectable iron reducers, where growth was stimulated when it was mixed with soil.

3.3 Fungi and Bacteria Colony Forming Units

Growth of bacteria, actinomycete and fungi was stimulated in soil with PSDF relative to no PSDF (Table 5.5). Repeated use of drilling fluid significantly increased viable bacteria and actinomycete colony forming units in the fluids. Application of raw, spent once and spent twice drilling fluid to clay loam soil stimulated a 100 to 1000 fold increase in aerobic bacteria. However, addition of drilling fluid on loam soil only stimulated a 100 fold increase in aerobic bacteria. Relative to controls without PSDF, application of PSDF to sand soil showed a 100 fold bacteria increase only in the case of spent once drilling fluid. Addition of PSDF stimulated fungal growth 10 to 100 fold. Only spent twice PSDF in loam soil had a fungal count > 10,000 colony forming units per gram of soil.

3.4 Analysis of Microbial Communities in Soils Mixed with PSDF

A summary of the microbial isolates obtained from treatments with and without PSDF is presented in Table 5.6. With PSDF, treatments in sand soil showed a significant increase in diversity of fungal genera isolated, while abundance of detected bacterial genera doubled in loam soil with PSDF. Relative to controls, bacterial species diversity only increased significantly in loam soil with PSDF.

Isolated bacterial genera were identified as *Achromobacter*, *Agrobacterium*, *Bacillus*, *Providencia*, *Pseudomonas* and *Rhodococci*. Fungi were identified as members of the *Aspergillus*, *Ascomycota*, *Aureobasidium*, *Cephaliophora*, *Cephalosporium*, *Cladosporium*, *Fusarium*, *Gonatobotrys*, *Penicillium* and *Trichoderma* genera. These isolates were morphologically distinct colony types picked from the counting dilution of each dilution plate. Therefore, they represent the most numerous bacterial and fungal in each soil type under control conditions and under the influence of PSDF.

Correspondence analysis showed that microbial communities in soil with PSDF were grouped by soil type (Figure 5.1). All groups of treatments, which displayed short gradients on their own, were associated with characteristic microbial genera. No significant separation by PSDF type was observed. *Bacillus* was the most common bacteria genus in PSDF and soil treatments. A total of 30 soil and PSDF treatments were used to generate this ordination, and 82.7 % of the microbial genera variation due to soil and PSDF application are explained in the first two axes. This analysis indicates in each soil type, soil microbial community composition was similar between the control and with PSDF application. Relative to pure PSDF without soil treatments, clay loam soil with spent twice PSDF showed similar microbial community composition.

4. Discussion

Recycling PSDF had a significant effect on the population and diversity of soil microorganisms by introducing microorganisms into drilling waste and increasing the number of sulphate reducing bacteria, nitrate reducing bacteria, iron reducing bacteria and total bacteria and actinomycetes. As there were no microorganisms in raw PSDF, the origin of the microorganisms was from the drilling process.

Magot (2005) found biogenic sulphide production and microorganisms induced a number of problems, including reservoir plugging, souring and corrosion of metal containing equipment in the oil fields, but the origin of microorganisms was unclear. Struchtemeyer et al. (2011) compared drilling mud with drilling water, the basal fluid for drilling mud, and substantiated that drilling mud was responsible for introducing microorganisms into oil and gas reservoirs. In our study, data before and after drilling showed the drilling process was the main origin of microorganisms in drilling fluid, and the number of times drilling fluid was recycled increased the opportunity for introduction of exogenous microorganisms. With increasing PSDF recycling, total petroleum hydrocarbons will increase in drilling fluid. Distribution of n-alkanes in spent PSDF was attributed to their selective utilization by sulphate reducing, denitrifying and iron reducing bacteria. Spent twice PSDF had significantly higher fractions F1 to F3 (C_6-C_{34}) petroleum hydrocarbons. Some earlier reports have shown that those petroleum hydrocarbons can supply potential electron donors by fatty acids, hydrogen and carbon resources for sulphate reducing bacteria (Beller et al. 1996, Rueter et al. 1994, Rabus et al. 1993), denitrifying bacteria (Ball et al. 1996, Rabus and Widdel 1995, Fries et al. 1994, Altenschmidt and Fuchs 1991, Evans et al. 1991, Schocher et al. 1991) and iron reducing bacteria (Lovley and Lonergan 1990).

