UNIVERSITY OF ALBERTA

THE GROWTH RESPONSE OF ASSINIBOINE POPLAR AND HARRINGTON BARLEY TO WOOD ASH ADDTIONS

BY

ALLAN BERTSCHI

A thesis submitted to the Faculty of Graduate Studies and Research in partial fulfillment of the requirements for the degree of Master of Science.

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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies and Research for acceptance, a thesis entitled THE GROWTH RESPONSE OF ASSINIBOINE POPLAR AND HARRINGTON BARLEY TO WOOD ASH ADDITIONS submitted by ALLAN BERTSCHI in partial fulfillment of the requirements for the degree of MASTER OF SCIENCE.

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ABSTRACT

Fly ash was added to two Alberta forest soils at ten different treatment levels in order to evaluate the growth response of hybrid poplar (*P. deltiodies* var. Assiniboine) and barley (*Hordeum vulgare* var. Harrington) and monitor soil environmental factors that may contribute to bioaccumulation of heavy metals in barley. Soil was sampled throughout the 90-days from the E.DyB soil and analyzed for SAR, EC, $pH_{(CaCl2)}$ and B, Cd and Zn. Both the soil types showed increased growth of barley of 49%. There was no significant increase in poplar growth in the O.GL soil at any treatment level and E.DyB showed increases of 45% in height and 31% increase in diameter.

Soil analysis revealed SAR and Cd levels were not affected by ash applications. Soil parameters EC, $pH_{(CaCl2)}$, B and Zn increased with each incremental treatment. All treatments increased in EC. The 20 Mg/ha treatment increased soil pH and stabilized at near neutral levels. Soil concentrations of B and Zn increased in all treatments and raised the fertility status of the soil.

To my family

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CHAPTER ONE

FLY ASH AS A SOIL AMENDMENT
LITERATURE REVIEW

1.1 Introduction

Land disposal of waste material from industrial and municipal sources is not a new concept. Significant literature on the subject is available. The University of Pennsylvania applied municipal sewage to a small basin (Sopper, 1986) over a 23+ year period. Results showed recharge to the local groundwater system and no significant changes in the amounts of trace elements in either the soil or the vegetation. Van Ham and Kimmins (1993) conducted a study on application of biosolids on forest soils in the lower Vancouver mainland of B.C. Early results did not identify any unacceptable environmental impacts. Land application of pulp mill waste is currently being investigated as an alternative method of disposing waste material. Nutter (1993) in a review paper describes early results that range from increased growth in trees with no increase in heavy metals to poor tree growth, mortality and soil toxicity. Environmental concerns about land disposal has led governments to adopt guidelines setting limits on amounts of heavy metals and other toxic substances that can be safely applied to land (Alberta Environment, 1982, 1984; Environment Canada, 1984).

1.2 Background

Wood ash is a residual material produced when bark is burned for the production of electricity. Pulp mills have become self sufficient in their electrical needs by burning bark and other hog fuels. Traditionally, landfilling was the only means of handling this solid waste stream. However, more stringent regulations on landfill design have increased the cost of landfilling high volume waste streams. Discovering alternative uses for various waste streams can reduce the amount of material that is currently landfilled and may improve mill efficiency.

Land spreading of ash on agricultural and silvicultural land may be one way of diverting large volumes of waste from landfills and increasing plant growth. Greenhouse and field scale studies in the United States, Finland, Sweden and Scandinavian countries have shown increased growth for both agricultural and forest crops (Lerner and Utzinger, 1986; Campbell, 1990; Ferm et al., 1992; Eriksson, 1994 and Meyers and Kopecky, 1998). Silfverberg and Hotanen (1989) report a 32-fold increase in productivity of Scots pine (*Pinus sylvestris* L.) over a 41-year duration. A 20% increase in above ground biomass of *Populus* Spp, over a 2-month period was reported by Etiegni et al., (1991). Agricultural crops, in greenhouse and field studies, show 20 - 60% increases in above ground biomass with wood ash additions to soil (Ohno, 1992). Abboud and Macyk (1996) combined fly ash and sludge for an Alberta pulp mill and also reported a 60% increase in above ground tissue of barley grown on a reclaimed oil lease.

Increases in agriculture and silvicultural crop yields from ash additions are attributed to the liming potential of the ash and the addition of macro and micronutrients. Several authors have characterized ash for its liming potential, macro and micronutrients and other elements (Lerner and Utzinger, 1986; Naylor and Schmidt, 1989; Campbell, 1990; Clapham and Zibilske, 1992; Muse and Mitchell, 1993 and Someshwar, 1996).

