

SUPERABSORBENT POLYMER EFFECTS ON SOIL PHYSICAL PROPERTIES AND  
USE AS A COMPACTION ALLEVIATION AMENDMENT IN SURFACE COAL MINE  
RECLAMATION

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**Title**

Superabsorbent polymer effects on soil physical properties and use as a  
compaction alleviation amendment in surface coal mine reclamation

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## ABSTRACT

Surface coal mine reclamation is challenged by alterations in soil characteristics, compaction being the most plant-yield reducing. Superabsorbent polymers (SAPs) ability to retain large volumes of water gives them the potential to alter soil properties. Laboratory objectives were to determine how SAPs alter water retention, liquid limit (LL), evaporation, saturated hydraulic conductivity (Ksat), and compression across five soil series. Increasing SAP application rates to 0.2% significantly increased plant available water (PAW), stage one evaporation duration, LL, stage two evaporative water loss, and significantly decreased stage one evaporative water loss, Ksat, and compression for various soil series. Field study investigated how SAP, deep ripping (R) and mulch (M) impacted penetration resistance and spring wheat yield (*Triticum aestivum*). Application of 0.04% SAP improved yields similar to R. Penetration resistance decreased with R, and SAP application showed reduction similar to R. While SAPs show potential as a reclamation tool, application longevity needs evaluation.

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## GENERAL INTRODUCTION

As anthropogenic disturbances increase so does the importance of efficient reclamation. In surface coal mining the removal and replacement of soil with heavy machinery often results in a compacted layer that restricts roots from accessing nutrients and water, (Batey, 2009), which further translates to reductions in plant productivity (Hetzler and Darmody, 1992; Dunker et al., 1995). The Surface Mining and Control Act places mine land into a performance bond for a minimum of 10 years or until reclamation requirements, such as, returning land to equal or greater productivity, are satisfied (SMCRA, 1977). Natural processes can take decades to improve soil properties (Bradshaw, 1997), and compaction greater than 1 m deep can remain for longer than 30 years in reclaimed mine land (Spoor, 2006, Batey, 2009). Thus, focus on research that better alleviates compaction will undoubtedly improve soil-water dynamics and plant growth and health.

The continual evaluation of reclamation tools and techniques to reduce the time of successful reclamation is needed. Inevitably, reconstruction of soil with heavy machinery leads to a layer that is compacted. This compaction is persistent and must be broken up mechanically. Deep ripping or subsoiling is the primary method used to alleviate compaction. These methods have been able to reduce compaction metrics (penetration resistance and bulk density) to a depth of 114 cm, which further translated to improved productivity. In the early years of reclamation, however, productivity is still low compared undisturbed sites and often times subsoiling will be necessary again, as re-setting has occurred.

Superabsorbent polymers (SAPs) are materials that have the ability to absorb and retain large volumes of water relative to their mass. These materials can increase plant available water (PAW) and potentially reduce compaction (Bai et al., 2010). Natural freeze-thaw cycles have shown to not be effective at reducing compaction (Bohrer et al., 2017), but amending soils with SAPs helps reduce compaction (Bai et al., 2010), along with improving soil-water characteristics that improve plant productivity and plant health.

## **THESIS ORGANIZATION**

This thesis consists of three chapters and is organized in a manuscript format. Chapter 1 is a literature review of surface coal mine soil disturbance and reclamation, as well as, the common issues faced with reclamation. This review outlines the history and standards of reclamation, as well as, how soil is altered during the mining process. Chapter 2 is a literature review regarding the benefits and uses of SAPs. The third chapter, "Superabsorbent polymer influence on soil properties as a function of various soil textures and application rates," highlights the ability of SAPs to change soil properties such as water retention, LL, evaporative loss, compression and saturated hydraulic conductivity. Chapter 4 is titled "Reducing coal mine re-spread compaction in North Dakota using tillage, M and a superabsorbent polymer" and examines the short-term success of tillage treatments (chisel/disc, deep ripping), soil amendments (M, SAP) and their combinations on penetration resistance and yield. This study provides insight into the potential use of SAPs as a tool to alleviate compaction at the field scale.

## **CHAPTER 1. SURFACE COAL MINING IN NORTH DAKOTA: A BRIEF OVERVIEW**

### **Introduction**

The 1977 Surface Mining Control and Reclamation Act (SMCRA) was the first comprehensive federal legislation to regulate the impacts of surface mining at the national level (Hamlet, 1980). The goal of the SMCRA was to mitigate negative effects of surface coal mining and set standards for land disturbed by coal mining to be returned to some level of pre-mine conditions (aesthetic and functionality) (Hamlet, 1980). The SMCRA established the Office of Surface Mining Reclamation and Enforcement (OSMRE) to directly enforce mining laws and cleanup abandoned mines. Today, OSMRE oversees state regulatory programs and develops new tools to improve reclamation. Prior to 1969, the state of North Dakota did not require reclamation, which resulted in spoil piles and surface cuts left on the landscape (USGS, n.d.). Strip mine regulation began in 1970 and 1971 and today is regulated by the Public Service Commission and the Department of Environmental Quality for North Dakota and Montana, respectively (NDPSC, 2009; MTDEQ, 2019).

North Dakota mining operations are conducted in a manner that protects the environment, public interest, and rights of property owners; returns land to a beneficial use, and restores productivity of agricultural land to premine levels (NDPSC, n.d.). Land that had been disturbed by surface coal mining is held under a performance bond by the state regulatory authority. Pre-defined performance standards must be met in order for land to be released and operators' responsibility to end. Prior to mining an application for mining requires extensive information on plans for mining process and soil reclamation. Before mining, the permitted area is soil surveyed by a professional soil classifier and prime soils identified, furthermore, productivity of prime farmlands, and average yields under high levels of management are determined (NDPSC, 2009). A monitoring plan is put in place to ensure protection of water quality during mining and the reclamation process, such that land disturbed by surface coal mining can be restored to support the uses prior to mining or better uses (NDPSC, 2009). Once coal has been extracted the area is backfilled, compacted, and graded to reshape all areas affected by surface coal mining operations to the gentlest topography consistent with unmined landscape to develop a post-mining landscape that provides maximum moisture retention, drainage that will complement surrounding

landscape, minimize erosion and support vegetation (NDPSC, 2009). Along with stabilizing and protecting all surface areas, air and water pollution need to be monitored (NDPSC, 2009).

Prime farm land classification refers to land that has soil characteristics and moisture supply to efficiently produce high yields when managed. Historically, prime farmland has been used for intensive agricultural purposes and is large enough in size to constitute a viable economic unit (NDPSC, 2009). The soil layer(s) to be removed will be determined by the pre-mine survey which may require segregation of suitable plant growth material (soil material containing A and B horizons and if approved, portions of the C horizon) (SPGM) in two or more layers, if material cannot be re-spread immediately material shall be stockpiled and stabilized with quick-growing plants to protect SPGM from erosion (NDPSC, 2009). Prime farmland classification requires that 1.22 m, or the thickness of the original soil profile down to subsurface horizon which inhibits root penetration, whichever is shallower, be replaced (NDPSC, 2009). The final surface soil layer, the topsoil, must be the approximate average thickness of materials saved (NDPSC, 2009).

Since 1975 there have been 1.1 million ha of reclaimed coal land in the United States (NMA, 2017). Of those reclaimed hectares, about 24,000 have been in North Dakota (D. Moos, personal communication, North Dakota Public Service Commission, Bismarck, ND, 2018). Reclamation is a resource intensive process that on average costs \$49,400 per ha (D. Moos, personal communication, North Dakota Public Service Commission, Bismarck, ND, 2018). Land permitted is held for a minimum of ten years under a performance bond by the state regulatory authority. With the scope and scale of surface mining continuing to grow, it is essential to understand how to optimize the reclamation process.

Even with advancing technology and knowledge there is inevitability going to be challenges to reclamation. Disturbance impacts the landscape, which in turn impacts ecosystem function (Lima et al., 2016). Ecosystem function is altered by changes in physical, chemical, and biological components resulting from the disturbance. Removal and replacement of soil is the primary disturbance in surface mining and collectively addressing changes in soil physical, chemical, and biological properties guides reclamation experts to improve soil function and its productivity potential (Figure 1).

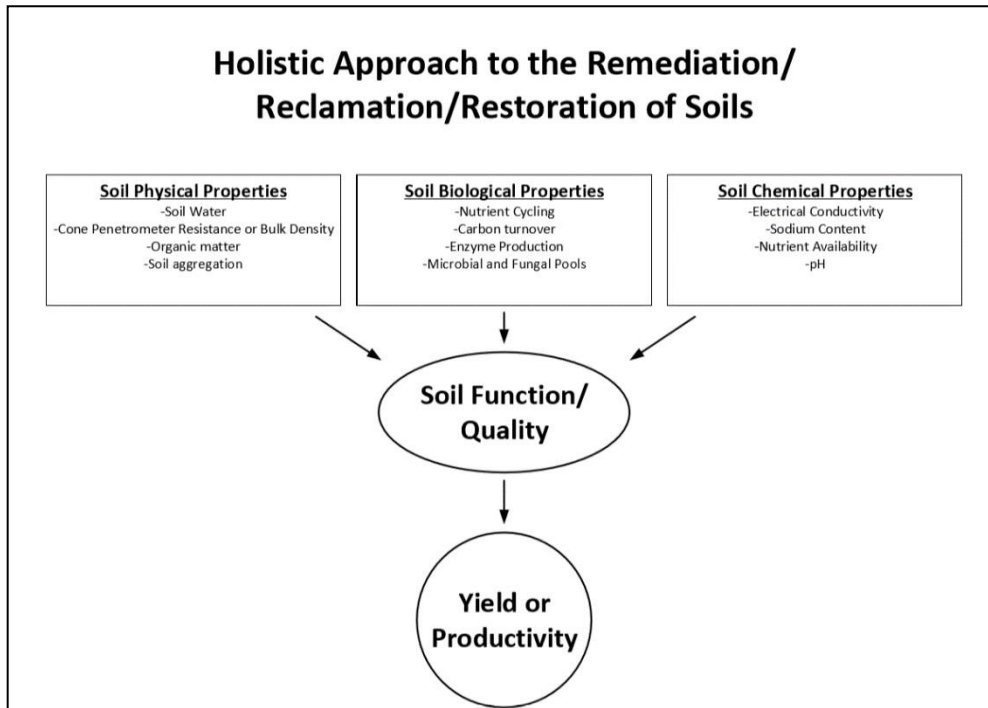


Figure 1. A holistic approach to remediation, reclamation and restoration of soils. Adapted from Barnhisel et al. (1992).

One of the major problems faced by the coal industry in reclaiming land to original productivity is compaction. The reconstruction of soil with heavy machinery can lead to a layer that is compacted to the degree that it is root restricting. The compacted post-mining soil has less pore space, which impacts soil function (Figure 2). Traditionally, tillage has been the primary method to alleviate compaction. In North Dakota the majority of research was performed by the Land Reclamation Research Center in the 1970s. As operations have continued to grow and new technology has become available, reevaluating and improving reclamation methods is critical. Coal mineland reclamation has been occurring since 1977 but limitations in soil function and recovery are still prevalent. Soil disturbance, reclamation standards, and mining and the reclamation process will be reviewed, as well as, what areas of reclamation need further research.

### **Mining Process**

After exploration and pre-mine surveying, the mining process begins. Suitable plant growth material (SPGM) is the soil material containing A and B horizons and if approved, portions of the C horizon. The SPGM is removed in two lifts by shovel or scraper; the first lift is classified as topsoil and second lift as subsoil. The two lifts (topsoil and subsoil) are handled separately throughout the mining

process (NDPSC, 2009). Both SPGM lifts are re-spread directly on previously mined areas or stockpiled for later re-spread. The material immediately under the SPGM overlying the coal is referred to as the overburden, which is removed by truck-shovel and subsequently by dragline. Overburden is removed to access underlying coal. After overburden is removed it is commonly called “spoil.” Lastly, the lignite coal is broken into smaller pieces and transported for electrical generation.

**Reclamation Process**

The primary goals of the reclamation process are to return the land to equal or greater productivity and to protect the environment. Environmental stewardship has become a pride for mining companies as the scale of mining operations and environmental awareness has increased. The reclamation process typically follows a five step process: (1) grading of spoil to approximate original contour, (2) re-spread subsoil and topsoil, respectively, (3) seedbed preparation (tillage, harrow, rock picking, seeding), (4) monitoring and, (5) bond release (BNI, 2019). It is important to note that the reclamation process starts before mining begins. Pre-mining analysis and survey are necessary for successful reclamation. The SPGM is used to guide the soil re-spread process in regards to the thickness of the soil re-spread. If an area has low quality SPGM or overburden (spoil), SPGM will be re-spread at a greater depth. Reclamation problems often develop when material with undesirable characteristics are deposited near the surface or mixed with surface materials. Thus, if undesirable characteristics can be recognized before mining, problems can be addressed to increase the success of the reclamation (USDA-NDAES Staff, 1977).

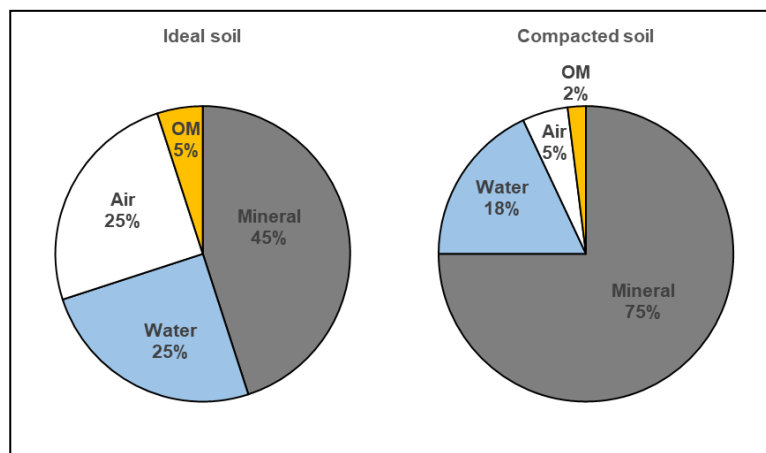


Figure 2. Ideal soil composition prior to mining and compacted soil as a result of mining on a volume basis.

## **Effects of Surface Mining on Soil Physical Properties: A Brief Overview**

Disturbance of soil physical properties is largely unavoidable as soil is removed to access coal, and reconstructed during the reclamation process. Material handling and reconstruction impacts the physical condition of soil (Dunker and Vance, 1992) and even with the segregation of soil layers there are changes to soil properties (Underwood and Sutton, 1992). When soil physical properties are impaired, such as with high levels of compaction, this can lead to reduced soil function, and furthermore reduced productivity (Figure 1). This overview will examine soil handling, how research has guided regulation requirements, the severity of compaction, methods to reduce or alleviate compaction, and how compaction influences soil-water dynamics and productivity.

### **Soil Handling and Reconstruction**

As a result of handling and reconstruction, a post-mine soil has drastically different properties than its pre-mine counterpart. To minimize the effects of reconstruction, reclamation requirements have evolved to address quality of spoil, topsoil depth, total respread depth, and mixing. Three studies were conducted in North Dakota to look at how depth of topsoil would impact plant productivity; two studies constructed wedges and one excavated a trench. The Stanton wedge, Zap double wedge, and Falkrik trench experiments were conducted in between 1974 and 1982 and provided valuable information on soil reconstruction and material handling. In Stanton, over a sodic spoil (SAR = 25), a wedge of subsoil or a 3:1 mixture of subsoil:topsoil were constructed to a peak height of 2 m, and left covered with subsoil or the 3:1 mixture, or capped with 20 or 61 cm of non-sodic topsoil; three replications were performed (Figure 3a). Planted to each of these treatments were three perennial forage species or spring wheat; the productivity of each was evaluated over three years (Doll et al., 1984). Generally, production of alfalfa (*Medicago sativa*), crested wheatgrass (*Agropyron cristatum*), and spring wheat increased with total respread depth, until midslope (halfway to wedge summit), which was attributed to higher soil moisture levels. Relative yields were determined as the percentage of highest average yield for each treatment over three years, where spring wheat had 74, 100, 98 and 83% relative yield on 0, 20, 61 cm of topsoil and 3:1 mixture of subsoil:topsoil, respectively (Doll et al., 1984).