Addition of PSDF to soil stimulated denitrifying bacteria and total microorganisms due to components in PSDF. Denitrifying bacteria could grow aerobically without nitrate or use nitrate as the electron acceptor for growth in the absence of oxygen. Chemolithotrophic nitrate reducing bacteria have been isolated from oil production fluids in a Canadian oil field (Gevertz et al. 2000). Since the amount of nitrate carried from PSDF is low, the stimulated number of denitrifying bacteria was not likely due to nitrate in PSDF. The large amounts of organic polymers and starch substrates added to PSDF could be their energy and carbon sources. Several groups of microorganisms, including polymer degraders, sugar or monomer metabolizers and volatile fatty acid degrading bacteria degrade organic polymeric substrates (McInerney et al. 1981) and were detected in drilling muds (Struchtemeyer et al. 2011). The capacity for true denitrification is limited to specific bacteria and active species are largely limited to *Pseudomonas*, *Bacillus* and *Parcoccus* genera (Christensen 1985, Valera and Alexander 1961). *Achromobacter* can grow anaerobically by using nitrate as an electron acceptor.

Achromobacter and *Bacillus* were detected in PSDF treated soils, and *Pseudomonas* was stimulated in loam and sand soil with PSDF. This stimulation of denitrifying bacteria suggests the necessity for nitrogen fertilizer inputs to enhance soil nitrogen availability for plant growth when utilizing PSDF as a land reclamation amendment.

The higher abundance of microorganisms with PSDF than without showed that adding PSDF to soil stimulated bacterial and fungal growth. This could result from bioaugmentation with addition of external microbial populations from PSDF to the soil (Okoh 2003), as well as biostimulation with organic polymers and total petroleum hydrocarbon in PSDF that increased nutrient and microbial activities of indigenous microbial flora (Okolo et al. 2005, Ogboghodo et al. 2004). The relatively higher bacterial than fungal counts in all treatments indicated high pH and salinity from PSDF are more favourable for bacteria. The occurrence of *Bacillus*, a known alkaliphile, in all spent PSDF treatments indicated the increase in pH associated with PSDF application stimulated alkaliphilic microorganisms.

Since microbial isolates were morphologically distinct colony types from each dilution they represent the most numerous bacteria and fungi in each soil type under control conditions and under the influence of PSDF. With spent PSDF without soil actimomycete, *Bacillus*, *Rhodococci* and *Aspergillus* genera were isolated. The ability to degrade hydrocarbons is widespread in *Rhodococci* (Warhurst and Fewson 1994) which play a significant role in the fate of organic pollutants in the environment and could be useful for treatment of contaminated soils or effluents (Bell et al. 1999). In PSDF with soil, genera of *Agrobacterium, Providencia, Pseudomonas*, *Aureobasidium, Fusarium* and *Penicillium* were stimulated relative to soils without PSDF. Actinomycete, bacterial genera *Bacillus* and *Pseudomonas* and fungal genera *Fusarium* and *Penicillium* can utilize drilling fluid waste as a carbon source (Struchtemeyer et al. 2011, Chen et al. 2009, BenkaCoker and Olumagin 1995, Nnubia and Okpokwasili 1993). Presence of *Fusarium* genera with PSDF in clay loam soil could indicate slight growth in ferric salts without oxygen and acidic soil (Gunner and Alexander 1964). *Agrobacterium*, a heterotrophic bacteria, could be stimulated by PSDF as an energy source (Bergey and Breed 1989). *Providencia* are gram negative bacteria, commonly found in soil, water and sewage. *Aureobasidium* is a cosmopolitan black yeast like fungus, widely distributed in hypersaline habitats (Nagahama 2006). The increased pH with PSDF in sand soil could stimulate growth of these organisms. These results suggest bioaugmentation with microbial species from spent PSDF may promote degradation and increase plant establishment and development in soil with spent PSDF.