Ash elemental composition can vary considerably between tree species and between the wood and bark of the same species. Environmental factors such as soil type and climate as well as industrial operation such as combustion temperature and ash collection systems will influence the elemental composition (Campbell, 1990). Someshwar (1996) compared ash from 10 species of bark and 9 species of wood and found bark ash to have considerably higher levels of aluminum (Al) and silicates (Si) than wood ash. This is likely due to imbedding of soil particles during logging operations. Unpublished and non-replicated data from Alberta Pacific found considerably higher levels of cadmium (Cd) and zinc (Zn) in bark relative to that of wood tissue within the same species.

In the United States, it is estimated that 4.6 million tonnes of boiler ash are generated annually form the production of energy in the forest products industry, of which 3.4 million tonnes are generated by the pulp and paper sector (Miner and Unwin, 1992 and Someswhar, 1996). A 1979 survey of U.S. pulp and paper mills indicated that 86% of the sludge generated was landfilled, 11% incinerated, and 2% land spread (NCASI, 1983). Miner and Unwin (1992) report that nationally >80% of the ash is land filled with only 5% being land spread. In the Lake states it is estimated that the amount of ash being land spread on agriculture and silvicultural land is approximately 33% of that produced (McGinnis et al., 1995).

Solid waste generated by Alberta Pacific Forest Industries (Al-Pac) located at Boyle AB, is estimated to be 48,000 m^3 annually of which fly ash accounts for half by volume or approximately 18,000 tonnes/yr. Current disposal method of this waste is to landfill, which is standard operating procedure for Alberta pulp mills. Al-Pac is interested in diverting some of their waste streams into a land-spread application program in hopes of enhancing tree and cereal crop growth while expanding the life of their landfill.

1.3 Guidelines and Standards for Land Spreading

Guidelines for application rates for land spreading pulp mill solid waste currently do not exist. Until such are available, Alberta Environment Protection will use and enforce application rates of heavy metals developed for other industries. Industrial application spreading rates are regulated by CCME, Alberta Tier 1 Criteria for Contaminated Assessment and Remediation and Alberta Environment Biosolid Regulations. Alberta Environment Protection (AEP) recently proposed to adopt the ruling set by the Ontario Provincial Government to limit soil concentrations of TCDD/TCDF (tetrachlorodibenzo-p-dioxin/tetrachlorodibenzofuran) to no more than 10 parts per trillion (10 pg/g). Total Toxic Equivalence (TEQ) of dioxin and furans were below detection limits of 0.005 ppm by Cantest and 1.16 pg/g Alberta Research Council (Abboud, 1994). Therefore, Alberta-Pacific's dioxin and furans are not a limiting factor in a land-spread program.

1.4 Limitations to Land Spreading Alberta Pacific's Fly Ash

Parameters of greatest concern in using Al-Pac's fly ash in a land-spread program are elements boron B, Cd and Zn as well as its high alkalinity and electrical conductivity. Alberta Environment has requested that soil parameters: SAR, EC, pH and elemental loading of B, Cd and Zn be monitored and analyzed statistically to aid in determining the potential of using Al-Pac's fly ash for land-spreading. Alberta Environment also requested plant tissue analysis be monitored for bioaccumulation of the above-mentioned elements for the determination of safe agronomic loading rates.

1.5 Justification for Research

The growth response of agricultural and silvicultural plants, heavy metal loading of soil and plant uptake of metals following wood ash additions has been the focus of many studies in the United States and Scandinavian countries. Even though there is much published literature, Alberta Environment regulators are reluctant to approve this by-product as a soil amendment because it lacks of organic matter and the lack of literature on application of wood ash on Canadian soils. The lack of organic mater can increase the availability of nutrients and toxins and if applied a to high of a rate could result in soil phyto-toxicity

1.6 Objectives

The objective of this study was to determine maximum loading rates that are environmentally safe and agronomic loading rates by controlled laboratory testing and analysis for the application of wood ash to enhance the growth rate of hybrid polar and cereal crops. The study was based on testing the capacity of the soil to receive and retain inorganic elements following the incorporation of wood ash at various loading rates.

The four main objectives of this study were:

1. To determine an application rate that will enhance the growth rate of agricultural crops on two Alberta forest soils.

2. To determine an application rate that will enhance the growth rate of hybrid poplar on two Alberta forest soils.

3. To determine an environmentally safe application rate that will not significantly increase elements in the soil that is regulated by the province of Alberta contamination criteria.