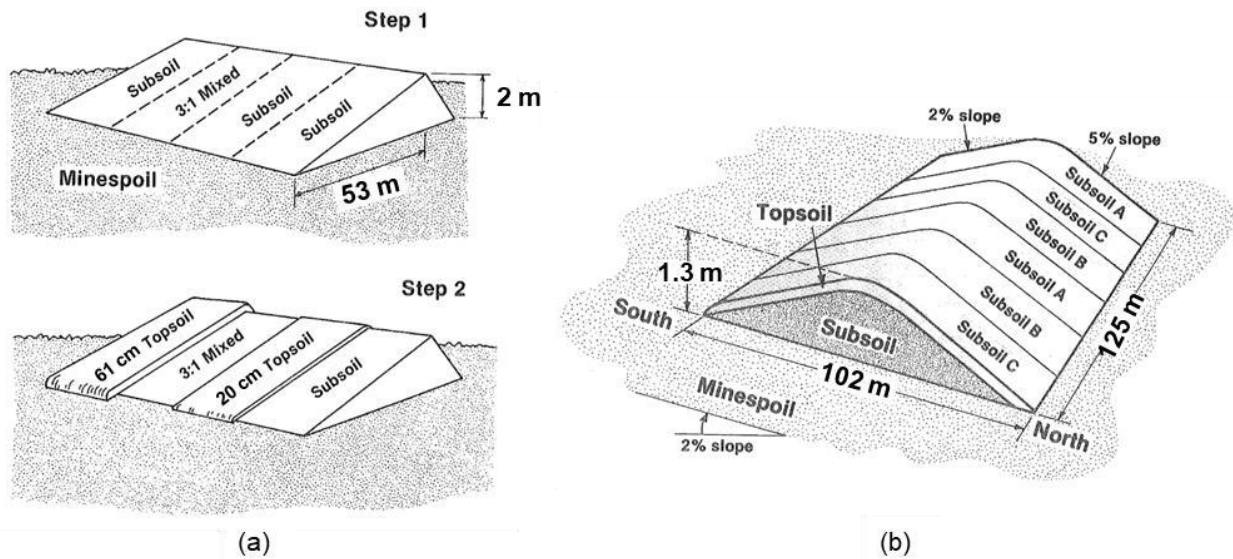


Figure 3. (a) One replication of the Stanton wedge experiment, and (b) Zap double wedge reconstructed soil. From Doll et al. (1984).

The Zap double wedge experiment shaped three subsoil materials of different quality (salinity, sodium, texture) into a 0 to 1.3 m thick wedge, with a slope of 1-2% and 5-6% for respective sides, and topsoil was uniformly respread to a 25 cm depth (Figure 3b) (Doll et al., 1984). Results were that finer textured subsoils (clay content >33%), even if they were saline ( $EC = 4$  to  $7 \text{ dS m}^{-1}$ ) or sodic ( $SAR = 5$  to  $6$ ), would be more productive than coarse textured subsoil (clay content = 13%) over a moderately saline ( $EC = 5 \text{ dS m}^{-1}$ ) moderately sodic ( $SAR = 15$ ) silty clay spoil (clay content = 46 %) (Doll et al., 1984). The properties of spoil or the material underlying SPGM can influence SPGM chemical properties depending on texture.

Another experiment was conducted at Falkrik where eight trenches were dug to a 4.5 m depth and filled with four types of overburden or spoil (clay loam, silty clay loam, gravelly loam sand overlain by clay loam subsoil, gravelly loamy sand) and covered with 23, 45, or 68 cm of topsoil to reach original soil surface elevation (Figure 4). After one growing season, wheat yield increased with increasing topsoil thickness, however, yields were not statistically different for topsoil respread depth over the clay loam overburden (spoil), which was attributed by the authors to its greater content of clay (Doll et al., 1984).

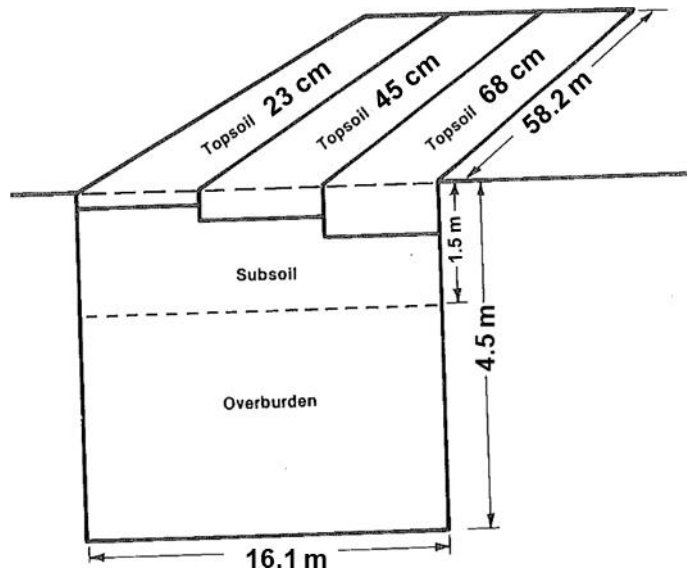


Figure 4. Falkirk trench experimental cross section. From Doll et al., (1984).

These three historical experiments provided necessary information to revise the then-existing regulations, such that required replacement depth would be dependent on soil texture, SAR, and EC (Table 1).

Table 1. Depth of soil replacement depending on spoil properties. Adapted from Doll et al., (1984).

Spoil Properties			Depth of soil replacement		
Texture	EC	SAR	Topsoil	Subsoil	Total
	dS m <sup>-1</sup>		-----cm-----		
Coarse <sup>†</sup>	<6	<12	30	61-76	91-106
Medium <sup>‡</sup>	<6	<12	30	30-46	61-76
---*	---	12-20	30	61-91	91-122
---*	---	>20	30	91-122	122-152

<sup>†</sup> Sandy loam or coarser texture

<sup>‡</sup> Loam or finer texture

\* Not applicable, SAR dominant property

The primary issues when reclaiming land are compaction, and long-term differential settling (D. Moos, personal communication, North Dakota Public Service Commission, Bismarck, ND, 2018).

Requirements were put in place to combat degradation of SPGM quality, such that if spoil was acidic, saline, sodic or coarse textured a greater depth of soil material would be replaced (Doll et al., 1984).

Today the common SPGM respread packages in North Dakota are 61, 91, or 122 cm of total depth replaced with 23-30 cm being topsoil. Early regulation (pre-1969) only required spoil to be leveled such that slopes were less than 25%, but regulations evolved in 1973 to the removal and replacement of

topsoil (Dodd et al., nd). Today, topsoil and subsoil are handled separately throughout the mining process, and mixing is prohibited unless approved by the North Dakota Public Service Commission (NDPSC, 2009). Mixing of SPGM and lower quality (saline, sodic, coarse texture) horizons may result in degradation of reclaimed soils (Semalulu and Barnhisel, 1992) with biological and chemical properties being negatively impacted (Croat et al., 2018). However, in surface coal mining, compromised soil physical properties are the predominant problems for reclamation.

### **Compaction**

Compaction is one of the most common changes in soil physical properties caused by surface coal mining (Henning, 1992; Simmons et al., 1992; Wells and Barnhisel, 1992). Penetration resistance (PR) and bulk density (BD) are common metrics of compaction, both of which correlate to soil strength. For example, on an undisturbed native site in China having a sandy loam texture, BD for the 0 to 15 cm and 15 to 30 cm depth increments ranged from 1.26 to 1.32 g cm<sup>-3</sup> and 1.35 to 1.43 g cm<sup>-3</sup>, respectively, and BD values on a reclaimed mine site nearby of similar soil texture significantly increased from 1.34 to 1.58 g cm<sup>-3</sup> and 1.56 to 1.68 g cm<sup>-3</sup>, respectively (Zhang et al., 2016). Similarly, on a clay loam soil, reconstructed soil PR was 919 kPa, which was significantly higher than plots receiving compaction alleviation by deep ripping to a depth of 40 to 45 cm, whose PR values ranged from 389 to 624 kPa (Bateman and Chanasyk, 2001). In a 13-year-old reclaimed pastureland located in Mississippi, with a silt loam texture, PR at the 15 cm depth was 3,000 kPa, which was significantly greater compared to a nearby unmined pasture site that had a PR of 2,500 kPa (Adeli et al., 2019). The reconstructing of a soil causes compaction, which is documented above as having greater PR and BD values, relative to non-mined sites.

In an effort to reduce compaction created by the respreading process, various equipment types and methods have been evaluated to lessen the initial compaction status of the soil. For example, using the mining wheel conveyor spreader replacement method, PR values ranged from 647 to 1,628 kPa between the 23 to 110 cm depth and the shovel and scraper method had significantly greater PR values (1,905 to 3,709 kPa) at the same depth (Thompson et al, 1987). Bulk density values in this same experiment averaged 1.55 g cm<sup>-3</sup> for the mining wheal conveyor method and 1.70 g cm<sup>-3</sup> for the shovel and scraper method (Thompson et al., 1987). Similarly, when subsoil was replaced by scrapers, PR

values down to 112 cm were greater than PR values after a truck replacement method, but these methods were not statistically different (Hooks and Vance, 1992). Even though replacement methods in North Dakota changed from using scrapers to end-dump trucks and shovels, the Land Reclamation Research Center concluded that there were no significant differences in compaction levels between the scraper method and end-dump truck/shovel method (D. Moos, personal communication, North Dakota Public Service Commission, Bismarck, ND, 2018). While replacement methods have been researched, findings are inconsistent or non-significant to justify a specific method.

As the reclamation process begins, reconstructed soil is in a compacted state that needs to be addressed mechanically. The use of subsoilers, such as chisel plows and deep rippers, are common compaction alleviation methods used by mine personnel (D. Moos, personal communication, North Dakota Public Service Commission, Bismarck, ND, 2018). For example, a double pass of ripping with a Kello-Bilt 5000 series subsoiler with a working depth of 40 to 45 cm resulted in PR being reduced from 919 kPa to 389 kPa on a clay loam soil (Bateman and Chanasyk, 2001). Bledsoe (1992) determined that BD was significantly reduced from 1.7 to 1.5 g cm<sup>-3</sup> at the 20-40 cm depth when tillage working depth was 40 cm, and BD at the 40-60 cm and 60-80 cm depths were significantly reduced from 1.80 to 1.55 g cm<sup>-3</sup> and 1.83 to 1.68 g cm<sup>-3</sup> for an 80 cm working depth. Deep ripping (vibratory action), deep ripping (cut lift action), and deep plowing with working depths of 80, 80 and 120 cm, respectively, significantly reduced the average PR for the 23 to 114 cm depth, from 3,630 kPa to 2,100-1,410 kPa and reduced soil strength values remained five years after tillage application (Dunker et al., 1995). Lastly, even after 30 yr, freezing and thawing did not reduce PR between the 15 and 45 cm soil depths at mine-sites in North Dakota, however, though not significant, PR below 45 cm increased during this time (Spoor, 2006; Batey, 2009; Bohrer et al., 2017). Given that compaction is present and natural processes are not successful at alleviating it, the use of subsoilers such as deep ripping are necessary to reduce compaction.

### **Soil-Water Dynamics**

Soil-water dynamics as a result of compaction are changed in post-mined soil. According to Batey (2009), porosity is reduced in mineland soils, which results in reduced water infiltration. In western North Dakota, where the amount of water available for plant growth is a limiting factor, the compacted layer compounds this effect with reduced infiltration and water storage (Thompson et al., 1987). For example,

PAW generally decreased in the topsoil after four and 11 years of reclamation, however, subsoil and spoil had an increase in PAW (Carter et al., 1987). Increase of PAW in subsoil and spoil with time may be a result of water being able to percolate with less resistance as pore distribution develops. Macro-pore space was significantly reduced from 0.29 and 0.21 cm<sup>3</sup> cm<sup>-3</sup> to 0.18 and 0.08 cm<sup>3</sup> cm<sup>-3</sup> for topsoil and subsoil, respectively, four years after reclamation, and after 11 years there was a slight increase in macro-porosity attributed to root growth (Carter et al., 1987). Reduction in macro-pores reduces connectivity, seen with significantly decreased saturated hydraulic conductivity (K<sub>sat</sub>) from 55 and 32 cm hr<sup>-1</sup> to 28 and 5 cm h<sup>-1</sup> for topsoil and subsoil, respectively, after four years but no significant changes were measured after 11 years (Carter et al., 1987). A decrease in hydraulic conductivity is a result of pore network connectivity being reduced from soil reconstruction (Hetzler et al., 1992). Steady state infiltration significantly decreased by 78.3% for a mine site undergoing natural recovery, relative to the average steady state infiltration for undisturbed reference sites (arbors, bushes, arbor/brush mixture). Post-mine growth of bushes, arbor/bush mixture or a grassland mixture significantly increased steady state infiltration, relative to the site undergoing natural recovery (Zhang et al., 2016). No significant differences in infiltration for sites with vegetation was likely due to roots creating macropores, which allows for greater water movement. Chong (1992) generally found that a reclaimed mine soil had less macro-pores (<1%) than a non-mined reference site, which was attributed to low infiltration and K<sub>sat</sub> decreasing with increasing soil depth such that K<sub>sat</sub> could not be measured. Soil-water dynamics on post mining soils are marked by changes in pore distribution, connectivity and hydraulic properties, however, changes from pre-mine conditions have not been extensively examined.

### **Productivity**

Compaction is a limiting factor for returning land to pre-mine productivity (Batey, 2009; Dunker et al., 1995). Plant growth is limited by compacted layers because reconstructed soils have weak or non-existent soil structure (Dunker and Barnhisel, 2000). Vance et al. (1992) found the average PR from 23 to 112 cm was linearly correlated with corn (PR value;  $r = -0.97$ ) and soybean (PR value;  $r = -0.82$ ) yield reductions, however, above 2,068 kPa further yield reduction was not significant. Similarly, on a silt loam texture, soybean yield significantly increased from 1,475 to 1,963 kg ha<sup>-1</sup> when deep ripping was implemented to a depth of 51 cm (Caldwell et al., 1992). A conventional chisel plow to a working depth of

20 cm in a silt loam had a corn yield of 1,735 kg ha<sup>-1</sup>, which was significantly lower than other tillage treatments, which yielded between 3,927 to 9,125 kg ha<sup>-1</sup>. These differences were attributed to tillage working depths greater than 80 cm reducing compaction lower in the soil profile (Simmons et al., 1992). A review of root growth response to PR indicated that root growth was reduced by 50% between 1,000 to 4,100 kPa and 600 to 800 kPa for corn and sorghum, respectively (Grossman et al., 1992). With root growth potentially restricted, as seen with shallower rooting depths, this increases a plant's susceptibility to moisture stress (Caldwell et al., 1992). After loam textured topsoil replacement depths of 0, 15, and 30 cm corn and soybean yielded 3,013 to 4,136 kg ha<sup>-1</sup> and 699 to 1,432 kg ha<sup>-1</sup>, which were significantly lower than the county averages of 8,561 and 2,623 kg ha<sup>-1</sup>, respectively, which was attributed to soil compaction (Henning, 1992). The surface mining process results in compaction that reduces productivity, which can be improved with deep tillage, however, the longevity of tillage needs to be evaluated.

### **Conclusions**

As technology has improved, and environmental regulations increased, so have the mining and reclamation processes. Even though reclamation standards are in place, inevitably a disturbance is occurring where soil is being reconstructed. Compaction is the leading issue faced by surface coal mines during reclamation. Historically, the winter freeze-thaw cycles were assumed to help alleviate soil compaction over time but recent research has indicated that this natural process for alleviating soil compaction has not been efficient or effective on North Dakota coal mines (Bohrer et al., 2017). Deep ripping initially reduces compaction, but needs to be examined over time. The alleviation of compacted mine reclamation sites appears to need strategic management to increase root penetration, water infiltration, and plant productivity.

## CHAPTER 2. SUPERABSORBENT POLYMER CHARACTERISTICS, PROPERTIES AND APPLICATIONS

### Introduction

Superabsorbent polymers are materials that can retain large volumes of water (Buchholz 1998; Ekebafé et al., 2011) and maintain their shape (granular, fiber, sheets etc.) after swelling (Zohuriaan-Mehr et al., 2008). There are many SAP products available on the market and the variability in type and quality ranges from \$10 to \$40 (USD) per kg. These materials have the ability to increase plant available water (PAW), reduce bulk density (Bai, 2010), and mitigate drought stress (Huttermann et al., 2009) in addition to promoting conditions conducive to plant growth, and controlling the transport of plant nutrients and water (Ekebafé et al., 2011). Given the nature of SAPs, they can be used as a tool to strategically alter soil properties. Understanding how soil properties can be changed, and to what degree SAPs can change them, will improve overall soil function and may lead to increased agricultural and reclamation successes.

Superabsorbent materials have a wide range of applications, and thus, are used across many disciplines, including medicine, civil engineering, soil science, agricultural production, and hygienic products (Lejcus et al., 2018). For example, one of the initial uses for SAPs was as an absorbent in baby diapers (Zohuriaan-Mehr et al., 2008). Today, specialized SAPs have been developed to respond to specific molecules, allowing them to be used as biosensors and a method of drug delivery (Zohuriaan-Mehr et al., 2008). Additional water held by SAP can improve workability of cement mixture and durability by limiting shrinkage (He et al., 2019). Furthermore, SAPs have been used to dewater mine tailings (Roshani et al., 2017) and, as a remediation tool (Shi et al., 2016). The nature of SAPs and the engineering of new SAP technology makes them a tool that can be utilized across many fields.

Water absorbing materials are classified by their absorption mechanism. Physical absorbers, such as SAPs have four mechanisms in which water is taken up: (1) by changes of their crystal structure, (2) physical entrapment of water by capillary forces, (3) a combination of mechanisms one and two, (4) combination of mechanisms one and two coupled with dissolution and expansion of chains limited by cross linkages (Zohuriaan-Mehr et al., 2008). Superabsorbent polymers are a network of polymer chains where water is drawn into the polymer through the process of osmosis (Elliott, 2013). Chains contain ions

such as  $\text{COO}^-$  and  $\text{Na}^+$ , which create a diffusion gradient whereby water is then absorbed. Absorption and expansion is limited by cross-linkages (chains) (Figure 5). Low density cross-linked SAPs exhibit higher absorbent capacity and greater swelling whereas high density cross-linked SAPs have lower absorbent capacity and swelling resulting in greater 'gel' strength. Water is held within cross linkages by hydrogen bonding (Elliott, 2013).