Addition rate of PSDF variably impacted colony forming units of microorganisms as soil properties interfered. Soil texture with PSDF was strongly correlated with microbial composition. Spatially diverse soils with the same soil texture had nearly identical bacterial communities as soil texture is important in determining soil water and nutrient status (Lauber et al. 2008, Girvan et al. 2003). Lauber et al. (2008) found soils with extractable phosphorus tended to have similar fungal communities. From the current study, baseline soil texture, pH and available nutrients may have a significant impact on bacterial and fungal communities in addition to PSDF impact on soil for land reclamation.

5. Conclusions

Utilizing PSDF with soil stimulated the soil microbial populations and increased diversity of the microbial community relative to treatments without PSDF. The drilling process with increasing recycling times could increase the opportunity to introduce exogenous microorganisms into drilling fluids. Organic components in PSDF and petroleum hydrocarbons from the drilling process will be the main energy supply resources for microorganisms other than inorganic substrates in PSDF. PSDF with high pH stimulated growth of alkaliphilic microorganisms. Initial soil texture, pH and nutrients can have a significant effect on microbial community structure.

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Table 5.1. Selected soil properties before PSDF application.

	Clay loam	Loam	Sand
Available nitrogen (mg kg^{-1})	< 2.0	84.3	1.0
Available phosphorus (mg kg^{-1})	< 5.0	34.0	14.0
Available potassium (mg kg^{-1})	312.7	350.7	43.3
Available sulphur (mg kg^{-1})	108.0	7.0	< 1.0
Cation exchange capacity (meq 100 g $^{-1}$)	23.3	24.0	2.0
Total carbon (%)	2.2	2.3	0.1
Total organic carbon (%)	1.7	2.0	0.1
Total nitrogen (%)	0.0	0.2	< 0.02
Hydrogen ion activity (pH)	7.9	4.8	6.5
Electrical conductivity (dS m^{-1})	2.0	1.4	0.1
Sodium adsorption ratio	9.9	0.1	0.3
Soluble sodium (mg kg^{-1})	207.7	3.3	1.0
Soluble calcium (mg kg^{-1})	46.4	82.4	2.2
Soluble magnesium (mg kg^{-1})	7.2	24.4	0.5
Soluble potassium (mg kg^{-1})	10.0	12.3	0.3
Soluble chloride (mg kg^{-1})	3.7	3.0	1.0
Soluble sulphate (mg kg^{-1})	127.0	5.1	0.5
Soluble nitrate (mg kg^{-1})	2.9	83.2	1.6
Total C6-C10 (mg kg^{-1})	< 1.0	< 1.0	< 1.0
Total C11-C40+ (mg kg^{-1})	460.0	590.0	13.3
Sand (%)	38.9	33.1	93.8
Silt (%)	28.2	41.3	1.7
Clay (%)	32.9	25.6	4.5

Table 5.2. Drilling fluid additives.

Function	Traditional Water Based Mud	Potassium Silicate Drilling Fluid
Control viscosity	Bentonite	Pregelatinized starch Xanthan gum
Thin the mud	Lignite or lignosulfonate	Potassium pyrophosphate
Disperse solid particles	Sodium hydroxide ($NaOH$)	Potassium hydroxide (KOH)
Increase density	Barite ($BaSO_4$) or hematite (Fe_2O_3)	
Control fluid loss		Polyanionic cellulose Pregelatinized starch
Stabilize the formation		Raw silicate Anionic water soluble polymer

Source: Marquis Alliance Ltd. (Ma 2008)

Table 5.3. Selected PSDF properties.