4. Determine the uptake of elements of concern in agricultural plant tissue.

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CHAPTER TWO

HARRINGTON BARLEY

BIOMASS RESPONSE TO ASH ADDITION

2.1 Introduction

Wood ash is a residual material produced when bark is burned for the production of electricity. Pulp mills are self sufficient in their electrical needs by burning bark and other hog fuels. Traditionally, landfilling is the primary means of handling this solid waste stream. However, more stringent regulations on landfill design have increased the cost of disposing high volume waste streams. Discovering alternative uses for various waste streams will reduce the amount of material that is currently landfilled while improving mill efficiency.

Land spreading of wood ash to increase the growth rate of agricultural crops has been investigated in many countries in both greenhouse and field trials. Forages show positive growth responses to ash additions at loading rates between 0.5-3% Weight of ash/weight of soil (w/w) or 10-50 Mg/ha equivalence. In a greenhouse study, Naylor and Schmidt (1989) report increases in alfalfa (*Medicago sativa*) biomass by as much as 144%. Meyers and Kopecky (1998) in a similar study report a 30% increase in alfalfa biomass. Alfalfa grown in a field study had a seasonal total increase of 41% over the control (Meyers and Kopecky, 1998). Fescue (*Paspalum dilatatum* Poir-Festuca *arundinacea* Schreb) yields increased by 61% (Muse and Mithchell, 1995). Cereal crops like wheat (*Triticum aestibum*) show increases as high as 69% (Etiegni et al., 1991) and oats (*Avena* *sativa* L., var 501) as high as 45 % (Krejsl and Scanlon, 1996). Meyers and Kopecky (1998) reported a 25% increase in above ground biomass of barley. Vegetable crops, in particular beans (*Phaseolus Vulgaree* L., blue pole) have shown increases in biomass as much as 64% (Krejsl and Scanlon, 1996). Biomass increases are attributed to the increase in soil pH and macro and micronutrients supplied by wood ash (Lerner and Utzinger, 1986; Erich, 1991; Meyers and Kopecky, 1998). Negative growth affects are seen at higher loading rates. For example, wheat is reported to stop growing at rates greater than 320 Mg of ash/ha (Etiegni et al., 1991).

Despite the extensive body of literature on using ash to enhance the growth of agricultural crops there is little scientific work reported on Canadian soils. One of the earliest references is Hopkins (1910) who reports of field trials with beneficial growth to wheat, oats and barley dating from 1891-1904. He further implies that ash or ash products may have been regulated during this time period. Abboud and Macyk (1994) combined wood ash and biosolids from an Alberta pulp mill and reported beneficial growth responses in both cereal and forage crops.

2.2 Objective

The objective of this greenhouse study was to determine if wood ash alone has an affect on the growth rate of cereal crops in two different Alberta forest soils. Ten loading rates per soil were tested. The hypothesis tested was:

HO: Wood ash addition to soil has no effect on the growth of Harrington Barley (*Hordeum Vulgare*).

2.3 Materials and Methods

2.3.1 Soils

Eluviated Dystric Brunisol (E.DyB) and Orthic Gray Luvisol (O.G.L) soils were collected from two fields near the Alberta Pacific pulp mill located 35 km northwest of Grassland, Alberta. E.DyB soils were collected from the Ap horizon of a Nestow soil series from quarter section SE 5-69-19 W 4th. O.GL soils were collected from the Ap horizon of a Tolman soil series from quarter section SE 36-68-20W 4th. Both soil types were air dried at 22^{o}C and ground to pass through a 2 mm sieve.

Soils of the Brunisolic order account for approximately 10% of the landscape in the County of Grassland. More than half of these soils are of the subgroup E.DyB. This subgroup is a low fertility soil that is mostly comprised of 70% Nestow soil series. The Nestow series is the dominant soil series in this particular field. These soils are well to rapidly drained. A thin Ae horizon has developed over a coarse sandy parent material. In its natural state the vegetation would be jack pine, which currently borders this field. Nestow soils have a low percent base saturation and are acidic with a pH as low as 4.5. Soil for this experiment was taken from an area where the agricultural capability is low, with a class rating of 6 (Tawatinaw Map Sheet, 1972). Physical and chemical characteristics of the soil taken from SE 5-69-19 W4th are presented in Appendix 1.