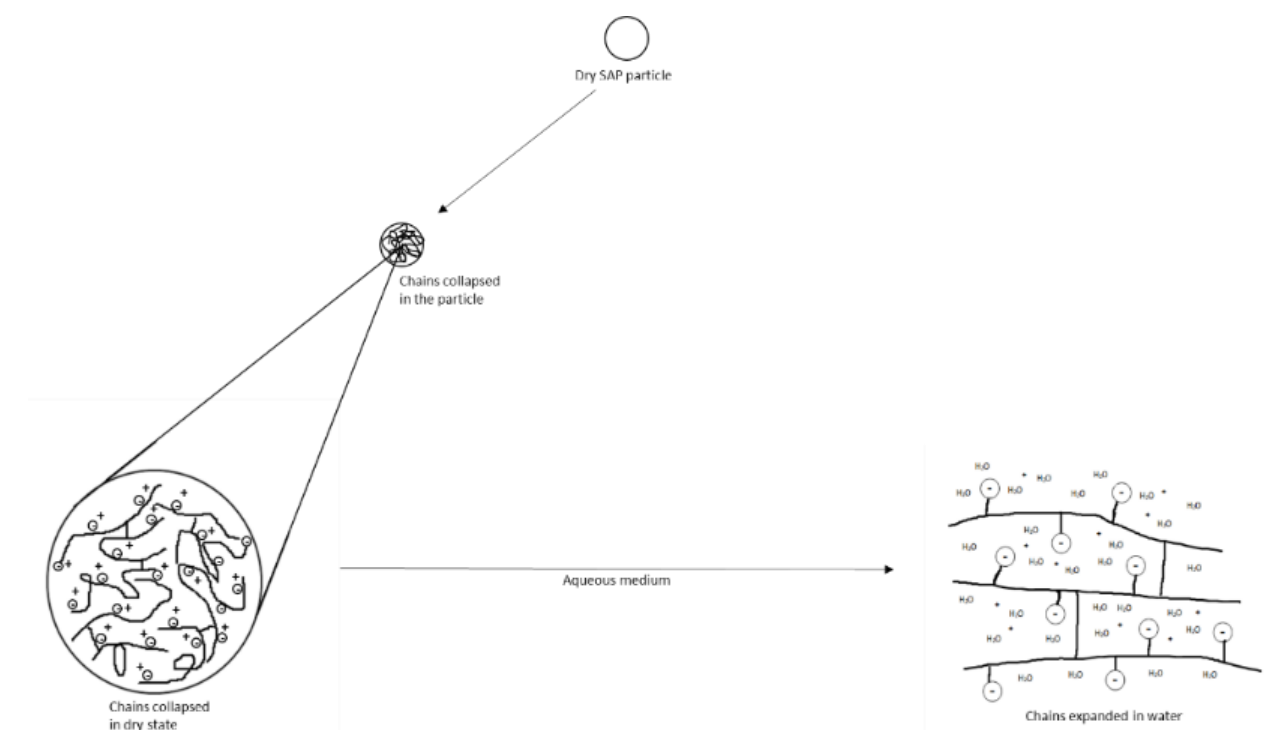


Figure 5. Superabsorbent polymer swelling. Adapted from Zohuriaan-Mehr et al. (2008).

Within soil and agricultural sciences, SAPs have been used since the 1950s to serve many functions (El-Hady et al., 1981). For example, the water retention capacity of SAPs has been shown to increase PAW, which can lower plant drought stress (Hutternman et al., 1999; Yun et al., 2017). In addition, SAPs can act as a reservoir holding on to nutrients from fertilizer preventing them from leaching from the root zone (Liang et al., 2007; Rudzinski et al., 2002) and have been used as a seed coating to increase seedling survival during water-limiting conditions (De Barros et al., 2017).

The primary use of SAPs has been their incorporation into soil to increase water retention and consequently improve plant growth (Eneji et al., 2013; Montesano et al., 2015; Dehkordi et al., 2017). The nature of SAPs to swell has been shown to reduce bulk density (Bai, 2010; Hou et al., 2017; Abrisham et al., 2018) and increase permeability (Han et al., 2010). The influence of SAP on soil evaporation has had



mixed results (Jabri et al., 2015; El-Asmar et al., 2017). The objectives of this review were to discuss how SAPs impact soil properties and potentially improve soil function within the context of environmental science and agriculture, as well as, highlighting areas for improved research.

### Search Methods

To examine the use and effect of SAP application within agriculture and the environment, a review of published literature using Web of Science was conducted on May 20<sup>th</sup>, 2019. For an article to be acceptable for this review it needed to include at least one of the topics from refined categories when term was basic searched (this searches titles, abstracts and keywords of manuscripts) (Table 2). In total, 30 sources were used for this review. Articles removed from this review encompassed polymer science, heavy metal mining, and de-watering of mine tailings.

Table 2. Terminology used in literature search.

Term	Categories	Topics
Superabsorbent Polymer	Environmental Science, Agronomy, Agriculture multi-disciplinary, soil science, Agricultural engineering, multidisciplinary science	Agriculture, yield, soil physical properties, bulk density, compaction, reclamation, water deficit, salinity, nutrients, water retention
Super-absorbent polymer	Environmental Science, Agronomy, Agriculture multi-disciplinary, soil science, Agricultural engineering, multidisciplinary science	Agriculture, yield, soil physical properties, bulk density, compaction, reclamation, water deficit, salinity, nutrients, water retention

Data obtained was organized by parameter and assessed to determine if SAP increased or decreased that soil metric relative to the control. For each paper the following information was collected: rate of application, method of mixing the soil and SAP, the depth at which the SAP was incorporated into the soil, the location of the experiment (Table 3), and the impact that SAP had on bulk density (BD), water content (WC), evaporation, saturated hydraulic conductivity (Ksat), infiltration, pH, electrical conductivity (EC), cation exchange capacity (CEC), nutrient retention, microbial activity, aggregate formation and yield or plant growth (Table 4). An increase in the metric was denoted with a "+", a decrease with a "-", and no change with a "0".

Table 3. List of literature articles included in this review with SAP information.

Reference	SAP rate of application	SAP method of mixing with soil	SAP incorporation depth (cm)
Abrisham et al., 2018	0, 1 and 3g dm <sup>-3</sup> of SAP: soil (0.07 and 0.2% BM) †	SAP hydrated then mixed with soil	
Bai et al., 2010	0, 0.05, 0.1, 0.2, 0.3% BM	mixing‡	0-8
Banedjschafie and Durner, 2015	0.2, 0.6, 1.0% BM	mixing	
Bhardwaji et al., 2007	0, 0.5, 2.5, 5.0% BM	mixing	
Cao et al., 2017	0, 0.25, 0.5, 0.75, 1, 2% BM	mixing	5
Chen et al., 2016	0, 0.1, 0.3, 0.6% BM	mixing	
Chen et al., 2018		NR§	
Dehkordi et al., 2017	0.05% BM	NR	
Dehkordi, 2018	(1.) 180kg ha <sup>-1</sup> ¶ (0.008% BM) (2.) 0.3 m <sup>3</sup> of SAP: water (0.008%)	(1.) Cast mixture of soil and SAP (2.) sprayed	
El-Asmar et al., 2017	0, 0.1, 0.2, 0.3, 0.4% BM	mixing and banding	0-15
Eneji et al., 2013	30 kg ha <sup>-1</sup> (0.001% BM)	applied to soil in lysimeter	0- 20
Grabinski et al., 2019	0, 10, 20, 30 kg ha <sup>-1</sup> (0.0004, 0.0008 0.001% BM)	NR	
Han et al., 2010	0.02, 0.05, 0.1, 0.2% BM	mixing and compacting	
Han et al., 2013	0.05% BM	mixing	
Hou et al., 2017	30, 60, 90 kg ha <sup>-1</sup> of 0.1% BM SAP:soil	mixing 10 cm	0-20
Islam et al., 2011	60 kg ha <sup>-1</sup> (0.003% BM)	in row with fertilizer	
Jabri et al., 2015	1.2 kg m <sup>-3</sup> (0.09% BM)	mixing	0-10
Li et al., 2014	(1.) 200kg ha <sup>-1</sup> (0.009% BM) (2.) 0.4m <sup>3</sup> SAP: water (0.009% BM)	(1.) Cast mixture of soil and SAP (2.) sprayed	
Montesano et al., 2015	0, 0.5, 1.0, 2.0% BM	mixing	
Prabha et al., 2014	0, 0.1, 0.2, 0.3, 0.4 ,0.5% BM	mixing	
Rostampour et al., 2013	0, 75, 150, 225 kg ha <sup>-1</sup> (0.003, 0.007, 0.015 BM)	by hand in rows	15-20
Salavati et al., 2018	80 kg ha <sup>-1</sup> (0.004% BM)	in furrow	10,15,20,25
Satriani et al., 2018	0, 5.0, 10.0 g plant <sup>-1</sup> (0.0004%, 0.0007% BM)	mixing by plant	
Volkmar and Chang, 1995	0.03, 0.12, 0.47, 1.87% BM	mixing	
Wilske et al., 2014	0.50% BM	mixing	
Yang et al., 2015	0.60% BM	mixing	20-30
Yu et al., 2011	0.50% BM	mixing	
Yu et al., 2012	0.05% BM	mixing	
Yuguo et al., 2013	0.02% BM	mixing	
Zhao et al., 2019	0, 0.1, 0.2, 0.5, 1.0% BM	mixing	10-20

† g dm<sup>-3</sup> percent by mass (BM), calculated using a standard bulk density of 1.33 g cm<sup>-3</sup>

‡ mixing was done using a variety of methods, such as by hand, or soil removal and mixture broadcast.

§ NR, mean not reported

¶ kg ha<sup>-1</sup> converted to % BM using an acre-furrow slice mass of 2,000,000 pounds.

Table 4. List of literature articles included in this review with changes in their respective parameters assessed.

Reference	BD	WC†	Evaporation	Ksat	Infiltration	pH	EC	CEC	Nutrient retention	Microbial activity	Aggregate formation	Yield/ plant growth	Type of study
Abrisham et al., 2018	-	‡			-			+					field (Iran)
Bai et al., 2010	-	+				+/-	+/-						lab
Banedjschafie and Durner 2015		+											lab & field (Iran)
Bhardwaji et al., 2007		+		+/-									lab
Cao et al., 2017					+				+				field (China)
Chen et al., 2016		+										-	lab
Chen et al., 2018		+							+				lab
Dehkordi et al., 2017		+										+	lab
Dehkordi, 2018		+								0			field (Iran)
El-Asmar et al., 2017		+	+/0									+	lab
Eneji et al., 2013									+			+	lab
Grabinski et al., 2019												+	field
Han et al., 2010	-			+									lab
Han et al., 2013				-									lab
Hou et al., 2017	-	+										+	field (China)
Islam et al., 2011		+							+			+	field (China)
Jabri et al., 2015		+	+										lab
Li et al., 2014		+/0								+	+		field
Montesano et al., 2015		+										+	lab
Prabha et al., 2014	-	+				+/-	+		+	+			lab
Rostampour et al., 2013		+										+	field (Iran)
Salavati et al., 2018												+	field (Iran)
Satriani et al., 2018												+	field (Italy)
Volkmar and Chang, 1995		+										0	lab
Wilske et al., 2014		-											lab
Yang et al., 2015					+/-								lab
Yu et al., 2011		+											lab
Yu et al., 2012			-										lab
Yuguo et al., 2013		+		-									lab
Zhao et al., 2019		+	-										lab

† WC includes relative water content, plant available water, water holding capacity, water use efficiency, gravimetric and volumetric water content.

‡ Symbols -, +, and 0 indicates a decrease, an increase, and no change, respectively, in each parameter.

## Superabsorbent Polymer Effects on Soil Properties

### Bulk Density

Of the five papers found for this review that examined BD, all of them reported a decrease. For example, in a greenhouse study on a sandy clay loam soil, 0.3% by mass (BM) SAP application significantly decreased bulk density from 1.27 to 1.17 g cm<sup>-3</sup> after undergoing four wetting-drying cycles (Bai, 2010). Bulk density of a sandy loam was shown to significantly decrease with 3 g dm<sup>-3</sup> (0.2% BM) SAP application from 1.56 to 1.45 g cm<sup>-3</sup> but the authors recommend the application rate of 1 g dm<sup>-3</sup> (0.07% BM) because it was statistically similar to the higher rate (Abrisham et al., 2018). Furthermore, for two consecutive years bulk density decreased from 1.72 and 1.71 g cm<sup>-3</sup> to 1.63 and 1.56 g cm<sup>-3</sup>, respectively, after 90 kg ha<sup>-1</sup> application of 0.1% SAP:soil mixture at the 0-30 cm depth of a sandy loam soil, but other treatments of 30 and 60 kg ha<sup>-1</sup> (0.001 and 0.003% BM), respectively, did not reduce BD to this degree (Hou et al., 2017). The same authors also found a significant increase in porosity after two years from 35.5 to 41.2% at the 30 cm depth with 90 kg ha<sup>-1</sup> application of 0.1% SAP: soil mixture. Similarly, bulk density was reduced from 1.05 to 1.00 g cm<sup>-3</sup> with 0.5% BM application under two watering intervals, which, correlated with porosity increasing from 67.7 to 77.9% (Prabha et al., 2014). The above studies indicate that SAP application can reduce BD, however, application rates are highly variable and the majority of these studies took place in laboratory experiments, making the feasibility of field scale compaction reduction a potential research endeavor.

### Soil-Water Dynamics

Of the 30 papers found for this review, 26 of them studied the influence of SAP on soil-water dynamics. Given the number of studies, SAP application method and rate varied, however, 12 studies mixed SAP on a mass basis with soil at rates of 0.05-2%. Even though seven studies were conducted in the field and 19 in the laboratory, soil-water dynamics had a positive response (i.e. improved water retention) to SAP application for the majority of studies (n=26).

The water retention and absorbency capacity of SAPs can increase PAW, as reported by three studies. For example, with no SAP application, PAW was 3.62% for a sandy loam soil, and with 1 g dm<sup>-3</sup> (0.07% BM) and 3 g dm<sup>-3</sup> (0.2% BM) PAW increased to 6.1% and 8.8%, respectively (Abrisham et al., 2018). When applied to dune sand at rates of 0.3, 0.6 and 1% SAP: soil, PAW significantly increased

from the control ( $0.005 \text{ g g}^{-1}$ ) by 0.56, 0.20,  $0.27 \text{ g g}^{-1}$ , respectively (Banedjschafie and Durner, 2015). With  $200 \text{ kg ha}^{-1}$  (0.009% BM) wet and dry SAP application to a loam soil, PAW significantly increased for the first two stages of wheat growth from 12% to 14.4-15.1% and 10% to 12.6-14.8%, but decreased for the filling stage, likely due to the plants taking up water in early growth stages (Li et al., 2014).

The response of soil water holding capacity (WHC), soil water content (WC), and water use efficiency (WUE) to SAP application was examined by 10 papers in this review. Wilske et al. (2014), reported that 0.05% BM application, degraded by 0.45 to 0.76% after six months, which though not significant, reduced soil WHC of a loamy-sandy texture range of soils. Of the ten papers noted above, each of the four studies that took place in the field applied SAP differently, with depths ranging from surface applied down to 20 cm below the soil surface. Dry application rates ranged from  $75 \text{ kg ha}^{-1}$  (0.003% BM) to  $225 \text{ kg ha}^{-1}$  (0.015% BM) and wet application rates were 5.4 and  $7.2 \text{ g}$  in  $0.3 \text{ m}^3$  and  $0.4 \text{ m}^3$  water, respectively. The greatest application rate in the Rostampour et al. (2013) sandy loam soil study,  $225 \text{ kg ha}^{-1}$  (0.015% BM) increased leaves relative water content (WC related to WC at full turgor) from 61 to 78% while the lowest application of SAP,  $75 \text{ kg ha}^{-1}$  (0.003% BM), only increased 6%. The same authors found a significant increase in WUE from 2.1 to 2.6-2.9 kg dry mass produced per  $\text{m}^3$  of water for 75, 150, and  $225 \text{ kg ha}^{-1}$  (0.003, 0.007, and 0.015% BM) of SAP application (Rostampour et al., 2013). With  $200 \text{ kg ha}^{-1}$  of wet and dry SAP applied to a loam soil surface, WC increased significantly for jointing, booting and filling stages of wheat growth from 13% to 14.8-15.2%, 10% to 12-13.9% and 6% to 9.8-10.1% (Li et al., 2014). Similarly, at  $180 \text{ kg ha}^{-1}$  (0.008% BM) SAP wet and dry application there was a significant increase in WHC from 11.6% to 15.8-16.2%, at the stem elongation growth stage, similar increases were seen in heading and doughing stage (Dehkordi et al., 2018). To a sandy soil undergoing wetting and drying cycles SAP application of 0.05, 0.1, 0.2 and 0.3% increased WC by 6.2 to 32.8% relative to the control whose water content range was 14.3% to 85.6% (Bai et al., 2010). Field study results showed some statistically improved WC and WHC for SAP amended soils, but not below a rate of  $75 \text{ kg ha}^{-1}$  (0.003% BM).