	Raw	Spent Once	Spent Twice
Density (kg m^{-3})	1.29	1.13	0.90
Hydrogen ion activity (pH)	11.4	10.8	10.7
Electrical conductivity (dS m^{-1})	13.9	61.8	44.3
Sodium adsorption ratio	7.1	866.7	436.0
Calcium (mg kg^{-1})	82.1	15.7	31.8
Magnesium (mg kg^{-1})	< 17.6	7.0	4.3
Sodium (mg kg^{-1})	255.1	11633.3	9406.7
Potassium (mg kg^{-1})	6033.4	19200.0	9646.7
Chloride (mg kg^{-1})	91.8	1416.7	2116.7
Sulphate (mg kg^{-1})	< 85.3	3016.7	2436.7
Nitrate and nitrite (mg kg^{-1})	0.4	7.2	< 5.6
Benzene (mg kg^{-1})	ND	2.0	0.4
Toluene (mg kg^{-1})	ND	5.8	1.3
Ethylbenzene (mg kg^{-1})	ND	1.6	0.5
Total xylenes (mg kg^{-1})	ND	11.3	3.5
F1 C_6 - C_{10} (mg kg^{-1})	ND	90.3	116.3
F2 C_{10} - C_{16} (mg kg^{-1})	ND	642.0	1470.0
F3 C_{16} - C_{34} (mg kg^{-1})	ND	1456.7	1810.0
F4 C_{34} - C_{50} (mg kg^{-1})	ND	578.7	548.0
Boron (mg kg^{-1})	0.1	5.1	9.6
Mercury (mg kg^{-1})	< 0.0002	0.1	0.1
Antimony (mg kg^{-1})	< 0.004	0.0	0.0
Arsenic (mg kg^{-1})	0.0	11.5	11.8
Barium (mg kg^{-1})	0.3	9430.0	4803.3
Beryllium (mg kg^{-1})	< 0.002	0.6	1.0
Cadmium (mg kg^{-1})	< 0.0002	0.2	0.2
Chromium (mg kg^{-1})	0.1	31.6	46.7
Cobalt (mg kg^{-1})	0.004	8.2	11.8
Copper (mg kg^{-1})	0.1	47.7	39.0
Lead (mg kg^{-1})	0.04	42.4	21.7
Molybdenum (mg kg^{-1})	< 0.02	4.0	4.0
Nickel (mg kg^{-1})	0.01	28.3	38.2
Selenium (mg kg^{-1})	< 0.004	1.03	1.03
Silver (mg kg^{-1})	0.02	0.6	0.5
Thallium (mg kg^{-1})	< 0.001	0.2	0.3
Tin (mg kg^{-1})	< 0.02	1.0	< 1.00
Uranium (mg kg^{-1})	0.01	0.9	1.3
Vanadium (mg kg^{-1})	0.1	37.4	60.9
Zinc (mg kg^{-1})	0.1	68.0	74.0

ND = not determined.

Table 5.4. Viable counts of specific anaerobic bacteria groups in drilling fluids, control soils and soils with drilling fluid.

		Raw PSDF			Spent Once PSDF			Spent Twice PSDF		
		Sulphate Reducer	Nitrate Reducer	Iron Reducer	Sulphate Reducer	Nitrate Reducer	Iron Reducer	Sulphate Reducer	Nitrate Reducer	Iron Reducer
		---------- Number g^{-1} ----------								
	PSDF	ND	ND	ND	ND	ND	ND	6.9×10^2	1.80×10^1	2.10×10^2
Clay Loam	Control	2.0×10^1	5.01×10^2	5.01×10^2	2.0×10^1	5.01×10^2	5.01×10^2	2.0×10^1	5.01×10^2	5.01×10^2
	Plus 40	ND	2.84×10^3	2.10×10^1	ND	1.68×10^6	4.73×10^2	ND	1.16×10^3	2.42×10^2
	Plus 120	ND	1.01×10^6	8.60×10^1	5.5×10^1	4.71×10^4	2.52×10^2	2.2×10^1	3.68×10^4	3.68×10^2
Loam	Control	2.4×10^1	2.52×10^4	1.13×10^5	2.4×10^1	2.52×10^4	1.13×10^5	2.4×10^1	2.52×10^4	1.13×10^5
	Plus 40	ND	4.31×10^5	2.95×10^5	6.1×10^1	$> 1.97 \times 10^6$	1.13×10^6	ND	7.69×10^4	4.27×10^4
	Plus 120	ND	4.29×10^4	7.03×10^5	2.7×10^1	1.26×10^5	4.70×10^5	8.9×10^1	5.60×10^5	6.38×10^4
Sand	Control	ND	2.77×10^2	2.66×10^3	ND	2.77×10^2	2.66×10^3	ND	2.77×10^2	2.66×10^3
	Plus 40	ND	1.19×10^4	2.93×10^3	ND	$> 1.68 \times 10^6$	3.47×10^3	ND	4.58×10^5	1.38×10^4
	Plus 120	ND	$> 1.82 \times 10^6$	7.87×10^3	ND	$> 1.88 \times 10^6$	8.09×10^3	ND	3.95×10^5	1.24×10^4