Luvisolic soils account for approximately 40% of the landscape of Grassland County. More than 30% of these soils are of the subgroup O.GL. This subgroup is a moderate fertility soil that is mostly comprised of 60% Tolman soil series. Tolman series are the dominant soil series in this particular field. These soils are well drained with a thick Ae horizon developed over glacial till. In its natural state the vegetation would be hardwood dominated by aspen and white birch. Tolman soils have a moderate base saturation and are moderate to slightly acidic with a pH of 6.2. Soil for this experiment was collected from an area where the agricultural capability is a class 3. If these soils are used for intensive agriculture they will require high maintenance inputs, especially nitrogen and phosphorus (Tawatinaw Map Sheet, 1972). Physical and chemical characteristics of the soil taken from SE 5-69-19 W4th are also presented in Appendix 1.

2.3.2 Ash

Bark and wood waste derived fly ash was collected from the power boiler at Alberta-Pacific Forest Industries Inc. near Grassland Alberta. Bark and wood waste is comprised of 80% hardwood and 20% softwood. Ash was air dried at 22° C and ground to pass through a 2 mm sieve. Fly ash from Alberta-Pacific is routinely collected and sent to Enviro-test Laboratories® in Edmonton, Alberta. Ash was characterized for total metals nutrients, available metal and nutrients and physical parameters and is presented in Appendix 1.

2.3.3 Design

Dry ash and soil were premixed in a cement mixer for 5 minutes per loading rate to ensure a thorough blend. Ten loading rates consisting of: control, 10, 15, 20, 40, 60, 80, 100, 120 and 200 Mg/ha equivalence, were potted up in 6-inch standard pots. Each loading rate was replicated 8 times for both soil types. Four pots were randomly selected and planted with Harrington barley (*Hordeum Vulgare*). Five seeds were planted and thinned back to three plants after germination.

Pots were randomly placed in a greenhouse with a diurnal cycle of 16-day light hours and 8 dark. Room temperature was maintained between 21-24°C. Pots were watered every other day to minimize the risk of leaching. Deionized water (18.0 megohm cm^{-1}) was used to eliminate additional nutrient and trace elements input.

Barley was grown for 90 days and harvested on an individual pot basis. Above ground biomass was collected. Weights were recorded after oven drying at 55°C for 72 hours.

2.3.4 Statistical Analysis

Statistical analysis of the data collected for heavy metal uptake was determined using the Fisher's Protected Least Significant Difference (LSD). LSD can only be completed if the Probability >F (Prop>F) calculated from the ANOVA is ≤ 0.05.

2.4 Results

Barley grown in E.DyB soil showed ($P > 0.001$) a positive growth response to ash additions (Table 2.1). Positive effect of the ash was seen in all loading rates except the 200 Mg/ha loading rate. A combined average across all loading rates showed a 37% increase in biomass. The greatest increase in above ground biomass was in the 100 Mg/ha treatments, in this loading rate there was a 49.6% increase in growth. The 200 Mg/ha loading rate was the only treatment that did not show a significant growth increase over the control even though, it still showed a positive increase of 17.5% over the control.

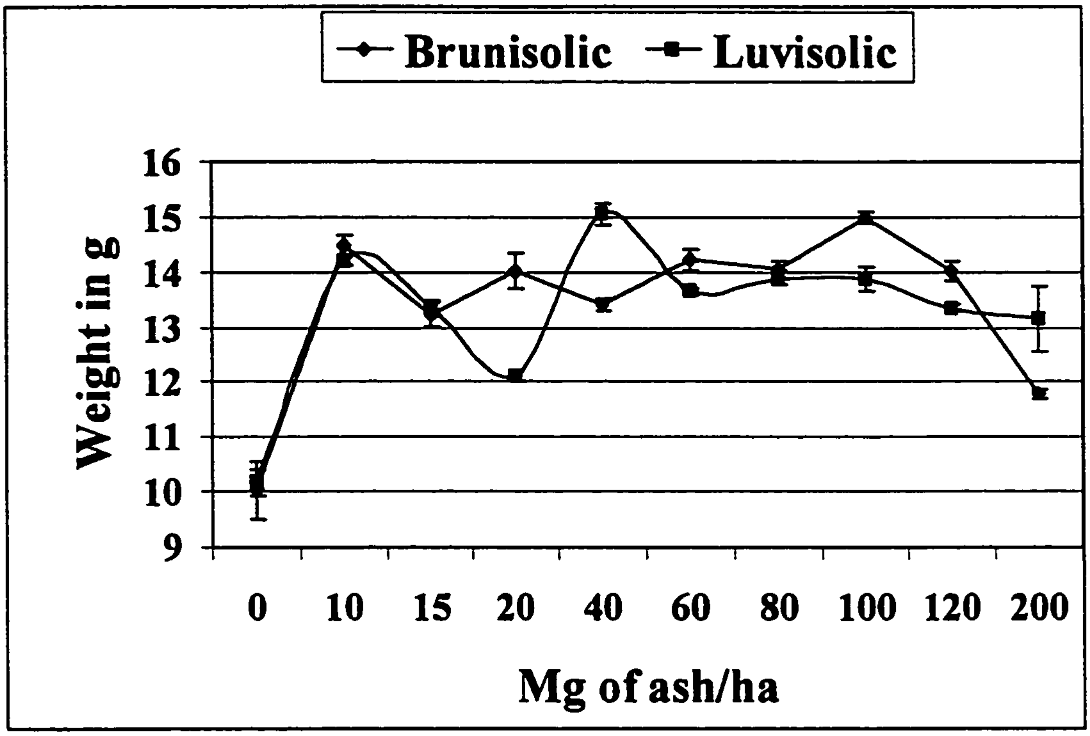
Results from the barley grown in ash treated Orthic Gray Luvisolic soil showed that there was a (P > 0.05) positive growth response (Table 2.i). The range of increase was from 19-48% when compared to the control with an average increase of 34%. The growth response was very consistent across the trial with the exception of 2 peaks in the 10 Mg/ha and the 40 Mg/ha treatments. The lowest response was in the 20 Mg/ha treatments, which still shows a 19% increase in biomass.