Of the six laboratory studies examining WHC, WC, and WUE, SAP application rates ranged from 0.02-2% by mass or  $30 \text{ kg ha}^{-1}$  (0.001% BM), all of which determined that SAP had an impact on soil water dynamics when using the mixing method. For example, saturated volumetric water content

increased up to  $0.19 \text{ cm}^3 \text{ cm}^{-3}$  with 0.02% BM application to a sandy loam soil, with no difference between two different SAPs. However, the polyacrylamide had a longer effective retention time (Yuguo et al., 2013). After 30 days of drying, water retention of a constructed soil consisting of sandy clay loam, sand, and cow dung in a ratio of 1:1:1 with SAP application of 0.1, 0.2, 0.3, 0.4, and 0.5% by mass, increased significantly from 8% to 12, 15, 17, 18, and 19%, respectively (Prabha et al., 2014). Similarly, after only seven days, SAP applied at rates of 0.25, 0.5, 0.75, 1.0, and 2.0% BM to the 15 cm depth of silt loam soil, significantly increased WC from 8.5% to 14.1-16.3% (Cao et al., 2017). When a sandy soil was amended with 0.5% BM SAP, WC at field capacity increased 60% relative to the control, but significant increase in WC at PWP was not seen until 2% BM application rate (Montesano et al., 2015). The results from the above studies indicate that SAP can increase WC and WHC, however, a specific rate cannot be recommended because variation in soil texture impacts the quantity of SAP that needs to be applied.

As stated above, SAPs have the ability to increase water in the soil and, consequently, have the potential to change evaporation dynamics. Of the papers reviewed, four studied the response of evaporation to SAP application. With SAP application by mixing or banding, evapotranspiration (ETc) for corn in a clay soil did not change, however, in a sandy clay loam, the ETc increased from  $5.08 \text{ kg pot}^{-1}$  with 0 SAP to 5.22, 5.31, 5.46, and  $5.58 \text{ kg pot}^{-1}$  for application rates of 0.1, 0.2, 0.3, and 0.4% BM, respectively (El-Asmar et al., 2017). However, banding of SAP resulted in higher ETc for the 0.2, 0.3 and 0.4% application rates, which was attributed to banding acting more like a reservoir for water that plants could better utilize (El-Asmar et al., 2017). In a loamy sand, sandy loam, sandy clay loam, and clay loam soils amended with 0.5% BM, after seven hours of drying at  $60 \text{ }^\circ\text{C}$ , retained  $0.12\text{-}0.35 \text{ g g}^{-1}$  of water compared to soils with no SAP application, which only retained  $0.001\text{-}0.08 \text{ g g}^{-1}$  of water. The water losses were faster in larger (3 to 4 mm diameter) SAP particles compared to 0.2-1.0 mm diameter particles (Yu et al., 2012). For a fine textured saline soil, application of  $1.2 \text{ kg m}^{-3}$  (0.09% BM) of SAP significantly decreased cumulative evaporative water loss, compared to the control of no SAP, from 69 mm to 52 mm after 35 d of drying at  $26.6 \text{ }^\circ\text{C}$ , (Jabri et al., 2015). A constructed soil that consisted of 40 cm of coarse soil overlay with 10 cm of SAP:soil mixture with 10 cm of sand on surface was used to examine water loss. Application of 0.2, 0.5 and 1% BM significantly decreased evaporative water loss at

the 2 and 18 cm depths after 10, 20 and 30 d of evaporation at 23 °C and the authors recommended that the 0.2% rate would allow for normal plant growth (Zhao et al., 2019).

The water retention abilities of SAP may also work well for irrigated soils. For example, Yang et al. (2015) reports on a 110 day drip irrigation study conducted on a constructed soil [sand (0-20 cm), loam (20-40 cm), silt (40-70 cm)] amended with 0.6% BM in the 20-30 cm depth. They observed the SAP amended layer had 12.5% volumetric WC, whereas the layers above and below only had 3.5% and 4.5% volumetric WC, which indicated that SAP influenced both downward water movement and retention.

Superabsorbent polymer application can potentially change pore size and pore network connectivity, which govern  $K_{sat}$  and infiltration. Field infiltration studies used application rates ranging from 0.07 to 0.6% BM and four  $K_{sat}$  laboratory studies used application rates ranging from 0.02-5.0% BM. Compared to the previous soil-water dynamic parameters, infiltration and  $K_{sat}$  had the greatest variability in response to SAP application. Using a sandy loam soil with 3 g dm<sup>-3</sup> (0.2% BM) SAP application, infiltration decreased from 7 cm h<sup>-1</sup> to 4.4 cm h<sup>-1</sup> (Abrisham et al., 2018). Similarly, for a silt loam soil, runoff was reduced by 24.6, 41.5, 46.5, 50.7% relative to the control (284 L of runoff) for SAP applications of 0.25, 0.5, 0.75, 1.0, and 2.0% BM, respectively, applied at the 5 cm depth (Cao et al., 2017). Yang et al. (2015), constructed a soil that consisted of the following layers from top to bottom: 0-20 cm sand, 20-40 cm loam and 40-70 cm silt. With SAP applied at 0.6% BM incorporated at the 20-30 cm depth, the 85 cm depth had a greater horizontal infiltration rate than vertical infiltration rate because of the SAP influence on this layer (Yang et al., 2015). With 0.02-0.2% BM SAP application to loam and sandy loam soils, BD significantly decreased from 1.55 g cm<sup>-3</sup> to less than 1.42 g cm<sup>-3</sup> for all treatments (Han et al., 2010). This would have increased soil porosity which may have induced large variations in  $K_{sat}$  (Han et al., 2010). For example, when saturated at a 2 mm h<sup>-1</sup> wetting rate, the  $K_{sat}$  of a sandy loam with no SAP application was 71 mm h<sup>-1</sup>, which decreased to 25.8-52.4, 37.3-54.0, and 34.2-65.9 mm h<sup>-1</sup> for three polymers (Alcosorb, Stocksorb 500 medium and Stocksorb 500 Micro, respectively) applied at 0.5, 2.5, 5.0% BM (Bhardwaji et al., 2007). At an increased saturation rate of 50 mm h<sup>-1</sup>,  $K_{sat}$  was greater than the 2 mm h<sup>-1</sup> saturation rate for 0.5, 2.5 and 5.0% BM (Bhardwaji et al., 2007). Decrease in  $K_{sat}$  is attributed to SAPs filling pore space and restraining water movement but its increase with time is attributed to shrinking and swelling of polymers undergoing wetting-drying cycles (Han et al., 2013). While studies

have examined SAP: soil mixtures influence on Ksat, the effects of freezing and thawing have not been investigated.

Preventing drought stress of plants is a driver for determining an irrigation schedule. Superabsorbent polymers' ability to retain water could potentially change irrigation intervals. Abrisham, (2018) examined complete irrigation, defined as soil fully rehydrated six times at 30 day intervals, and limited irrigation as soil partially rehydrated three times at 60 day intervals. When water deficit conditions were created by limited irrigation, an SAP application of  $3 \text{ g dm}^{-3}$  (0.2% BM) had similar seedling establishment percentage compared to complete irrigation (Abrisham, 2018). Plant characteristics such as shoot height, shoot fresh weight biomass and aboveground biomass under limited irrigation were all greater relative to the control (no SAP) when SAPs were applied (Abrisham, 2018). When no SAP was applied, there was a significant difference between irrigation intervals demonstrating SAP's ability to reduce potential drought stress. After 30 days, a constructed soil (1:1:1 sandy clay loam:sand:cow dung) with 0.3, 0.4 and 0.5% SAP BM had a soil water content of 17.0, 18.2 and 19.0%, respectively, compared to the control which only retained 8.0% (Prabha et al., 2014). Higher water retention would potentially allow for drought or water deficit stress to be mitigated.

In summary, while SAPs can influence soil-water dynamics, determining the right rate for the intended function will be influenced by soil texture, method of application, and water availability. The ability of SAP to increase PAW is beneficial, and the results of this review indicate that greater benefits may be seen in coarser-textured soils. The studies above used many application rates across various soil textures, such that, selecting an ideal application rate is difficult. Since there are a large number of SAPs on the market understanding performance differences will be integral for determining site specific feasibility.

### **Chemical Properties**

Superabsorbent polymer application may influence soil chemical properties, such as pH, EC, CEC, and nutrient retention. Of the papers reviewed (Table 4), seven papers examined changes to soil chemical properties with SAP application with varying response, except nutrient retention, which for all five papers reviewed had a positive response. For the three field studies, the application methods and rates included hydrated SAP mixed with soil in planting holes at  $1\text{-}3 \text{ g dm}^{-3}$  (0.07-0.2% BM), mixing with



the 5 cm depth at 0.25-2.0% BM and, in row application with no planting depth reported at 60 kg ha<sup>-1</sup> (0.003% BM). Of the five laboratory and greenhouse studies, all used a mixing method and four applied SAP BM ranging from 0.05-2%, the remaining study applied SAP at 60 kg ha<sup>-1</sup> (0.003% BM).

Understanding how SAPs may influence CEC, pH, and EC is important because these properties can affect plant growth.

Soil pH may be affected by SAP application. For example, in a constructed sandy clay loam soil with 0.5% powdered SAP application undergoing two watering intervals over a two week study period, soil pH between watering intervals was not different but pH was reduced from 6.04 to 5.7 and 5.8 this decline was attributed to the carboxylate ions present within the SAP (Prabha et al., 2014). Given that the SAP was in powder form the higher surface area may have compounded the effect on soil pH (Prabha et al., 2014). The type of SAP, and time that it has been in soil, may increase or decrease soil pH. In a field study, sodium polyacrylate applied at a rate of 0.3% BM was the only treatment that did not decrease soil pH but potassium polyacrylate applied at 0.05% BM initially increased soil pH but after 12 days pH decreased relative to the control (Bai et al., 2010). The differences in soil pH are attributed to the different chemical composition of SAPs applied, as well as wetting and drying. Superabsorbent polymers can influence soil pH and changes will be a function of the type of SAP, length of time after application, and the buffering capacity of the soil.

Application of SAP can change EC, as reported by two papers in this review, but the responses were varied. For example, in a constructed sandy clay loam soil the control soil had an EC of 2.95 dS m<sup>-1</sup>, whereas after 13 weeks an application rate of 0.5% SAP treated soil increased EC to 5.3 dS m<sup>-1</sup> (Prabha et al., 2014). The increase in EC was associated with the acids and ions present in SAP. In a soil with changing water states, SAP application increased EC initially, but after 12 and 21 days after application EC significantly decreased 51.7-65.5%, and 42.5-51.3%, respectively, relative to the control (0.3 dS m<sup>-1</sup>) for two polymer types (Bai et al., 2010). The above studies show that SAP effects EC, however, the responses were variable which may be a result of SAP composition.

Just as SAPs can alter soil pH and EC, they can change cation exchange capacity (CEC). For example, SAP application of 3 g dm<sup>-3</sup> (0.2% BM) to a sandy loam soil increased CEC from 6.2 to 8.2 cmol<sub>c</sub> kg<sup>-1</sup> but no change was observed at an application rate of 1 g dm<sup>-3</sup> (0.07% BM) (Abrisham et al.,

2018). Similar to changes in soil pH and EC, changes in CEC are associated with the composition of SAPs. Of the papers reviewed only one study assessed CEC response to SAP application.

Understanding the ability of SAPs to change CEC is important as it may influence soil fertility and plant growth.

Another consideration is the ability of SAPs to act as nutrient reservoirs. Above ground biomass of drought stressed forage oat decreased under low and medium fertilizer levels but when SAP was applied at 60 kg ha<sup>-1</sup> (0.003% BM) biomass significantly increased from 1.1 and 1.7 Mg ha<sup>-1</sup> by 69.4% and 30.7%, respectively (Islam et al., 2011). Reduction in growth indicated that less fertilizer could be used when accompanied by SAP, however, this study took place in an arid region receiving only 40 cm of annual precipitation. Superabsorbent polymer application at 0.5% BM increased N, K, and Ca nutrient retention from 0.18, 221.0, and 112.4 mg kg<sup>-1</sup> to 10, 1.6 and 27.3 mg kg<sup>-1</sup>, respectively, relative to the control (Prabha et al., 2014). One of the papers was a review paper that identified numerous studies that improved nutrient retention when SAP was part of fertilizer application (Chen et al., 2018). When SAP was applied at 30 kg ha<sup>-1</sup> (0.001% BM) to a loamy sand, nitrate leaching decreased from 135.5 mg L<sup>-1</sup> to 61.5 mg L<sup>-1</sup> (Eneji et al., 2013). Under controlled rainfall simulation an application rate of 0.75% BM decreased N, P, and K runoff losses by 14.9, 14.2, 13.1%, respectively, relative to the control (Cao et al., 2017). While the overall response of nutrient retention to SAP application was positive, the application rate and water entering the system was a factor.

In summary, while SAPs can be beneficial to nutrient retention, their impact on soil pH and EC is highly variable. The above studies had different application rates, as well as, types of SAP. The above results highlight that changes in soil chemical properties will be dependent on application rate, water dynamics, and SAP composition.

### **Biological Properties**

While the primary uses of SAPs are to conserve water and promote plant growth, three studies from this review examined microbial activity and one study found an increase in aggregate formation. Of these studies, two of them were performed in the field with dry application rates of 180 kg ha<sup>-1</sup> (0.008% BM) and 200 kg ha<sup>-1</sup> (0.009% BM) or wet application of 5.4 g per 0.3 m<sup>3</sup> (0.008% BM) or 7.2 g per 0.4 m<sup>3</sup> (0.009% BM) SAP:water mixture applied to soil surface and the only laboratory study used 0.1, 0.2, 0.3,

0.4 and 0.5% BM. Even though different SAPs, application rates, and methods were deployed, there was no negative responses to any soil biological properties.

Application of SAPs can increase soil microbial abundance and activity. For example, when 5 g of soil was sampled in the booting and filling growth stages of wheat, bacterial abundance significantly increased with 200 kg ha<sup>-1</sup> (0.009% BM) dry application and wet application of 7.2 g of SAP with 0.4 m<sup>3</sup> of water to a loam soil (Li et al., 2014). In the jointing stage, bacterial abundance increased for one polymer but decreased for another under bulk dry application (Li et al., 2014). Similarly, with 0.5% BM to a constructed soil, bacteria and fungi counts increased 16% and 18%, respectively, when compared to soil with no SAP (Prabha et al., 2014).

While the abundance of soil microbes changes with SAP application, the microbial activity characterized by microbial biomass carbon (MBC) and soil microbial respiration (SMR) should also be examined. For example, when SAP was applied at 180 kg ha<sup>-1</sup> (0.008% BM) dry and wet applied at 5.2 g SAP with 0.3 m<sup>3</sup> of water there was no significant change in MBC, but there was a decrease in SMR when sampled during the plants' doughing stage (Dehkordi et al., 2018). Increased MBC and SMR in other growth stages were attributed to the microbes being able to utilize SAP as a carbon source (Li et al., 2014). Superabsorbent polymer application can increase microbial activity and abundance but the effect is largely dependent on growth stage of plant.

Superabsorbent polymers alter the soil environment by physical presence and increased water content, which potentially impacts biological processes such as aggregate formation. Of the 30 studies reviewed, only one measured SAP impacts on aggregate formation. Both 200 kg ha<sup>-1</sup> dry (0.009% BM) application and wet application of 7.2 g SAP with 0.4 m<sup>3</sup> of water sprayed for two SAPs increased macro aggregates (>0.25 mm) from 54.0 and 54.4% by 14.1 to 16.4% and 15.9 to 16.6% when sampled at the plants' booting and filling stage, respectively (Li et al., 2014). This increase in macro aggregates is attributed to SAPs absorbed to soil particles preventing dispersion. Given there was no change in aggregate formation in the first growth stage, SAPs may not have had adequate time to bind to soil (Li et al., 2014). Follow up studies should be conducted to learn more about how SAPs affect aggregate formation since soil texture, application rate, and time after application are contributing factors.

While SAPs can improve microbial abundance and activity, and aggregate formation, selecting the appropriate rate and application method needs further research. The above studies had variable application rates, methods, and SAPs, which makes determining a standard rate difficult. Only three studies in this review examined SAP influence on soil biological properties, making determinations even more complex, due to small sample size.

### **Yield and Plant Growth Characteristics**

One of the primary reasons for applying SAPs is to improve plant yields or their production. Of the eight studies that focused on this, only one study reported a negative response. This negative response was attributed to higher concentration of acrylic acid and  $\text{Na}^+$  within the SAP, which inhibited maize growth and altered root morphology with increasing application rate (Chen et al., 2016). Of plant response studies, only 50% were performed in the field. Application methods included hydrating SAP and subsequent mixing with soil, dry mixing with soil, hand application in row, and a surface-sprayed SAP: water mixture. Field application rates varied from  $30 \text{ kg ha}^{-1}$  (0.001% BM) to  $225 \text{ kg ha}^{-1}$  (0.015% BM) and laboratory/greenhouse application rates from 0.05% to 2% BM. Even with variation in application method and rate there was an overall positive response of plants to SAP application.