Rate 40 and 120 = PSDF loading rate at 40 and 120 m^3 ha^{-1}
ND = not detected.

Table 5.5. Colony forming units of microorganisms in soil with PSDF.

		Raw PSDF		Spent Once PSDF		Spent Twice PSDF	
		Bacteria and Actinomycete (Number g^{-1})	Fungi (Number g^{-1})	Bacteria and Actinomycete (Number g^{-1})	Fungi (Number g^{-1})	Bacteria and Actinomycete (Number g^{-1})	Fungi (Number g^{-1})
PSDF		ND	ND	1.0×10^{4}	2.5×10^{1}	7.3×10^{5}	ND
Clay Loam	Control	5.5×10^{4}	1.0×10^{2}	5.5×10^{4}	1.0×10^{2}	5.5×10^{4}	1.0×10^{2}
	Plus 40	5.3×10^{5}	9.2×10^{2}	1.5×10^{7}	1.1×10^{2}	1.0×10^{5}	4.5×10^{2}
	Plus 120	3.8×10^{6}	3.8×10^{3}	1.2×10^{6}	5.6×10^{3}	9.4×10^{6}	1.9×10^{3}
Loam	Control	9.6×10^{5}	2.6×10^{2}	9.6×10^{5}	2.6×10^{2}	9.6×10^{5}	2.6×10^{2}
	Plus 40	8.7×10^{5}	2.5×10^{3}	1.8×10^{6}	7.6×10^{3}	1.2×10^{6}	4.6×10^{3}
	Plus 120	1.2×10^{6}	2.0×10^{3}	9.1×10^{5}	4.7×10^{3}	2.2×10^{6}	1.3×10^{4}
Sand	Control	6.7×10^{5}	7.2×10^{1}	6.7×10^{5}	7.2×10^{1}	6.7×10^{5}	7.2×10^{1}
	Plus 40	1.1×10^{6}	7.6×10^{2}	1.1×10^{7}	7.9×10^{3}	6.7×10^{6}	7.2×10^{2}
	Plus 120	8.8×10^{6}	6.5×10^{2}	1.1×10^{7}	1.5×10^{3}	8.2×10^{6}	8.5×10^{1}

Rate 40 and 120 = PSDF loading rate at 40 and 120 m^{3} ha^{-1}.
ND = not detected.

Table 5.6. Presence or absence of microbial isolates in PSDF and soil.

Treatment	Raw PSDF	Spent Once PSDF	Spent Twice PSDF	Loam		Clay Loam		Sand	
				Control	PSDF	Control	PSDF	Control	PSDF
Bacteria									
Achromobacter spp.	–	–	–	+	+	+	+	+	+
Agrobacterium spp.	–	–	–	–	+	–	–	–	–
Bacillus spp.	–	–	+	+	+	+	+	+	+
Providencia spp.	–	–	–	–	+	–	–	–	–
Pseudomonas spp.	–	–	–	–	+	–	–	–	+
Rhodococci spp.	–	+	+	–	–	+	+	+	+
Actinomycete	–	–	+	+	+	–	+	+	–
Fungi									
Aspergillus spp.	–	+	–	–	+	+	+	–	+
Ascomycota spp.	–	–	–	–	–	+	–	–	–
Aureobasidium spp.	–	–	–	–	–	–	–	–	+
Cephaliophora spp.	–	–	–	–	–	–	+	–	–
Cephalosporium spp.	–	–	–	+	+	–	–	–	–
Cladosporium spp.	–	–	–	–	–	+	+	–	–
Fusarium spp.	–	–	–	–	–	-	+	–	–
Gonatobotrys spp.	–	–	–	–	–	–	–	+	+
Penicillium spp.	–	–	–	–	–	–	–	–	+
Trichoderma spp.	–	–	–	+	+	–	–	+	+