Table 2.1. Above ground biomass of Harrington Barley (*Hordeum Vulgare*) grown in Eluviated Dystric Brunisolic soil and Orthic Gray Luvisolic soil at various loading rates of wood ash.

Treatment Mg/ha	Brunisolic Soil Mean g/pot ♦ (SE) ♦ LSD	Luvisolic Soil Mean g/pot ♦ (SE) ♦LSD
0	10.03 (0.23) ♥c	10.17 (0.52) ♥d
10	14.48 (0.08) ab	14.20 (0.20) ab
15	13.24 (0.11) b	13.37 (0.22) bc
20	14.03 (0.10) ab	12.12 (0.31) c
40	13.41 (0.20) b	15.06 (0.11) a
60	14.23 (0.05) ab	13.65 (0.19) bc
80	14.05 (0.14) ab	13.90 (0.15) ab
100	15.00 (0.20) a	13.88 (0.12) ab
120	14.03 (0.07) ab	13.36 (0.17) bc
200	11.79 (0.58) c	13.15 (0.08) bc

♦SE = Standard Error based on four replicates in each treatment.
♦LSD = Least Significant Difference ($p<0.05$), indicates that at least two treatments are statistically significantly different at the $p = 0.05$ level.
♥a, b, c, d = treatments means with the same letter are not statistically significant from each other. Soil types were statistically analyzed separately.

Figure 2.1 Above ground biomass of Harrington Barley (*Hordeum Vulgare*) grown in Eluviated Dystric Brunisolic soil and Orthic Gray Luvisolic soil at various loading rates of wood ash.



2.5 Discussion

Wood ash has been long recognized as a source of potassium K (1-13%) for plant growth (Hopkins, 1910). More recent analysis of wood ash show that there are significant levels of Ca (13-32%), P (0.3-1.25%), Mg (1-3%), as well as appreciable concentrations of B, Cu, Fe, Mn, S, and Zn (Naylor and Schmidt, 1986). The high nutrient concentration of these ashes has contributed to significant agronomic benefit in terms of growth and yield of various agricultural crops. Fly ash from Alberta-Pacific is very comparable to nutrient characteristics of other wood ash found in the literature (Appendix 1). Both Eluviated Dystric Brunisol (E.DyB) and Orthic Gray Luvisol (O.GL) soils are high maintenance soils in terms of the nutrient composition; therefore, the addition of wood ash should benefit both soils because of nutrient composition and acid neutralizing capacity (ANC).

Harrington barley grown in E.DyB soils treated with fly ash resulted in an ($p<0.001$) increase in biomass. Harrington barley had a 38% increase in above ground biomass compared to the control. With no other nutrient additions to the soil we can assume that the ash provided the barley with the necessary nutrients for the increased growth. Similar responses were noted by Meyers and Kopecky (1998) and Krejsl and Scanlon (1996) and Etiegni et al. (1991) where they reported an average increase in growth of barley by 25%, oats at 45% and wheat at 69% at similar loading rates. These authors attribute the growth response to increased soil pH and increased nutrient from the ash.