Superabsorbent polymer application can improve plant growth. For example, cucumber total fresh biomass and fruit fresh biomass increased by 840 g and 494 g per plant, respectively, with  $2 \text{ g L}^{-1}$  of SAP in a sandy soil (Montesano et al., 2015). Another study with various potato cultivars found increased tuber yield when planted at 10, 15, 20 and 25 cm depth with SAP applied in furrow at  $80 \text{ kg ha}^{-1}$  (0.004% BM) to a clay loam soil and the highest yield was found at the 25 cm sowing depth (Salavati et al., 2018). A similar response was found in a sandy loam soil for 0.1% BM SAP:soil mixture when applied at 60 and 90  $\text{kg ha}^{-1}$ , which yielded potatoes, 38.2 and 50.5% higher than the control ( $24.2 \text{ Mg ha}^{-1}$ ) when SAP was applied at 20 cm (Hou et al., 2017). While SAP application can improve plant growth, the depth in which it is applied may improve its effectiveness.

Under stress conditions SAPs have a greater influence on plant response (Rostampour et al., 2013; Eneji et al., 2018). For example, sorghum dry matter only increased when there was a water deficit in sandy loam soil with SAP application rate of 75, 150 or  $225 \text{ kg ha}^{-1}$  (0.003, 0.007, 0.015% BM) (Rostampour et al., 2013). At the application rate of  $30 \text{ kg ha}^{-1}$  (0.001% BM) to a sandy loam soil, corn

biomass increased 99% under deficit irrigation and 39% under moderate irrigation, and only 11% under full irrigation and, overall, plants under water deficit showed reduced stress signals with SAP application (Eneji et al., 2018). Over a three year study, SAP application at 30 kg ha<sup>-1</sup> (0.001% BM) increased wheat grain yield relative to the control and further yield increase was found in years of water deficit (Grabinski et al., 2019). Similarly, yield of dry bean grown in a silty clay loam with 5 g plant<sup>-1</sup> (0.0004% BM) SAP application at 70% crop evapotranspiration (ET<sub>c</sub>) was similar to no polymer application at 100% ET<sub>c</sub> (Satriani et al., 2018). With salt (NaCl), drought, and combined (NaCl + drought) stress introduced, eucalyptus dry weight increased from 52.56 g to 45.56, 44.14, and 47.68 g, respectively, and the increase in dry weight was attributed to SAPs mitigation of salt and drought conditions (Dehkordi et al., 2017).

In low fertility conditions, SAPs have shown to improve plant metrics. For example, using an SAP application rate of 60 kg ha<sup>-1</sup> (0.003% BM) in standard, medium, and low fertility conditions, relative to no SAP applied, forage oat tillers increased by 5.5, 11.6, 18.6%, above ground biomass increased by 30.4, 30.7, 69.46%, and grain yield increased by 30.7, 29.7 and 70.8%, respectively. Plant growth can be improved with SAP application, specifically under stress or deficit conditions, which may make SAPs attractive for these conditions.

Application of SAP at recommended rate does not always perform the desired function. For example, two SAPs applied at the recommended rate were not effective at improving yields of canola and barley but four times this rate improved species yield (Volkmar and Chang, 2011). In another field-scale study 1 g dm<sup>-3</sup> (0.07% BM) and 3 g dm<sup>-3</sup> (0.2% BM) were found to improve seedling establishment, however, these application rates were not significantly different (Abrisham et al., 2018). Application of SAPs has proven to improve plant growth, however, the technical aspects, such as, application rate and method need to be refined.

In summary, while SAPs can be beneficial to plant growth, selecting the proper rate for the intended function is important, and the recommended rate of application rate to soil as stated by Volkmar and Chang (1995), 0.03% BM, has not been fully vetted. The above studies used many rates of application across many soil textures, which confounds the ability to predict a uniform rate.

## **SAP Performance Considerations**

Superabsorbent polymer performance is going to be dependent on field conditions, application depth, and rate of application. Since SAPs will be incorporated with soil it is important to consider their absorbency under load. With increasing BD and application depth, SAPs absorption time increased and overall absorbency decreased (Lejcus et al., 2018) and in addition, confinement by the soil matrix restricted SAP function (Yu et al., 2012). Salt type and concentration within the soil system can further diminish SAP absorbency (Jabri, 2015). These changes in capacity should be taken into consideration when determining SAP application method, rate of application, and depth of placement.

## **Environmental Impacts**

Some common concerns about the use and application of SAPs include safety, toxicity, and environmental fate. Superabsorbent polymers are “irreversible,” meaning that they cannot be returned to starting materials or converted to toxic metabolites (Zohuriaan-Mehr et al, 2008). The majority of the material safety data sheets for SAPs classify these compounds as being pH neutral, inert, and overall “Safe and Non-toxic,” and are currently not regulated as a hazardous material. A few examples include: 1) Zap Zorb Crosslinked Sodium polyacrylate (9003-04-7, Zappa Tec LLC, McLeansville NC) is only an irritant if exposed over the recommended amount (8 hour exposure limit of 0.05 mg/m<sup>3</sup>), and 2) composted polyacrylate is labeled as nontoxic to aquatic or terrestrial organisms at predicted exposure levels from current product specific application rates.

When SAPs are degraded in the soil, the products are carbon dioxide, water, and ammonia (De Barros et al., 2017). While the products of degradation are not toxic, there is limited understanding in the breakdown mechanisms of these compounds and the cumulative effects, if any, that may occur within the soil ecosystem. The general half-life of SAPs in soil is five to seven years (Ekebafe, 2011), and their longevity will be influenced by soil pH (Sadd et al., 2009). The stable carbon backbone, low solubility, and high mass due to crosslinking make SAPs resistant to breakdown by bacteria (Stahl et al., 2000). Biodegradation can occur when mineralization and solubilization, which can be aided by white rot fungi and microbes, are maximized (Stahl et al., 2000). If the application of SAPs in soil for the above mentioned uses increases, its degradation and longevity in soil may need further exploration.

Given that SAPs do not degrade readily and persist for several years it is essential to understand their mobility. If SAPs become mobile their effectiveness may diminish and could potentially enter the ground water system. Even though these materials are non-toxic, some state regulations do not allow these materials to enter the groundwater. When 30 pore volumes of water was passed through sand test columns, 99.7% of polyacrylate absorbent and 92% of linear poly acrylic acid was retained within the column (Sack et al., 1998). Mobility of SAPs may be dictated by soil texture and organic matter. Soils with higher organic matter and clay contents will likely limit SAP mobility. Long term studies of SAP mobility have not been conducted.

### **Future Research**

Superabsorbent polymer performance may change over time as a result of field conditions. These changes have been studied within the laboratory and on relatively short time scales. When thinking about applying these materials at the field scale, the longevity of performance over the course of multiple years needs to be evaluated. The degradation of the SAPs under various field conditions should be studied. Depth of positioning, rate of application, and method of application should all be evaluated at the field scale to determine benefit-cost ratio for SAP application. Overall, SAPs need to be examined *in situ* over longer time domains, as well as, how soil mechanical properties are changed.

### **Conclusions**

Superabsorbent polymers have primarily been used to improve and prolong soil water holding capacity and increase time between irrigation events. There has been positive results for SAPs' ability to reduce compaction. However, the majority of SAP studies were performed in the laboratory or on small field scales and results may not translate to larger scale projects or may not be economically feasible at such a scale. Large scale and long term projects are recommended to evaluate the function, performance, and overall benefits to crop yields and plant productivities.

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## CHAPTER 3. SUPERABSORBENT POLYMER EFFECTS ON SOIL PHYSICAL PROPERTIES FOR VARIOUS SOIL TEXTURES AND APPLICATION RATES

### Abstract

Superabsorbent polymers (SAPs) are materials that can absorb significantly more water or aqueous solution than their mass. The nature and properties of SAPs make them a widely utilized material across many disciplines. A series of laboratory studies were conducted to determine the effects of four rates of SAP application (0, 0.04, 0.08, and 0.2% by mass) across five soil textures on plant available water (PAW), Liquid limit (LL), evaporation, and changes in compression and saturated hydraulic conductivity (Ksat). The average PAW increase across all soil series was 0.04 and 0.13 cm cm<sup>-3</sup> for 0.08 and 0.2% application rates, respectively. The water content where the LL occurred generally increased, with the 0.2% rate having a significant increase for the Fargo, Williams, and Delamere soil series. The 0.2% rate increased the average stage one evaporation duration for all soil series from 2.3 to 7.7 d and decreased water loss, whereas for stage two evaporation water loss increased. After undergoing six freeze-thaw cycles, compression generally decreased, and Ksat decreased with increasing SAP application. Superabsorbent polymers can change soil physical properties at different rates and soil textures; translation to in field setting should be further investigated.

### Introduction

The ability of superabsorbent polymers (SAPs) to retain large volumes of water relative to their mass gives them the potential to alter soil properties (Hutterman et al., 1999; Bai, 2010; Yu et al., 2011; Han et al., 2013). Super-absorbent polymers are natural or synthetic materials that absorb more than 10 times their mass of aqueous solution (Buchholz 1998; Zohuriaan-Mehr et al, 2008). These materials can have a similar appearance to granulated sugar with a particle shape (granular, fiber, sheets etc.) that is preserved after water absorption and swelling (Zohuriaan-Mehr et al, 2008). Cost is dependent on the type and quantity of SAP purchased but commonly ranges from \$10 to \$40 (USD) per kg.

The influence of SAPs on soil physical properties such as bulk density, plant available water (PAW), Atterberg limits, evaporation, and saturated hydraulic conductivity (Hutterman et al., 1999; Bhardwaj et al., 2007; Bai, 2010; Yu and Shi et al., 2012; Zhao et al., 2019) will be dependent on the rate of application, soil wetting-drying, soil freezing-thawing, and soil texture. Changes in soil-water status will

likely, to some degree, also alter soil biological and chemical properties (Prabha et al., 2014). Since the outcome of successful soil reclamation is to achieve pre-disturbance soil function, SAPs may be used as a tool to potentially decrease the time to obtain pre-disturbance soil function.

Compaction of soils is a major obstacle in coal mine-land soil reclamation (Spoor et al., 2006; Batey, 2009) and since SAPs will expand when water is absorbed, the ability of these polymers to break up compaction (Bai, 2010; Han, 2010) may make them ideal in situations where compaction is prevalent (Ekebafé et al., 2011). Their ability to function in this role will be a function of rate of application rate, wetting and drying, and freezing and thawing cycles. For example, in a greenhouse study on a sandy clay loam textured soil, SAP application rates of 0.05, 0.1, 0.2 and 0.3% increased gravimetric water content by 6.2 to 32.8% and bulk density decreased by 5.5 to 9.4% relative to the control after undergoing wetting-drying cycles (Bai, 2010). Although decreases in soil bulk density with SAP application have been reported (Hou et al., 2017; Abrisham et al., 2018), no studies were identified when freezing and thawing was a treatment.

The water retention capacity of SAPs give them the ability to mitigate drought stress (Huttermann et al., 1999), and increase time between irrigation intervals (Prabha et al., 2014; Abrisham, 2018), which allows for plant stress to be reduced and plant growing conditions to improve (Abrisham, 2018). However, if SAP application rate is too high there can be detrimental effects on plant survival (Busscher et al., 2009; Han et al., 2010) and the function of SAPs to increase PAW is limited by ions present (Jabri et al., 2015) and expansion restricted by the soil matrix (Yu et al., 2011). How SAPs function in a given system will dictate potential desirable effects.

Since evaporation and saturated hydraulic conductivity result from, connectivity of pores, and sizes of pores, the application of SAPs may influence these metrics. For example, SAP mixed with a loamy sand, sandy loam, sandy clay loam or clay loam soils prolonged the first stage of evaporation, but evaporation rate was constant (Suleiman and Ritchie, 2003) relative to the control (Yu and Shi et al., 2012). When a soil was constructed with 10 cm sand, 10 cm SAP:soil mixture, 40 cm soil and packed to a bulk density of  $1.30 \text{ g cm}^{-3}$ , evaporation in all layers decreased relative to the control, which had no SAP (Yang et al., 2015; Zhao et al., 2019). Although a reduction in hydraulic conductivity occurred with SAP

application in a sand-textured soil (Bhardwaj et al., 2007), saturated hydraulic conductivity increased as time increased, which was attributed to wetting and drying cycles (Han et al., 2013).

Given the importance of decreasing the time to successful reclamation after major disturbances, such as soil replacement after extraction of coal, further investigation of using SAPs for this purpose is warranted. Understanding how soil physical properties can be changed with SAP will improve overall soil function and lead to increased agricultural and reclamation success. Using the information obtained from various lab experiments, the objectives of this research were to i) quantify SAP influence on soil water retention, LLs, evaporation, post freeze-thaw hydraulic conductivity, and compression, and ii) infer the potential for SAP to amplify conditions for freeze-thaw cycles to alleviate soil compaction. The findings of these experiments are necessary in finding alternative approaches to reduce the time to successful soil reclamation.

## **Materials and Methods**

### **Soil Properties**

Five soils (0-15 cm depth) from North Dakota were used for this study. The soils are classified as Fargo (Fine, smectitic, frigid Typic Epiaquerts), Bearden (Fine-silty, mixed, superactive, frigid Aeric Calciaquolls), Glyndon (Coarse-silty, mixed, superactive, frigid Aeric Calciaquolls), Williams (Fine-loamy, mixed, superactive, frigid Typic Argiustoll), and Delamere (Coarse-loamy, mixed, superactive, frigid Typic Endoaquolls) soil series (Table 5.). Particle size distributions were determined by sedimentation using the pipette method (USDA, 2014). Inorganic C (IC) and total C (TC) were determined by high-temperature combustion (Primacs TOC Analyzer, Skalar Analytical B.V., Breda, Netherlands) where soil organic C (SOC) was the difference between TC and IC. Soil pH and electric conductivity (EC) were determined using 1:1 ratios of soil:deionized water (DI) using a pH electrode (AB15, Fisher Scientific, Hampton, NH, United States) and EC electrode (Senslon378, Hach, Loveland, CO, United States), respectively. Semi-quantitative clay speciation and mineralogical analysis was conducted by X-ray diffraction (Code 9 XRD, Activation Laboratories Ltd, Ancaster, ON, Canada) (Table 6).

### **Polymer Properties**

Superabsorbent polymer (sodium polyacrylate, crosslinked; 9003-04-7, Zappa Tec LLC, McLeansville NC), had white grains ranging from 0 to 3 mm in size. The manufacturer reported a pH range of 5.5 to 6.5, bulk density of 0.6 to 0.9 g cm<sup>-3</sup> and DI water absorbency range of 240 to 300 g g<sup>-1</sup>.

Table 5. Basic properties for soils used in series of lab experiments.

Soil	Sand	Silt	Clay	SOC	IC	pH	EC
	-----g kg <sup>-1</sup> -----						dS m <sup>-1</sup>
Fargo	27	456	517	33.2	1.1	7.5	0.42
Williams	382	319	298	14.9	0	7.8	1.87
Glyndon	195	512	293	27.2	3.9	8.7	3.91
Bearden	563	235	202	17.7	2.7	8.4	1.42
Delamere	756	149	95	10.6	0	7	0.16

Table 6. Mineralogical analysis and semi-quantitative clay speciation.

Mineral (% by weight)	Soil				
	Fargo	Williams	Glyndon	Bearden	Delamere
Quartz	26.3	52.7	36	37.3	67
Plagioclase	5.7	15.4	8.8	7.7	9.1
K Feldspar	3.1	4.7	6	5	5.4
Muscovite/Illite	4	4.9	2.4	3.1	1.6
Kaolinite	1	0.6	1	0.5	trace
Chlorite	trace	0.5	trace	0.5	trace
Smectite†	12	10	12	7	4
Amphibole	n.d.‡	n.d.	n.d.	1.2	n.d.
Heulandite	0.3	n.d.	n.d.	n.d.	0.3
Dolomite	n.d.	n.d.	1.1	0.9	n.d.
Calcite	n.d.	n.d.	n.d.	0.7	n.d.
Amorphous	47.6	11.2	32.7	36.1	12.6
Clay fraction (% by weight)					
Smectite	70	62	81	65	68
Illite	24	30	15	28	26
Kaolinite	4	4	4	4	4
Chlorite	2	4	trace	3	2

† the amount of smectite is a rough estimate calculated from the relative proportions of smectite and illite in the <2 µm size fraction.

‡ n.d. = not detected

### Sample Preparation and Experimental Setup

Soil samples were air-dried and ground to pass through a 2 mm sieve and the polymer was passed through a 1 mm sieve. The SAP: soil treatments for all laboratory studies were 0x, 1x, 2x, and 5x, where 1x was 4 g SAP kg<sup>-1</sup> dry soil. Application rate 1x was based on water absorbency at a soil bulk

density of  $1.32 \text{ g cm}^{-3}$  and 65% water-filled pore space (see appendix A for calculations). Each SAP: soil sample was dry mixed in plastic bags before experiment use.