– = absent.
+ = present.

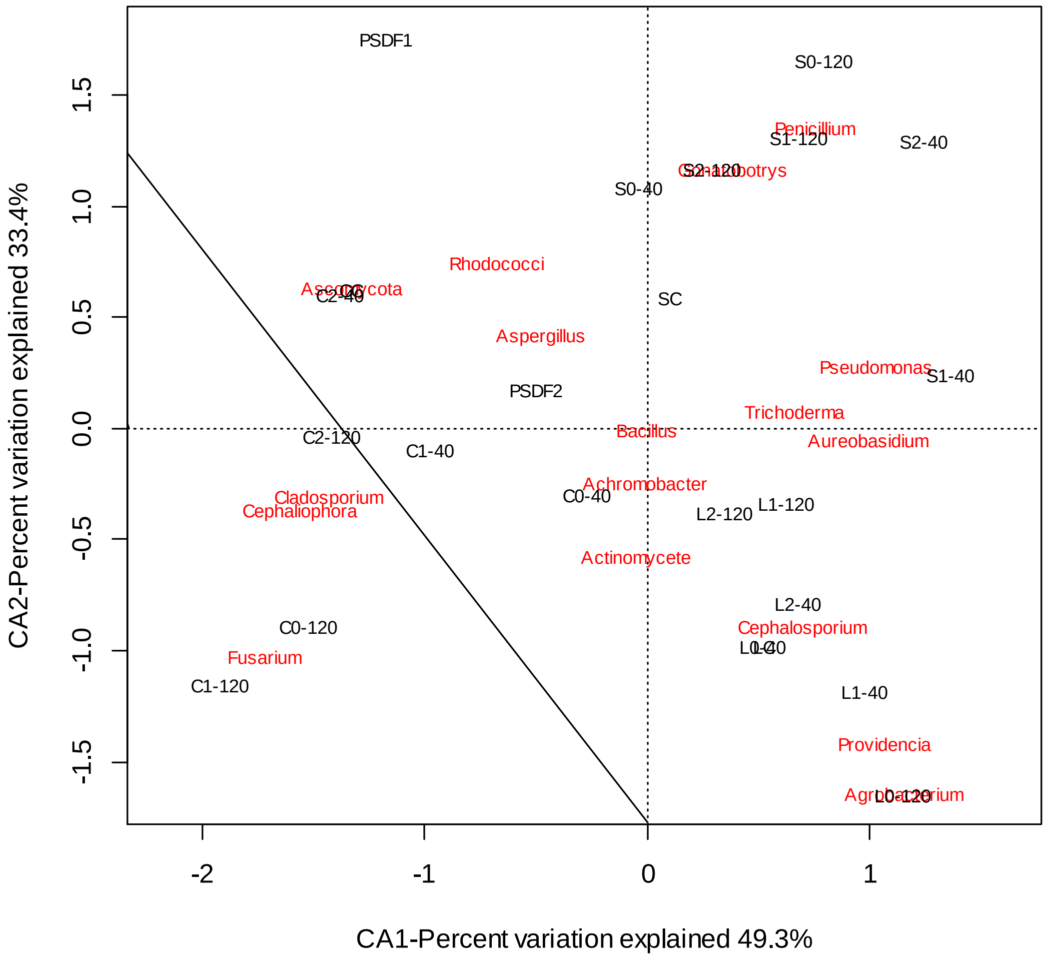

Figure 5.1. Correspondence analysis of microbial communities in PSDF and soil. C, L and S = clay loam, loam and sand soil. 0, 1 and 2 = raw, spent once and spent twice PSDF. 40 and 120 = PSDF loading rate at 40 and 120 m^3 ha^{-1}.