Harrington barley grown in O.GL soils also resulted in increased biomass very similar to the E.DyB soil Figure 2.1. All loading rates showed positive responses to the ash addition. Overall the treated soil had a 34% increase over the control and the positive response even evident in the lowest loading rate of 10 Mg/ha treatment. Growth was very consistent across the trials with little variation between treatments.

Statistical analysis of the growth response showed that only the highest loading rate of 200 Mg/ha, in the E.DyB soil, did not have significant increase in growth compared to the control. This loading rate was included in the experiment for the purpose of finding the upper loading limit. Etiegni et al. (1991) found that growth rate of wheat ceased at 340 Mg/ha of wood ash and Krejsl and Scanlon (1996) reported reduced growth of oats in 50 Mg/ha treatments. All other experiments in the literature show positive linear growth rates with increased loading rates up to a maximum treatment of 50 Mg/ha. Mathematically, loading rates of 200 Mg/ha would push total zinc (Zn) concentrations of soil well over Alberta Tier One Guidelines of 120 ppm, therefore, loading rates higher than this were not included in this experiment.

There were two peaks in growth response in E.DyB soil, one in the 10 Mg/ha loading rate and in the 100 Mg/ha rate. The increased growth in the 10 Mg/ha loading rate was 44% while the 100 Mg/ha loading rate had a 49% increase. O.GL soil also had two peaks in growth response. The first peak was in the 10 Mg/ha loading rate of 41% increase in biomass. The second peak was in the 40 Mg/ha loading rate of 49.5% increase in biomass. In order to sustain intense agricultural practices both soil types will require additional nutrient inputs in order to remain productive (Tawatinaw Map Sheet, 1972). Increasing the percent Base Saturation (%BS), Cation Exchange Capacity (CEC) and soil pH will increase soil fertility with respect to agricultural crops. Kahl et al. (1996) reported that ash additions as low as 6 Mg/ha were sufficient enough to favorably alter the %BS, CEC and soil pH resulting in improved soil fertility. Therefore, the soil fertility of E.DyB and O.GL soils was likely improved by the addition of ash resulting in the increased growth.

Further research needs to be done at a lower range in order assess the optimal loading rate to maximize agronomic productivity and preserve environmental sustainability.

2.6 Conclusion

The increased growth of barley in fly ash treated soils seen in this experiment is consistent with other research in the literature involving cereal crops. Fly ash additions showed a positive increase in above ground tissue of Harrington barley in both the E.DyB and the O.GL soils. Controls for both soil types were similar in average above ground biomass. In both soils there was a positive response to the fly ash addition. Barley showed similar growth response in both soils, however, biomass peaks at a lower loading rate in the E.DyB soil than in the O.GL soil.

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CHAPTER THREE

ASSINIBOINE POPLAR

GROWTH RESPONSE TO ASH ADDITION

3.1 Introduction

Wood ash is a residual material that is produced when wood and bark is burned for the production of electricity. Pulp mills are self sufficient in their electrical needs by burning bark and other hog fuels. Traditionally, landfilling is the primary means of handling this solid waste stream. However, more stringent regulations on landfill design have increased the cost of disposing of high volume waste streams. Discovering alternative uses for various waste streams will reduce the amount of material that is currently landfilled while improving mill efficiency.

Land spreading of wood ash to increase the growth rate of silvicultural crops has been investigated in many countries in both greenhouse and field scale studies. Growth responses are variable depending on trees species, loading rates, soil type and duration of study. Responses range from phyto-toxicity, to no benefit to significant growth increases. No growth response was observed in a 9-year-old stand of Western Red Cedar (*Thuja plicata* Donn ex D. Don) with the application of 5 Mg/ha of ash on a Ferro-Humic Podzol (McDonald et al., 1993). No effect was seen in two separate 2 year studies in which surface additions of ash were added to a 22 year old stand of Norway Spruce (*Picea abies* (L.) Karst) in Sweden (Clarholm, 1994) or in a 100 year old stand of Scots Pine (*Pinus sylvestris* L.) in Finland growing in Histosols (Fritze et al., 1989). Surface additions of ash are reported to be toxic to Jack pine (*Pinus banksiana*) establishment. Thomas and Wein (1994) reported that 2-3 cm of ash deposited by wildfires inhibit germination and early survival of Jack Pine. However, Thomas and Wein noted that these effects were ameliorated after 2 years of high precipitation.