### **Study 1: Water Retention**

Water retention at -10 and -1,500 kPa matric potentials was determined using pressure plates (Soil Moisture Equipment Corp., Santa Barbra Ca, USA) (Richards, 1948; Richards Fireman, 1943). Each sample was 7.5 g of dry soil mixed with respective SAP rate, replicated three times. Prior to pressurization, SAP: soil mixtures were poured into rubber rings on the pressure plate and saturated for 0.5 h by capillary rise with non-degassed DI water. Soils less than and greater than 30% clay were subjected to pressures for 72 and 96 h, respectively, until drainage ceased. Gravimetric water contents were then determined by weighing samples before and after drying at  $105 \text{ }^{\circ}\text{C}$  for 48 and 72 h for <30% and >30% clay samples, respectively. The gravimetric water contents were converted to a volumetric water content using a standard bulk density of  $1.32 \text{ g cm}^{-3}$ . Plant available water (PAW) was determined as the difference between volumetric water content at FC and permanent wilting point (PWP, -1,500 kPa).

### **Study 2: Liquid Limit**

The LL, which is the water content corresponding to 20 mm of cone penetration, was determined using a tabletop penetrometer (Model-S165-02KIT, Humboldt Manufacturing, Raleigh, NC, United States) according to the multi-point fall cone method (British Standard Institution 1377-2, 1990). SAP:soil mixtures were prepared with 400 g of soil and respective SAP mass added to this soil to achieve the 0x, 1x, 2x, and 5x rates, each replicated three times. Distilled water was incorporated into mixture and given 0.5 h to equilibrate. The volume of water added was dependent on soil texture and SAP application rate, but the amount added targeted an initial penetration reading of 15 mm (data not shown). Next, 150 g subsamples from the SAP: soil mixture were packed into brass cups with a palette knife with as few strokes as possible and leveled with top of cup. With the cone penetrometer locked in its raised position, the cone was lowered to just touch the soil surface near the center of the brass cup, and electronic meter set to 0 mm. The cone was then released for 5 s to penetrate soil, locked in place, and the electronic meter used to determine the depth of penetration. As stated above, the addition of water for each soil was targeted to achieve a first penetration value of about 15 mm, which for the Delamere soil (0x) was 104 mL and was 188 mL for the 5x treatment. With the cone removed, approximately 10 g of SAP: soil mixture

from cone penetration location was quantified for gravimetric water content. Additional DI water was then added, mixed, allowed to equilibrate for 0.5 h, and penetration procedure repeated for a total of five times (below, at, and above LL). The target range for five penetration values was 15 to 25 mm, with two values below the LL, one around the LL, and two above. The penetration values were used to interpolate the water content where the LL occurs.

### **Study 3: Evaporation**

To determine SAP effects on evaporation a 30 d study was conducted in a temperature controlled (CT) room where temperature was  $21\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  and relative humidity was  $41\% \pm 5\%$  (General HT20, New York City, NY, United States). Using  $7.62 \times 7.62$  cm polyvinyl chloride (PVC) columns, two layers of tile drain cloth were stretched across their bottoms and secured with rubber bands. In each column, 150 g of each SAP: soil mixture was added in 50 g increments. To achieve uniform packing, a 3.2 kg weight was dropped twice from 0.61 m to strike a rubber stopper placed on the soil surface after each of the first two increments were added. For the third increment the weight was dropped four times. Average soil bulk densities across all SAP: soil mixtures ranged between  $1.3\text{ g cm}^{-3}$  and  $1.45\text{ g cm}^{-3}$ . After packing, samples were saturated to their surfaces with non-degassed DI water via capillary rise. Before the start of evaporation, the open column bottoms were covered with two layers of Saran wrap (S.C. Johnson & Son, Inc, polyethylene food wrap) and sealed with electrical tape. Within the CT room, columns were rotated daily within each block to minimize potential location variation. Columns were weighed every 24 h ( $\pm 1$  h) for the duration of the study. Evaporation rate was calculated as mass loss of water between measurements. Stage one duration was determined as the number of days before evaporative water loss was monotonically decreasing. The day before and the first day of consistent decrease were averaged to yield stage one duration. Next, stage one and stage two mean evaporation were determined.

### **Study 4: Compression**

Using the PVC columns and SAP:soil mixtures preparation procedure as in Study 3, each treatment was exposed to six freeze-thaw cycles, with one cycle defined as 24 h at  $-6\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  (freezing) and 24 h thawing at  $21\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  at  $41\% \pm 5\%$  relative humidity. Trial runs were conducted to determine time and temperature to freeze or thaw entire volume of soil. Compression was determined before and after freeze thaw cycles by using a force gauge modified with a  $60^{\circ}$ , 1.905 cm long cone, and handle,

each threaded into load cell (Extech 475055, Boston, MA, United States). Readings were taken within a 4cm<sup>2</sup> area centered in the column. With the cone positioned on the soil surface, pressure was applied to handle with consistent downward force until the base of the load cell was reached and the peak compression (gram force [gf]) was recorded from the force-gauge digital display. Change in compression was the difference between compression before and after each freeze-thaw cycle.

### **Study 5: Saturated Hydraulic Conductivity**

Using the PVC columns and SAP: soil mixtures preparation procedure as in Study 3, saturated hydraulic conductivity (K<sub>sat</sub>) before and after freeze-thaw cycles was determined by constant head method (Han, 2013). A Mariotte bottle of non-degassed water with 0.005 M CaCl<sub>2</sub> was used to maintain a constant head at a 2 cm ponded depth; a scouring pad 1.02 cm thick was placed on the sample surface to minimize disturbance by solution. Once a constant head was applied, samples were allowed to drain freely for 0.08 h before measuring the volume of discharge per time. Saturated hydraulic conductivity was calculated using Darcy's law:

$$K = \frac{Q}{Ax} \left( \frac{dL}{dh} \right) \quad (1)$$

where K is the saturated hydraulic conductivity (cm s<sup>-1</sup>), Ax is the cross-sectional area of sample (cm<sup>2</sup>), Q is the discharge (cm<sup>3</sup> s<sup>-1</sup>), dL is sample length (cm) and, dh is the hydraulic head gradient which is the 2 cm ponded depth plus sample length (cm). Change in K<sub>sat</sub> was the difference between K<sub>sat</sub> before and after undergoing freeze-thaw cycles.

### **Statistical Analysis**

The effects of SAP application rate, soil texture and their interaction on water retention and LL, evaporation metrics, compression, and saturated hydraulic conductivity were analyzed using a factorial analysis of variance (ANOVA) general linear model procedure with pairwise comparisons conducted with Tukey honestly significant difference (HSD) post-hoc at  $\alpha = 0.05$ . Water retention and LL statistical tests were performed with SAS 9.4 (SAS 19; Cary, NC) by North Dakota State University statistical consulting service. Remainder of statistical tests were performed with R 3.6.1 software (R Core Team, 2019).

## **Results and Discussion**

### **Study 1: Water Retention**

Superabsorbent polymer application rate, soil texture, and their interaction significantly affected water retention (Table 7 and Table 8). Field capacity and PWP significantly increased as application rate

increased for all soils except the Bearden, which showed no differences across application rates. In general, FC increased more than PWP resulting in an increase in PAW with application rate (Table 7 and Table 8).

Table 7. Analysis of variance summary for superabsorbent polymer application rate, soil texture and their interaction on plant available water (PAW,  $\text{cm}^3 \text{cm}^{-3}$ ), liquid limit (LL,  $\text{g g}^{-3}$ ), stage 1 evaporation duration (days), stage 1 evaporative water loss ( $\text{g kg}^{-1}$ ), stage 2 evaporative water loss ( $\text{g kg}^{-1}$ ), compression, and saturated hydraulic conductivity ( $\text{Ksat}$ ,  $\text{cm d}^{-1}$ ).

Study	DF	SSE	MSE	F value	p value
PAW					
Rate	3	0.13	0.13	400.96	<0.001
Texture	5	0.08	0.00	34.04	<0.001
Texture*Rate	15	0.08	0.02	62.46	<0.001
LL					
Rate	3	36,293	0.00	96.90	<0.001
Texture	5	11,082	2,216	17.75	<0.001
Texture*Rate	15	7,722	514.84	4.12	<0.001
Stage 1 evaporation duration					
Rate	3	333.20	111.06	34.03	<0.001
Texture	5	1,010	202.60	61.91	<0.001
Texture*Rate	15	155.80	10.39	3.18	<0.01
Stage 1 evaporative water loss					
Rate	3	6.23	2.08	67.77	<0.001
Texture	5	17.15	3.43	111.99	<0.001
Texture*Rate	15	1.34	0.09	2.92	<0.01
Stage 2 evaporative water loss					
Rate	3	3.14	1.11	24.90	<0.001
Texture	5	1.03	0.21	4.65	<0.01
Texture*Rate	15	1.03	0.07	1.54	0.06
Compression					
Rate	3	22.38	7.46	5.75	<0.01
Texture	5	36.99	7.40	5.70	<0.001
Texture*Rate	15	44.69	2.98	2.30	<0.05
Ksat					
Rate	3	2,794	931.20	445.40	<0.001
Texture	5	1,494	298.80	142.90	<0.001
Texture*Rate	15	4,164	277.60	132.80	<0.001

The largest increase in PAW was in the Williams soil (sand content of  $382 \text{ g kg}^{-1}$ ), which increased from  $0.12 \text{ cm}^3 \text{cm}^{-3}$  to  $0.34 \text{ cm}^3 \text{cm}^{-3}$  at the 0x and 5x rate, respectively. The change in PAW



from the 0x to the 5x was 0.16, 0.08, 0.22, 0.01 and 0.06 cm<sup>3</sup> cm<sup>-3</sup> for Fargo, Glyndon, Williams, Bearden, and Delamere, which had sand contents of 27, 195, 382, 563, 756 g kg<sup>-1</sup>, respectively (Table 5).

Montesano et al. (2015) observed somewhat similar results as the study presented here. They reported an increase in water retained at FC in a sandy soil with 0.5% SAP rate BM. At PWP, water retained only increased when SAP was applied at a 2% BM. In comparison to our study, their 0.5% rate is 2.5 times higher than the 5X rate used in our study. Therefore, we observed significant effects in water retention at lower application rates. Abrisham et al. (2018) also observed similar trends. They reported soil PAW to significantly increase from 0.03 cm<sup>3</sup> cm<sup>-3</sup> to 0.06 and 0.09 cm<sup>3</sup> cm<sup>-3</sup> for a sandy loam soil with 0.07% and 0.2% SAP application rates, respectively, on a mass basis. Finer textured soils, such as Fargo and Williams used in the present study, innately retain more water because of their clay content. Even though PAW significantly increased by 0.16 and 0.22 cm<sup>3</sup> cm<sup>-3</sup> at the 5x rate for the Fargo and Williams soils, respectively, this additional water may not necessarily translate into improved crop yields since there is already adequate water for growth without the SAP. Although the increases in PAW were less than 0.1 cm<sup>3</sup> cm<sup>-3</sup> for the Glyndon, Bearden, and Delamere soil series, small increases in PAW can be valuable to help increase plant yields in arid regions or areas with coarse textured soils (Prabha et al., 2014; Abrisham, 2018).

## **Study 2: Liquid Limit**

Superabsorbent polymers have the potential to change a soil's mechanical behavior as a function of water. Such changes in soil mechanical behavior may be observed via shifts in the LL, plastic limit (PL), and plasticity index (PI; where  $PI = LL - PL$ ). The granular characteristics of the SAP used in the present study prevented us from being able to determine the PL since the soils tended to lose their cohesiveness and fall apart during the standard rolling method. However, the SAP rate, texture, and their interaction significantly affected the LL (Table 7 and Table 8). In general, LL increased with an increase in SAP rate for all soils except the Glyndon and Bearden soils. The 5x application rate increased the LL from 0.53 to 1.30 g g<sup>-1</sup> for the Fargo, 0.30 to 1.13 g g<sup>-1</sup> for the Williams, and 0.35 to 1.11 g g<sup>-1</sup> for the Delamere soil series.

Table 8. Mean values for volumetric water content (VWC) calculated from a  $1.32 \text{ g cm}^{-3}$  bulk density, at field capacity (FC), permanent wilting point (PWP), and plant available water (PAW= FC-PWP) where FC is at -10 kPa. Mean values for gravimetric water content (GWC) at the liquid limit (LL).

Series	SAP Rate	FC	PWP	PAW	LL
		----- $\text{cm}^3 \text{ cm}^{-3}$ -----			$\text{g g}^{-1}$
Fargo	0†	0.18A‡	0.04A	0.14A	0.53A
	1	0.20A	0.04A	0.16AB	0.74A
	2	0.22A	0.05A	0.17B	0.86A
	5	0.28B	0.08B	0.30C	1.30B
Williams	0	0.13A	0.01A	0.12A	0.35A
	1	0.19A	0.02A	0.17AB	0.63AB
	2	0.24B	0.02A	0.22B	0.79BC
Glyndon	5	0.43C	0.09B	0.34C	1.13C
	0	0.16A	0.02A	0.14A	0.34A
	1	0.17AB	0.02A	0.15AB	0.45A
	2	0.20B	0.02A	0.18B	0.53A
Bearden	5	0.27C	0.05B	0.22C	0.63A
	0	0.18A	0.03A	0.15A	0.46A
	1	0.18A	0.03A	0.15AB	0.49A
	2	0.18A	0.03A	0.15B	0.52A
Delamere	5	0.19A	0.03A	0.16C	0.65A
	0	0.04A	0.01A	0.02A	0.30A
	1	0.08B	0.04B	0.04A	0.55AB
	2	0.10C	0.03B	0.07B	0.72B
	5	0.14D	0.06C	0.08B	1.11C

† Application rate of 0, 1, 2, 5 correspond to 0, 0.04, 0.08, and 0.2% SAP: soil, respectively.

‡ Means within soil series and column followed by the same letter are not significantly different at  $P < 0.05$ .

### Study 3: Evaporation

Superabsorbent polymers have the ability to increase water in the soil and consequently have the potential to change evaporation dynamics. Application rate, texture, and their interaction significantly affected stage one evaporation duration as well as stage one and two evaporative water loss (Table 7 and Table 9). Stage one evaporation is limited by atmospheric conditions, and evaporation rate is near constant in this stage (Suleiman and Ritchie, 2003; Hillel, 1980). Overall, SAP application increased stage one duration, decreased evaporative water loss during stage one, and increased evaporative water loss for stage two (Table 9). This is consistent of what can be expected from the higher water retention at FC and PWP as previously described above. In general, this means that evaporative losses over time were moderated by increases in SAP application rate. At the 5x application rate, SAP significantly increased

the stage one duration for all soil series by 2.3 to 7.7 d. Although there was an increase in stage one duration, the decrease in evaporative water loss in stage one allows for more water to be retained in the soil. This is important since stage one evaporation is often the largest fraction of the total soil surface evaporation loss (Qiu and Ben-Asher, 2010) and thus influences the amount of water available for plants and available for expansion at the time of freeze-thaw cycles to alleviate soil compaction. In coarser textured soils which, inherently hold less water, SAPs prolong the amount of time there is water available to plants (Abrisham et al., 2018). For example, the coarsest soil series in this study, the Delamere, had significantly decreased evaporative water loss during stage one from 1.83 to 1.37 g kg<sup>-1</sup>, at 1x SAP rate. Given the Fargo, Williams, Glyndon and Bearden soil series, did not have a significant decrease in stage one evaporative water loss at 1x rate indicates that more SAP is needed to decrease evaporative water loss in the constant rate stage.

Table 9. Mean values for stage 1 evaporation duration, stage 1 and 2 evaporative water losses.

Series	SAP Rate	Stage 1 Evaporation Duration	Stage 1 Evaporative water loss	Stage 2 Evaporative water loss
		Days	-----g kg <sup>-1</sup> -----	
Fargo	0†	5.50A‡	2.79A	0.40A
	1	6.50A	2.61A	0.64B
	2	8.50B	2.41A	0.85C
	5	7.80B	1.91B	1.05D
Williams	0	11.2A	1.90A	0.34A
	1	17.8A	1.60AB	0.76A
	2	16.8A	1.24BC	1.28A
	5	18.5A	0.89C	1.03A
Glyndon	0	6.50A	2.47AB	0.37A
	1	6.80B	3.01A	0.51AB
	2	9.50C	2.27AB	0.72B
	5	9.50C	1.98AB	1.14C
Bearden	0	5.80A	2.60A	0.34A
	1	5.80A	2.38A	0.42AB
	2	6.50A	1.93B	0.57BC
	5	9.80B	1.73B	0.75C
Delamere	0	10.1A	1.83A	0.42A
	1	12.8A	1.37B	0.68A
	2	19.1B	1.23B	0.99A
	5	17.8B	0.99C	0.74A

† Application rate of 0, 1, 2, 5 correspond to 0, 0.04, 0.08, and 0.2% SAP: soil, respectively.

‡ Means within soil series and column followed by the same letter are not significantly different at P < 0.05.