CHAPTER 6. SYNTHESIS AND FUTURE RESEARCH

1. Research Summary

Potassium silicate drilling fluids (PSDF) are relatively new water based mud systems developed to reduce environmental impacts. Sodium concentrations in traditional drilling fluids were considered to have the most detrimental environmental impact; thus replacing sodium with potassium in newer drilling fluids would reduce concentrations of sodium. Hypothetically, high concentrations of potassium in PSDF could serve as a nutrient amendment for land reclamation, thus not only would land disposal be practical, but also provide benefits for reclamation. Objectives of this study were to assess PSDF properties and effects on vegetation, soil and water, and determine the impacts of soil texture, plant species and PSDF recycling times, disposal method and rate. Application of spent PSDF on slightly acidic and medium textured soils at controlled rates resulted in positive changes to soil physical, chemical and microbiological properties and enhanced plant establishment, development and productivity.

In the first greenhouse experiment, the effects of fresh raw PSDF on four different textured soils, a selected agricultural, barley *(Hordeum vulgare* L.) and a native grass species, slender wheat grass *(Agropyron trachycaulum* (Link) Malte ex H.F. Lewis) with two application methods at four rates were evaluated. By using raw drilling fluid, the influence of the soils being drilled could be removed, and the impact of the drilling fluid itself could be more clearly interpreted. Raw PSDF had no detrimental effects on soil and vegetation at rates ≤ 45 m^3 ha^{-1}. Available potassium for plants increased significantly with PSDF. Barley and wheat grass plants survived and developed through physiological growth stages in different textured soils with sprayed and incorporated PSDF. Fertilizer had a significant effect on vegetation. Raw PSDF as a land reclamation amendment will likely give best results on acidic and well drained soils with inorganic fertilizer.

In the second greenhouse experiment, the impacts of spent and raw PSDF on soils and plants were studied. PSDF at high concentrations had some positive effects on soil and plants. PSDF significantly increased available potassium, ratio of soluble potassium to sodium, soil sodicity alleviation and cation exchange capacity. PSDF at ≤ 60 m^3 ha^{-1} rates increased barley productivity under greenhouse conditions. Effect of PSDF type or application method on vegetation was insignificant. Soil texture had a significant effect on vegetation. PSDF on slightly acidic and medium textured soil will have maximum benefits from high potassium in PSDF to soil properties and plants growth. These results provide important implications for land disposal of PSDF and its use in reclamation.

In a laboratory column experiment, changes in soil and hydrologic properties following PSDF application were investigated. Hydraulic conductivity and field capacity were reduced and available potassium increased with PSDF. Incorporated PSDF in the upper 10 cm of soil accelerated PSDF element transport through soil columns to leachate. Loam soil with PSDF had better physical and chemical properties than those of sand soils. No detrimental effects on soil physical and chemical properties and leachate quality were detected when applying PSDF at rates ≤ 60 m^3 ha^{-1}.

Influence of PSDF application on soil microbial community was studied in the laboratory. PSDF stimulated microbial populations and increased diversity of the microbial community relative to soils without PSDF. Organic components in PSDF and petroleum hydrocarbon from the drilling process will be the main food supply resources for microorganisms other than inorganic substrates in PSDF. The drilling process with increasing recycling times could increase the opportunity to introduce exogenous microorganisms into drilling fluids. Initial soil texture, pH and nutrients can have a significant effect on microbial community structure in treatments with PSDF.