Other studies indicate beneficial growth responses of trees to wood ash additions. In a 3-year study, willow trees increased in growth by 65-75% with an application of 10 Mg/ha (Weber et al., 1985). In a greenhouse study, Etiegni et al. (1991) reported that Poplar trees planted in Inceptisols and Ultisols with 40 Mg of ash/ha equivalents increased growth by 15% in height and 9% in diameter over a 2-month period. In Finland, Histosol soils treated with 3 rates of ash between 0 - 16 Mg/ha showed a 25 - 32 fold increase in Scots pine over 41 years (Silfverberg and Hotanen, 1989). Ash additions also improved visible nutrient deficiencies of Scots pine growing in peatlands. Thirteen years after ash application, symptoms of nutrient disorders such as leader die back, short needles and mortality were substantially reduced (Ferm et al., 1992). Ferm also reports that improved soil nutrient conditions resulted in an increase in tree volume by 4.6 times that of the control over the same 13-year period.

With increased awareness of CO_2 emissions and potential offset credits assigned to tree plantations there is an increased trend around the world for afforestation of marginal agricultural fields. Intensive agricultural practices such as cultivation, summer fallow and the harvesting and removal of grain and straw can reduce essential soil micronutrients through leaching and transportation. Alberta Agriculture reports that barley will remove 10.63 kg/ha of micronutrients annually when both grain and straw are removed from the field (Alberta Agriculture, 1992). Replenishing micronutrients with wood ash prior to afforestation will enhance soil micronutrients and may result in increased growth potential depending on tree species and soil properties.

3.2 Objective

The objective of this study was to determine if wood ash has an effect on the growth rate of rooted poplar cuttings in two different Alberta forest soils. Ten loading rates per soil were tested. The hypothesis tested was:

Ho: Wood ash addition to soil has no effect on the growth rate of Assiniboine poplar (*P. deltoidies* var. Assiniboine).

3.3 Materials and Methods

3.3.1 Soils

Eluviated Dystric Brunisol (E.DyB) and Orthic Gray Luvisol (O.GL) soils were collected from two fields near the Alberta Pacific pulp mill located 35 km northwest of Grassland, Alberta. E.DyB soils were collected from the Ap horizon of a Nestow soil series from quarter section SE 5-69-19 W 4th. O.GL soils were collected from the Ap horizon of a Tolman soil series from quarter section SE 36-68-20W 4th. Both soil types were air dried at 22^{O}C and ground to pass through a 2 mm sieve.

Soils of the Burnisolic order account for approximately 10% of the landscape in the County of Grassland. More than half of these soils are of the subgroup E.DyB. This subgroup is a low fertility soil that is mostly comprised of 70% Nestow soil series. The Nestow series is the dominant soil series in this particular field. These soils are well to rapidly drained. A thin Ae horizon has develop over a coarse sandy parent material. In its natural state the vegetation would be jack pine, which currently borders this field.

Nestow soils have a low percent base saturation and are acidic with a pH as low as 4.5. Soil for this experiment was taken from an area where the agricultural capability is a class 6 (Tawatinaw, Map Sheet, 1972). Physical and chemical characteristics of soil taken from SE 5-69-19 W4th are presented in table form in Appendix 1.

Luvisolic soils account for approximately 40% of the landscape of Grassland County. More than 30% these soils are of the subgroup Orthic Gray Luvisol. This subgroup is a moderate fertility soil that is mostly comprised of 60% Tolman soil series. Tolman series is the dominant soil series in this particular field. These soils are well drained with a thick Ae horizon developed over glacial till. In its natural state the vegetation would have been hardwood dominated by aspen and white birch. Tolman soils have a moderate base saturation and are moderate to slightly acidic with a pH 6.2. Soil for this experiment was collected from an area where the agricultural capability is a class 3. If these soils are to be used for intense agriculture they will require high maintenance inputs especially high amounts of nitrogen and phosphorus (Tawatinaw, Map Sheet, 1972). Physical and chemical characteristics of soil taken from SE 5-69-19 W4th are presented in table from in Appendix 1.

3.3.2 Ash

Bark and wood waste derived fly ash was collected from the power boiler at Alberta-Pacific Forest Industries Inc. near Grassland Alberta. Bark and wood waste is comprised of 80% hardwood and 20% soft wood. Ash was air dried at 22° C and ground to pass through a 2 mm sieve. Fly ash from Alberta-Pacific is routinely collected and sent to Enviro-test Laboratories® in Edmonton, Alberta. Ash was characterized for total metals nutrients, available metal and nutrients and physical parameters and is presented in table form in Appendix 1.