The second stage of evaporation is characterized by a monotonic decrease in evaporative water loss, also referred to as the falling rate stage until a new equilibrium rate is gradually obtained (Hillel, 1980). The second stage is dependent on soil properties such as hydraulic conductivity and pore size distribution (Shokri et al., 2010). During stage one evaporation, the surface soil layers function to transmit water to the atmosphere with little or no resistance, whereas after the stage transition occurs, the surface soil layer acts primarily as a resistance layer to water vapor diffusion as water lower in the soil profile evaporates (Amano and Salvucci, 1999). Stage two evaporative loss generally increases with SAP application rate, which is attributed to SAPs retaining more water through the first stage of evaporation. The largest increase for stage two evaporative water loss was  $0.65 \text{ g kg}^{-1}$  for the Fargo soil series at the 5x rate.

Variability in stage two evaporative water loss in response to SAP application rate may be due SAP distribution, initial water absorbed and swelling. Superabsorbent polymers cause soil swelling (Han et al., 2010), which potentially changes pore size distribution and connectivity (Han et al., 2010; Prabha et al., 2014), which would change how water is transmitted and evaporated as well as the diffusive tortuosity for vapor transport. Similarly, the SAP granular size could dictate evaporation; for example Yu et al. (2012) found that water loss was faster for 3-4 mm diameter particles compared to 0.2-1.0 mm diameter particles. Given, that only four studies were identified and that they had inconsistent results in regards to evaporation (El-Asmar et al., 2017; Jabri et al., 2015; Yu et al., 2012; Zhao et al., 2019), future research should examine the effect of SAP characteristics and application rates on evaporation stages.

#### **Study 4: Compression**

Compression is used as a metric of compaction and the change in compression after soil: SAP mixtures undergoing six freezing and thawing cycles was significant for SAP application rate, texture, and their interaction (Table 7 and Table 10). The average decrease in soil compression across all textures was 76, 96 and 56 gf, for 1x, 2x, and 5x, respectively (Table 10). However, the Delamere soil had no significant changes in compression across any application rates.

Compression for Delamere the coarsest textured soil was not reduced at any SAP application rate, which indicates that decreasing soil compression with high concentrations of sands may not be possible with the rates of SAP used in this study, followed by freezing and thawing. However, based on

the results from the four other soils, SAPs' potential to reduce compression is promising such that the largest reductions occurred for the 1x or 2x rates and in the Glyndon and Bearden soils. Bai et al. (2010) reported somewhat similar results for a sandy clay loam for bulk density as a metric for soil compaction. They observed that a 0.3% by mass SAP application significantly reduced bulk density after four wetting and drying cycles, but not after freezing and thawing cycles. Both freezing and thawing and wetting and drying cycles occur in the field, thus a combination study with SAP: soil mixtures should be evaluated for the textures in which they will be used. While SAPs have shown reductions in compaction metrics (Bai et al., 2010; Hou et al., 2017; Abrisham et al., 2018), our research appears to be the first to examine the effect of freezing and thawing on compression.

Table 10. Mean values for change (final – initial) in compression and saturated hydraulic conductivity (Ksat) after undergoing six freezing-thawing cycles.

Series	SAP Rate	Compression change	Ksat change
		gf	cm d <sup>-1</sup>
Fargo	0†	-40.0A‡	5.22 x 10 <sup>-01</sup> A
	1	0.00B	2.67 x 10 <sup>-01</sup> A
	2	-70.0C	6.69 x 10 <sup>-01</sup> AB
	5	-40.0AB	-3.53 x 10 <sup>-00</sup> C
Williams	0	-7.00A	-2.06 x 10 <sup>-01</sup> A
	1	-10.0B	7.01 x 10 <sup>-01</sup> B
	2	-80.0C	-6.46 x 10 <sup>-01</sup> A
	5	-50.0ABC	-1.83 x 10 <sup>-00</sup> C
Glyndon	0	-20.0A	4.79 x 10 <sup>-01</sup> A
	1	-30.0B	6.97 x 10 <sup>-01</sup> A
	2	-110.0C	3.27 x 10 <sup>-01</sup> AB
	5	-90.0C	-3.65 x 10 <sup>-01</sup> AC
Bearden	0	-40.0A	1.32 x 10 <sup>-01</sup> A
	1	-330.0B	-1.56 x 10 <sup>-02</sup> B
	2	-200.0C	1.98 x 10 <sup>-01</sup> A
	5	-70.0A	1.15 x 10 <sup>-01</sup> A
Delamere	0	-10.0A	-1.45 x 10 <sup>-01</sup> A
	1	-10.0A	-1.18 x 10 <sup>-00</sup> B
	2	-20.0A	-1.18 x 10 <sup>-00</sup> B
	5	-30.0A	-7.89 x 10 <sup>-01</sup> C

† Application rate of 0, 1, 2, 5 correspond to 0, 0.04, 0.08, and 0.2% SAP: soil, respectively.

‡ Means within soil series and column followed by the same letter are not significantly different at P < 0.05.

## **Study 5: Saturated Hydraulic Conductivity**

Change in K<sub>sat</sub> after SAP: soil mixtures undergoing six freezing and thawing cycles was significant for SAP application rate, texture, and their interaction (Table 7 and Table 10). Saturated hydraulic conductivity for Delamere, the coarsest soil series, had a -0.15, -1.18, -1.18, and -0.80 cm d<sup>-1</sup> change after the freeze-thaw cycles for the control, 1x, 2x, and 5x application rates, respectively. In contrast, Fargo, Williams, and Glyndon have high clay contents, which are dominated by 2:1 smectite, a high shrink swell clay. The 5x SAP application rate significantly decreased K<sub>sat</sub> relative to the control from 0.52 to -3.53 cm d<sup>-1</sup>, -0.21 to -2.12 cm d<sup>-1</sup>, and 0.55 to -0.18 cm d<sup>-1</sup> for Fargo, Williams, and Glyndon soil series, respectively. The shrinking and swelling may be creating micropores or reducing connectivity which would reduce K<sub>sat</sub> in some soils. Whereas the reduction in K<sub>sat</sub> for Delemere, however, may be attributed to SAPs swelling and filling pore space, which restrains water movement (Han et al., 2013). The variability in these results may be a function of heterogeneous distribution of the SAP within the columns. If SAP particles tended to concentrate in some regions as compared to being evenly dispersed within some columns, then the variability in water flow pathways and K<sub>sat</sub> would be expected to be variable among columns.

A few studies have examined hydraulic conductivity response to SAP application. Initially, reduction of hydraulic conductivity occurred with SAP application, (Bhardwaj et al., 2007) however, increased with time (Han et al., 2013). Change in K<sub>sat</sub> with time was attributed to shrinking and swelling of polymers undergoing wetting-drying cycles (Han et al., 2013). While studies have examined SAP: soil mixtures influence on K<sub>sat</sub>, the effects of freezing-thawing have not been included.

### **Implications for Alleviating Soil Compaction**

Superabsorbent polymers have the potential to retain water for longer periods and if more water is retained in soils at the time of freeze-thaw cycles, then the potential for expanding ice to alleviate soil compaction would be improved. This series of studies provides evidence that increased SAP rates retain more water, lower evaporative water losses, and increase soils' LL (i.e., soil will remain more rigid for fracturing during freeze-thaw). This evidence implies that SAP may improve soil conditions at the time of winter freezing so that expanding ice may enhance alleviation of soil compaction. Moreover, this study also provides evidence that soil compression and K<sub>sat</sub> are significantly decreased following SAP

applications with subsequent freeze-thaw cycles. Therefore, the SAP-enhanced soil conditions at the time of freezing appear to translate into metrics that indicate alleviation of soil compaction is being enhanced.

### **Conclusions**

The objective for these studies was to evaluate the response of soil physical properties to SAP application. Superabsorbent polymer application increases the amount of water within the soil, as well as, prolonged water retention time by increasing stage one evaporation duration while decreasing evaporative water loss during this stage. With more water available for longer time periods SAPs can be utilized to mitigate drought stress and increase the time between irrigation intervals. After undergoing six freeze-thaw cycles, compression and saturated hydraulic in SAP treated soils were significantly altered. Improved soil conditions by SAP water retention increase the likelihood that upon freezing, expansion of ice will alleviate soil compaction, as seen with SAP treated soils having significantly reduced compression. Application of SAP has the ability to improve soil physical properties, which could potentially translate to improved soil function and furthermore productivity. To further understand the ability of SAPs to promote compaction alleviating conditions, different application methods, rates, compaction severity, water content, and number of freezing and thawing cycles should be examined for the soils to which SAPs will be applied.

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## **CHAPTER 4. REDUCING COAL MINE RE-SPREAD COMPACTION IN NORTH DAKOTA USING TILLAGE, MULCH AND A SUPERABSORBENT POLYMER**

### **Abstract**

Surface coal mining faces the challenge of returning land to pre-mine productivity while combating subsurface soil compaction. The heavy machinery that is used for removal and re-spread of soil often causes compaction at levels that limit plant productivity. However, recent research has indicated that the natural process (wetting-drying and freezing-thawing) for alleviating soil compaction has not been efficient or effective at North Dakota coal mines. The objectives of this study were to evaluate the effects of deep ripping (R), SAP, and surface applied mulch (M), and their combinations on penetration resistance and hard-red spring wheat productivity and protein content. The use of R, alone or in combination with SAP or M, most notably increased wheat yield compared to the control; the greatest yield was for R and SAP combination. There were no treatment effects on wheat protein. Similarly, the use of R in all combinations decreased penetration resistance. Understanding the short-term effectiveness of tillage treatments and amendments to reduce penetration resistance and the implications on productivity will further the success of reclamation.

### **Introduction**

Surface coal mining faces the challenge of returning land to pre-mine productivity while combating subsurface soil compaction. Severe compaction is associated with mineral extraction and soil reconstruction (Dunker et al., 1992), and is the leading problem for North Dakota surface coal mine reclamation (D. Moos, personal communication, North Dakota Public Service Commission, Bismarck ND, 2018). The heavy machinery for removal and re-spread of soil often causes compaction at levels that limit plant productivity. Historically, the winter freeze-thaw cycles were assumed to help alleviate soil compaction over time. However, recent research has indicated that this natural process for alleviating soil compaction has not been efficient or effective at North Dakota coal mines (Bohrer et al., 2017). The alleviation of compacted mine reclamation sites appear to need strategic management and thus this research will examine tillage combinations and SAPs ability to reduce compaction, and increase efficiency of meeting plant productivity reclamation standard.

Tillage is a mechanical method that has been used to reduce compaction. The use of deep ripping or subsurface tillage was introduced in 1977 on reclaimed prime farmland to meet Surface Mining and

Control Reclamation Act (SMCRA) yield standards (Sweigard et al., 2007). By breaking up the compacted root restricting layer, plants are then able to access resources lower in the profile. Yields have been shown to increase following subsoiling due to increased moisture utilization (Kamprath et al., 1979) and studies have shown that deep tillage can improve productivity of mined land (Bledodsoe et al., 1992; Dunker et al., 1992). Initially, improvements are seen with tillage, however, loosened soil can be prone to resettling, causing increased compaction. Deep ripping does reduce compaction in a reclamation setting (Dunker et al., 1992), however, the longevity of tillage treatments in severely compacted mine systems is unknown. It is important to reevaluate traditional and new methods to continually improve ecosystem function (agricultural productivity) and the reclamation process.

Once land has undergone a significant disturbance it is imperative that the reclamation process is successful and efficient in order to bring disturbed land back to proper function. The objectives of this field research were to i) quantify penetration resistance as a function of tillage (i.e., chisel plow and deep ripper), soil amendments (i.e., super- absorbent polymer and surface applied straw consisting of russian wildrye, intermediate wheatgrass, pubescent wheatgrass, and alfalfa [*Elymus junceus*, *Agropyron intermedium*, *Agropyron trichophorum*, *Medicago sativa*, respectively]), and their combinations, ii) quantify hard-red spring wheat response to the tillage and soil amendment treatments. Findings from this research will provide information on the short-term effectiveness of tillage treatments and amendments to reduce penetration resistance, how reduction translates to productivity and furthermore, the success of reclamation.

## **Materials and Methods**

### **Site Description**

Research plots were located at the Falkirk mining company, approximately 80 km north of Bismarck in McLean County North Dakota (47°19'51.2"N, 101°12'37.51"W) (Figure 6). The regional climate is classified as snow, fully humid and warm summer (Dfb) (Kottek et al., 2006). The total rainfall from May to September in 2018 and 2019 was 29.2 cm and 44.3 cm , respectively; total potential evaporation (Penman) from May to September in 2018 and 2019 was 109.9 cm and 100.9 cm, respectively (NDAWN - Turtle Lake, 2017).

The research plot area was mined from day of year (DOY) 292 in 2014 to DOY 211 in 2015. This area was considered prime farmland which warranted 1.2 m of soil replacement, with 0.2 m being topsoil.

Soil replaced was mixed mine land soil regraded 0 to 3% slope that contained Williams (Fine-loamy, mixed,superactive, frigid Typic Argiustoll), Falkirk(Fine-loamy, mixed, superactive, frigid Pachic Haplustoll), Flaxton (Fine-loamy, mixed, superactive, frigid Pachic Argiustoll), and various complex soil series with texture class resulting from the top 0.15 m being silty clay loam. The effects of SAP, M, and tillage in eight treatment combinations were evaluated using a randomized complete split block design with three replicates. Chisel disc (CD) treatment was the control. Each plot was 0.07 ha (Figure. 7). The split-plot design was used to accommodate planting and harvesting of both the pre-crop mix (a typical post-mining practice) and row crops.

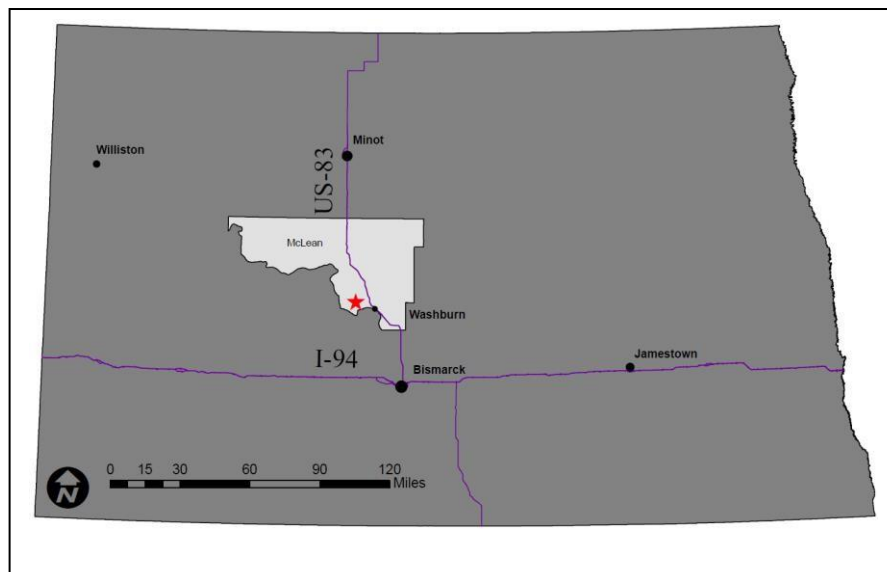


Figure 6. Study site location is denoted by the star in McLean County, ND.

### **Treatment Installation and Plot Management**

Plots were established DOY 163 in 2018, when 1 m of subsoil was returned by a 789 Cat end dump truck, and respread with a D11 Cat dozer. Superabsorbent polymer (sodium polyacrylate, crosslinked; 9003-04-7, Zappa Tec LLC, McLeansville NC) had white grains ranging from 0 to 3 mm in size. The manufacturer reported a pH range of 5.5 to 6.5, bulk density of 0.6 to 0.9 g cm<sup>-3</sup> and DI water absorbency range of 240 to 300 g g<sup>-1</sup>. Superabsorbent polymer was applied on 193 DOY, at 25.8 kg ha<sup>-1</sup> onto the subsoil surface using Stihl® SR 450 backpack sprayer/duster (SR 450 Stihl®, Virginia Beach, VA, United States.) set to dry mode. Trial runs were conducted to determine working width of 4 m and discharge rate of 0.53 kg min<sup>-1</sup>, which for this unit was at 1/3 throttle with metering lever 7 clicks away from closed at a walking speed 0.89 m s<sup>-1</sup>. After

polymer application, D11 Cat dozers respread 0.22 m of topsoil on DOY 211 in 2018. A three shank DM1 subsoiler was used at a working depth of 0.56 m for R treatments and, Wishek disc and John Deere chisel plow with spikes were used to chisel and disc plots at a working depth of 22.9 cm on DOY 233 in 2018.

After tillage installation, M was broadcasted at 23.5 kg ha<sup>-1</sup>, which had a ratio of 4:7:7:3 of Russian wildrye, intermediate wheatgrass, pubescent wheatgrass, and alfalfa. In 2019 hard red spring wheat (HRSW) SY Ingmar variety, was seeded DOY 135, using a drill seeder at a population of 123.3 kg ha<sup>-1</sup> in 20 cm rows. Concurrently, 65.0 kg ha<sup>-1</sup> of Mesz 10 fertilizer (12N, 4P, 0K, 10S) was applied to wheat plots. Weed control in the wheat was accomplished with Huskie Complete (1,001 mL ha<sup>-1</sup> (a.i. 33, 134, and 129 mL of Pyrasulfotole, Bromoxynil Octanoate, Bromoxynil Heptanoate, respectively), Olympus (14.6 mL ha<sup>-1</sup> (a.i. 10.2 ml of Propoxycarbazone-sodium) and Promax (219 mL ha<sup>-1</sup> (a.i. 108.6 ml of Glyphosate) on DOY 163 in 2019. Pre- crop mix, which had the same composition of M was broadcasted at 23.5 kg ha<sup>-1</sup> DOY 183 in 2019. Weeds were not controlled in the pre-crop mix.