2. Implications For Reclamation And Management

Use of PSDF in land reclamation, provides a practical and economic solution to excess disposal in overcrowded landfills. Applied PSDF at rates ≤ 60 m^3 ha^{-1} to slightly acidic and medium textured soil for reclamation will likely give the best response of soil properties and plant growth. Wheat grass and barley could respond well to PSDF for reclamation under various end land uses, such as pastures and cultivation. Inorganic nitrogen and phosphorus fertilizer is necessary for successful PSDF land reclamation. Incorporating PSDF in soil will likely be more beneficial for vegetation than spraying it with elevated soil water content and macronutrients, but if PSDF application rate is higher than 120 m^3 ha^{-1}, incorporating PSDF will increase the risk of soil salinity in the upper 10 cm and reduce leachate quality relative to spraying. The effects of number of recycling times of PSDF on plant establishment and development were various, but increasing recycling times could increase the opportunity to introduce exogenous microorganisms into drilling fluids and increase petroleum hydrocarbon as energy resources for soil microorganisms.

3. Research Scientific Knowledge Base

This study has helped to advance the scientific knowledge base by filling some of the gaps in the current research. The results have helped expand information on PSDF addition to different types of soils and their potential impacts on soil and vegetation properties. Studying each parameter separately has helped increase the knowledge base for soil physical, chemical and microbiological properties as well as plant establishment and development through physiological growth stages. Using these individual parameters as indicators of land reclamation has helped address the interrelationships among the soil-plant-water systems and discuss the effectiveness of PSDF as a reclamation amendment. This study has provided findings for using PSDF in land reclamation on fine and coarse, alkaline and acidic soils, with and without inorganic fertilizers, with incorporated and sprayed PSDF application method at lower or higher than regulated rates. This research also provided detailed information on the improvements of macronutrient potassium from PSDF application, which were limited studies in previously documented.

4. Limitations Of The Research

The site specific soil chemical, physical and microbiological properties result in different responses of vegetation to PSDF. This variability may generate different soil-water-plant effects with PSDF disposal. This study has covered three to four typical soil textures with potential for PSDF disposal in Alberta, but it could have included more soil types.

Due to budget limitations, we only did greenhouse experiments for one crop and one native grass species. The effects of PSDF on plant community succession and sustainability were not assessed. At the beginning of the research, based on the limited information about drilling waste to soil and plants, PSDF environmental impacts were unknown, therefore we only can conduct greenhouse experiments. Now with the known positive responses from soil and vegetation to PSDF in our study which support drilling fluid disposal regulations (Alberta Energy Resources Conservation Board 2012), plant community establishment with grasses, woody species, forbs and trees will be possible to evaluate in the field over various time periods.

Plant responses to high potassium PSDF could be studied in the future. In our research, a direct positive relationship between plant and potassium availability in soil was detected. High potassium will improve plant salt tolerance (Chen et al. 2007, Taiz and Zeiger 1998). Since sodium causes one of most detrimental environmental effects from drilling fluids, replacing sodium with potassium in PSDF has verified improved soil potassium fertility. However, scale is a limitation, and our plants harvested in the greenhouse were too small to do plant tissue test of uptaken elements. In the future this could be possible to measure in the field with large scale experiment, to fully explain the mechanism of plant positive response to PSDF.

5. Future Research

This research provided information on PSDF effects on soil, plant and water properties at a greenhouse and laboratory meso scale. Future research should build upon these results and study these impacts in the field. Since each disposal site provides unique challenges, such as soils, vegetation and meteorological conditions, various sites should be studied. The field experiment should be conducted focusing on whether disposal of PSDF at different rates will affect soil physical, chemical and microbiological properties and plant establishment and development.

This research delivered results on short term responses of the soil-plant-water system to PSDF application. Long term effectiveness of soil macronutrients especially potassium, impacts on salinity and the elements uptaken by plants, changes in plant community structure, composition and function should be investigated. Different disposal method effects on plant community succession over the long term should also be addressed. Other amendments to ameliorate sodium related issues in clay loam soils with PSDF, and water availability related to organic matter content in soil with PSDF should also be studied.

This research provided information about the soil microbial community population and diversity change with PSDF, but did not study the direct relationship between soil microbial properties and vegetation growth. Future study is needed to clarify the importance of microorganisms to soil property relationships, functional diversity and impacts on vegetation establishment and development with PSDF.

6. References

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