### **Data Collection**

Using a research combine, 15 m<sup>2</sup> from each plot was harvested DOY 220 in 2019, and the yield was adjusted to 13.5% moisture. Grain samples were dried at 78 °C for 72 hours, ground to pass a 0.5 mm sieve, and sent to Agvise Laboratories in Northwood, ND to be analyzed for total N using combustion method. Protein was then estimated by multiplying total N by a 5.6 constant.

Penetration resistance, bulk density and, volumetric soil water contents were taken concurrently on DOY 240-241 in 2019. Penetration resistance was determined using FarmQA penetrometer (CTS-1000, Amity Technology, Fargo, ND, United States). The electrically driven penetrometer measured the voltage draw off the battery to determine penetration approximately every 1.02 cm based on the cone index. A Samsung tablet connected to Garmin GPS (Garmin Ltd., GLO 2, Olathe, KS, United States), recorded penetration results by bluetooth connection to penetrometer. Results stored on tabled, were synced to FarmQAs platform, and exported for analysis. Around each access tube, within 0.5 m, three penetration readings were taken.

The day after planting, access tubes were installed in the plant row for each plot using an auger, shaper tool and slide hammer and the volumetric soil water contents at 0.1, 0.2, 0.3, 0.6, and 1.0 m were determined using a PR2 Profile Probe (Delta-T Devices, Cambridge, United Kingdom). Soil cores (7.62 cm

diameter) were taken within 0.5 m of access tubes using a hydraulic probe. Soil samples, 5.1 cm in length and centered to correspond to each sensor depth along the PR2 Profile probe, were obtained to quantify BD, volumetric water content, and gravimetric water content. Bulk density and volumetric water contents were used to compare PR readings; where volumetric water contents at depths between those directly measured were linearly interpolated.

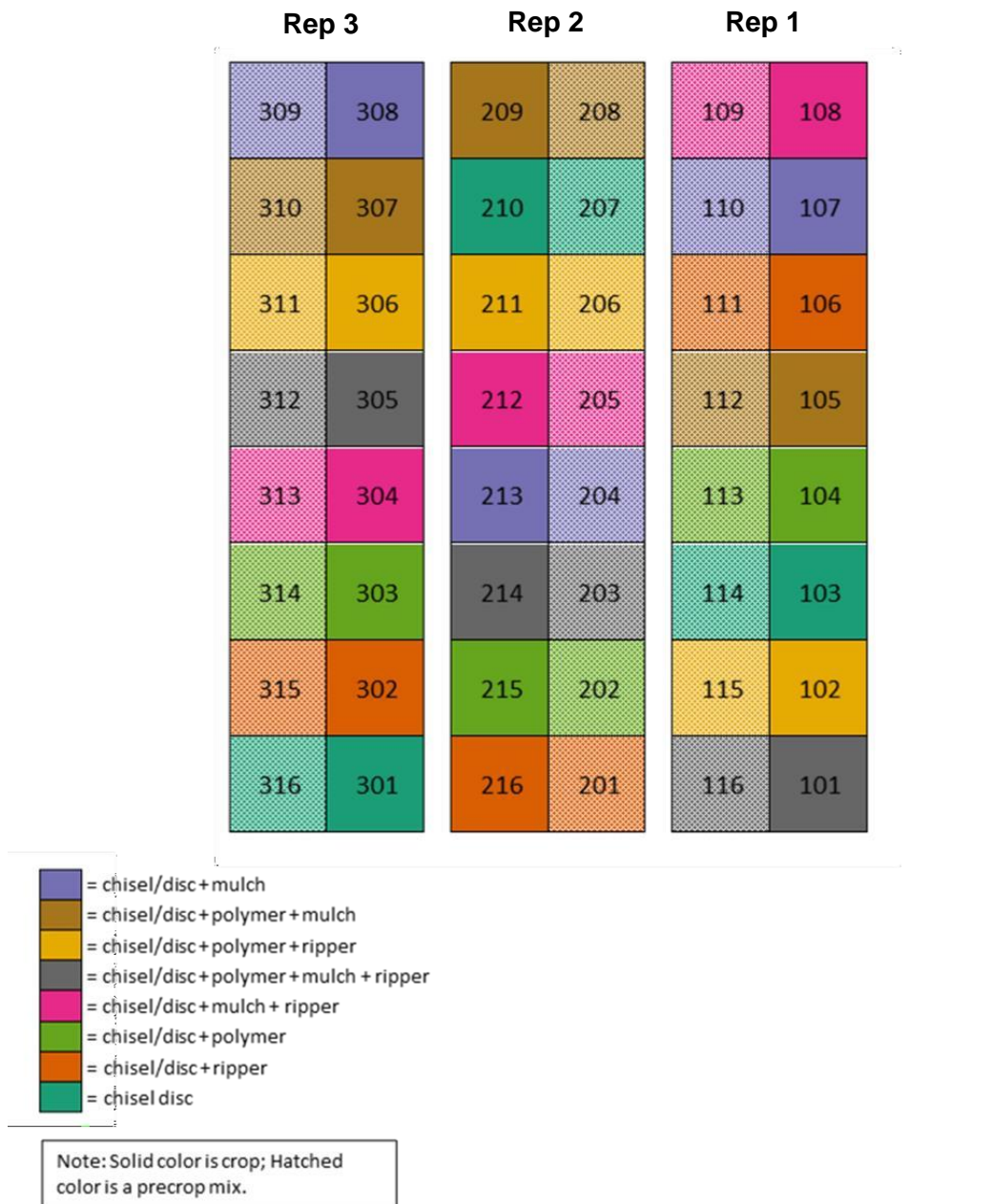


Figure 7. Field site complete split-plot design.

## Statistical Analysis

The effects of tillage, M, SAP, and their interactions on wheat yield and grain protein were analyzed using a factorial analysis of variance (ANOVA), general linear model with Tukey honestly significant difference (HSD) post-hoc test to separate means in R 3.6.1 statistical software (R Core Team, 2019). Soil PR, BD, VWC were analyzed for five depth intervals (0-15, 15-25, 25-35, 35-50 and 50-80 cm), where the PR value reported for each treatment is the mean PR within the designated depth. Penetration resistance, BD and VWC results were then statistically analyzed the same way as wheat yield.

## Results and Discussion

### Yield and Grain Quality

The main objective of this study was to determine the response of wheat yield to soil amendments [SAP and M] and R (Table 9). Mined land is legally required to be returned to pre-mine productivity (NDPSC, 2009). Therefore, the effectiveness of the post-mining soil treatments is essential to meet the pre-mine productivity criteria. After one growing season, treatments that received R significantly increased yield from 0.06 to as much as 1.38 Mg ha<sup>-1</sup>. Simmons et al. (1992) observed a significant increase in corn (*Zea mays*) yield with R compared to chisel disking, which was attributed to deeper alleviation of soil compaction. While the CD + SAP + R treatment in the present study improved yields to 1.38 Mg ha<sup>-1</sup>, these yields are only 43% of the 2018 McLean County average of 3.20 Mg ha<sup>-1</sup> (USDA-NASS, 2019). Similarly, Henning et al. (1992) reported that after two growing seasons, corn and soybean (*Glycine max*) yields were less than 55% of the county average. Soil structure can be weak or destroyed as a result of the soil reconstruction process (Dunker and Barnhisel, 2000), which may result in root growth being restricted and decreased yields.

Ripping and SAP were the treatments that had the most consistent influence on increasing wheat yields. This result gives promise but having increased site years (locations and years) is needed to further evaluate the use of SAP for increasing crop yields. However, wheat yields were not different from the control when M was added with SAP. Therefore, the M appeared to interfere with the SAP due to unknown reasons. There were no significant differences in protein content across treatments, which indicate that treatments did not affect the wheat's ability to uptake N (Table. 11).

Table 11. Mean yield and crude protein content for hard red spring wheat (HRSW) Ingmar variety.

Treatment	Yield	Crude Protein
	Mg ha <sup>-1</sup>	%
CD <sup>†</sup>	0.60A <sup>‡</sup>	16.9A
CD + M	0.68A	17.9A
CD + M + SAP	0.73A	18.0A
CD + M + SAP + R	1.08B	19.8A
CD + SAP	1.14B	17.2A
CD + M + R	1.15B	18.6A
CD + R	1.18BC	17.0A
CD + SAP + R	1.38C	19.6A

\* Significant at P < 0.05

<sup>†</sup> CD represents chisel disk; M represents mulch; SAP represents superabsorbent polymer; R represents deep ripping.

<sup>‡</sup> Means followed by the same letter are not significantly different at P < 0.05.

### Penetration Resistance

One of the main objectives of this study was to determine the response of PR, BD and VWC to soil amendments [SAP and M] and R (Table 12). Penetration resistance is a measure of soil strength and is affected by BD and VWC (Vaz et al., 2011). High soil strength that results from soil reconstruction decreases productivity (Dunker et al., 1995; Dunker and Barnhisel, 2000). Natural alleviation, such as with freeze-thaw cycles, is not efficient as it can take upwards of 30 or more years (Spoor, 2006; Batey, 2009; Bohrer, 2017). Given that land is required to be returned to pre-mine productivity, compaction alleviation is necessary. For all depth intervals, R significantly reduced PR relative to CD. At the 0-15 cm depth interval, PR was reduced from 1.76 MPa to 1.37 MPa for CD + SAP. Similarly, PR was reduced to 1.31 MPa for CD + SAP + R. However, for the 35-50 cm and 50-80 cm depth interval the CD + SAP was not significantly different from CD. The function of SAP may be impaired below 35 cm as absorbency can be restricted from the weight of the overlying soil. For the top three depth intervals SAP application significantly decreased PR relative to CD, however, when M + SAP was applied there was no change relative to the CD, indicating that there is some unknown interaction between SAP and M. Furthermore, for PR at all depth intervals, M without R was non-significant. These results reflect the same effects observed in the crop yields.

Penetration resistance is typically reduced with increased soil water content, however, at the 50-80 cm depth, which had the highest average VWC (0.25 cm<sup>3</sup> cm<sup>-3</sup>) across all treatments, also had the highest



average PR (5.44 MPa). This soil may be inherently stronger at the depth as even the higher VWC did not result in a lower PR. The relationship between BD and VWC is important to examine because this will determine a soils water filled pore space (WFPS). With the amount of WFPS increasing, so does the potential for freezing and thawing to break up compaction. Given the compacted nature of this soil, notably below 25 cm with the absence of the R treatment, the stress produced by freezing water expansion may not be able to shear the high strength soil. The weight of overlying soil could also be a factor in the ability of freezing water expansion to shear soil. Within other depth intervals there does not appear to be a correlation between VWC and PR. For example, at the 15-25 cm depth interval, seven VWC values are statistically similar, but PR are statistically different and range from 3.15 to 4.81 MPa. Therefore, the effects of SAP and R on PR appear to be an inherent effect on soil strength after one winter that exceeds any effects that soil moisture conditions alone would have on softening the soil layers.

Table 12. Mean values for bulk density (BD), volumetric water content (VWC) and penetration resistance (PR).

Treatment	BD	VWC	PR
Depth 0-15 cm	g cm <sup>-3</sup>	cm <sup>3</sup> cm <sup>-3</sup>	MPa
CD†	1.51A‡	0.12B	1.76A
CD + R	1.42B	0.15A	1.12C
CD + SAP	1.45C	0.12B	1.37B
CD + M + R	1.44B	0.10B	1.24B
CD + M + SAP + R	1.45C	0.14A	1.25B
CD + SAP + R	1.45C	0.14A	1.31B
CD + M + SAP	1.50A	0.12B	1.56A
CD + M	1.46C	0.11B	1.54A
Depth 15-25 cm			
CD	1.46A	0.11A	4.22A
CD + R	1.34B	0.12A	2.73B
CD + SAP	1.51C	0.11A	3.72C
CD + M + R	1.54C	0.08B	3.20B
CD + M + SAP + R	1.44A	0.11A	3.15B
CD + SAP + R	1.47A	0.11A	3.08B
CD + M + SAP	1.41A	0.11A	4.81A
CD + M	1.33B	0.11A	4.15A
Depth 25-35 cm			
CD	1.41A	0.11A	4.44B
CD + R	1.56C	0.12A	3.87A
CD + SAP	1.51B	0.15C	3.35A
CD + M + R	1.53B	0.08B	3.73A
CD + M + SAP + R	1.53B	0.09B	3.67A
CD + SAP + R	1.47A	0.10A	3.53A
CD + M + SAP	1.50B	0.11A	5.37B
CD + M	1.41A	0.10A	4.44B
Depth 35-50 cm			
CD	1.75A	0.10C	5.68B
CD + R	1.50B	0.14A	4.05A
CD + SAP	1.60B	0.15A	4.50A
CD + M + R	1.50B	0.14A	4.25A
CD + M + SAP + R	1.49C	0.24B	4.05A
CD + SAP + R	1.47C	0.14A	4.05A
CD + M + SAP	1.67A	0.13A	4.36C
CD + M	1.62A	0.13A	5.32B

Table 12. Mean values for bulk density (BD), volumetric water content (VWC) and penetration resistance (PR) (continued).

Treatment	BD	VWC	PR
Depth 50-80 cm	g cm <sup>-3</sup>	cm <sup>3</sup> cm <sup>-3</sup>	MPa
CD	1.82A	0.21B	5.92A
CD + R	1.58C	0.29C	5.04B
CD + SAP	1.75B	0.24A	5.61A
CD + M + R	1.78A	0.24A	5.16B
CD + M + SAP + R	1.67C	0.28C	5.12B
CD + SAP + R	1.72B	0.25A	5.08B
CD + M + SAP	1.83A	0.23B	5.78A
CD + M	1.74B	0.25A	5.88A

† CD represents chisel disk; M represents mulch; SAP represents superabsorbent polymer; R represents deep ripping.

‡ Means followed by the same letter within the same depth interval and column are not significantly different at  $P < 0.05$ .

### Conclusions

The main objectives of this research were to determine the effects of R and soil amendments of a superabsorbent polymer and M on soil PR and crop yield. Deep ripping alone and in combination significantly reduced PR, which correlated to higher yields. While these yields were low relative to the county average, after one growing season the application of 0.04% SAP proved to decrease PR and increase yield, in a manner that it was similar to R. Given, that R has a high work requirement (time and operation cost), the use of SAPs may be an alternative after considering product availability and costs. To more completely determine the use of SAPs as a reclamation tool, compaction and yield should be investigated for a longer period of time. The method and rate of SAP application should be further investigated to better understand field performance and when it may be effective to apply. Similarly, R has proven effective after one growing season, the longevity maybe variable depending on when resettling of the soil occurs.

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## GENERAL CONCLUSIONS

The general goal of this research was to determine the effect of SAP application on soil physical properties, as well as, examine its potential to alleviate compaction and improve yield after surface coal mining. Superabsorbent polymers retain water within soil for extended periods of time due to decreased evaporative water loss. Increased soil LL with SAP application will make soil more ridged upon freezing. With SAPs increasing and prolonging soil water retention, conditions that are conducive to compaction alleviation by freezing are promoted. After undergoing six freeze-thaw cycles compaction was significantly reduced and hydraulic conductivity decreased. In the field, the greatest amount of compaction alleviation occurred with deep ripping alone or in combination with chisel disc, SAP, or M. Notably, SAP application alone decreased compaction to the degree that it was similar to deep ripping treatments. Highest yields were seen on plots that received deep ripping in any combination or SAP application. After one growing season, deep ripping and SAP application were successful at reducing compaction, which translated to improved yield. While yields were improved, the highest yield obtained was only 43% of the county average, indicating the need to lengthen the field study to assess if yields continue to increase over time.

## APPENDIX. FIELD APPLICATION RATE CALCULATION

Where:

BD = Standard bulk density (1.32 g cm<sup>3</sup><sup>-1</sup>)

PD = Standard particle density (2.66 g cm<sup>3</sup><sup>-1</sup>)

WFPS = Water filled pore space (0.65)

Ac = 4046.86 m<sup>3</sup>

Depth = 1 m

F = to increase chance of effectiveness doubled amount (2)

1. % solid space =  $\left(\frac{BD}{PD}\right) \times 100$   
= 49.624 %
2. % pore space = 100 – (% solid space)  
= 50.376 %
3. Volume of pore space =  $Ac \times depth \times \left(\frac{\% \text{ pore space}}{100}\right)$   
= 2038.64375 m<sup>3</sup>
4. Volume of WFPS = Volume of pore space × WFPS  
= 1325.11844 m<sup>3</sup>
5. Amount of SAP =  $\left(\frac{x(\text{kg})}{\text{Volume of WFPS}}\right) = .004$   
= 5.3005 kg SAP
6. Field rate = Amount of SAP × F  
= 10.6009 kg SAP
7. Field application rate conversion = Field rate ×  $\frac{1000g}{1kg} \times \frac{.00220462 \text{ lbs}}{1g}$   
= 23 lbs ac<sup>-1</sup> = 25.8 kg ha<sup>-1</sup>