3.3.3 Design

Dry ash and soil were premixed in a cement mixer for 5 minutes per loading rate to ensure a thorough blend. Ten loading rates consisting of: control, 10, 15, 20, 40, 60, 80, 100, 120 and 200 Mg/ha equivalence, were potted up in 6-inch standard pots. Each loading rate was replicated 8 times for both soil types. Four pots were randomly selected and planted with rooted cuttings of Assiniboine poplar (*P. deltoidies* var. Assiniboine).

Pots were randomly placed in a greenhouse with a diurnal cycle of 16-day light hours and 8 dark. Room temperature was maintained between 21-24 ^{o}C. Pots were watered every other day to reduce the risk of leaching. Deionized water (18.0 megohm cm^{--1}) was used to eliminate additional nutrient and trace elements input.

Poplar trees were grown for 70 days and data was collected on an individual pot basis. Root collar and total height were recorded. Height was measure by resting a meter sick on the soil surface and measuring to the bottom of the terminal bud. Using a caliper and measuring the tree at the soil surface measured diameter at root collar. Two diameter measurements were recorded at right angles to each other and averaged and then recorded.

3.3.4 Statistical Analysis

Statistical analysis of the data collected for heavy metal uptake was determined using the Fisher's Protected Least Significant Difference (LSD). LSD can only be completed if the Probability >F (Prop>F) calculated from the ANOVA is ≤ 0.05.

3.4 Results

Results from the caliper measurements indicated ($P < 0.05$) a benefit to Assiniboine poplar growth from the addition of ash to E.DyB soil (Table 3.1). Modest loading rates of ash showed significant benefits to root collar caliper growth while grater than 40 Mg/ha rates showed diminishing responses. Significant increases in root collar caliper were seen in 10, 15, 20 and 40 Mg/ha treatments. The greatest response in caliper was in the 15 Mg/ha treatments, which showed an increase of 31% over the control. Treatments 10, 20 and 40 Mg/ha showed increases of 28, 22 and 29%, respectively. Loading rates 60, 80, and 100 Mg/ha were insignificant even though growth was 2-12% greater than the control. The 120 Mg/ha treatments showed no response while the 200 Mg/ha loading rate showed a slight decrease in growth compared to the control.

Ash additions to O.GL soils had no significant effect on root collar caliper diameter (Table 3.1). Growth response was negatively impacted at higher loading rates relative to the control. Reduced growth was seen in all loading rates from 40 Mg/ha and greater with the exception of 80 Mg/ha. Ash did have a slight positive effect on growth in treatments 10, 15, 20 and 80 Mg/ha; these increases however, were insignificant. Positive growth response to these loading rates ranged from 1 - 13%.

Table 3.1. Root collar caliper of Assiniboine poplar (*P. deltoidies* var. Assiniboine) grown for 70 days in Eluviated Dystric Brunisolic soils and Orthic Gray Luvisols at various loading rates of wood ash.

Treatment Mg/ha	Brunisolic Soil Mean Diameter♦ (SE)♦ LSD (mm)	Luvisolic Soil Mean Diameter ♦ (SE) ♦LSD (mm)
0	3.5 (0.16) c	4.24 (0.44) ab
10	4.47 (0.18) a	4.79 (0.43) a
15	4.6 (0.18) a	4.80 (0.12) a
20	4.28 (0.21) ab	4.30 (0.31) ab
40	4.53 (0.22) a	3.73 (0.19) b
60	3.69 (0.43) bc	3.80 (0.09) b
80	3.93 (0.34) bc	4.38 (0.24) ab
100	3.58 (0.22) bc	3.68 (0.31) b
120	3.5 (0.19) c	3.80 (0.31) b
200	3.38 (0.24) c	3.68 (0.26) b

♦SE = Standard Error based on four replicates in each treatment.
♦LSD = Least Significant Difference ($p<0.05$), indicates that at least two treatments are statistically significant at the $p = 0.05$ level.
♥a, b, c = treatments means with the same letter are not statistically significant from each other. The two soils were analyzed separately.

Results from the height measurements also indicated ($P < 0.05$) a benefit from the addition of ash to E.DyB soil (Table 3.2). Assiniboine poplar showed a positive response to the additions of ash, even at the lowest loading rate of 10 Mg/ha. Significant increases in height were noted in loading rates 15 and 40 Mg/ha. The 15 Mg/ha treatment showed the greatest increase in height with a 45% increase over the control while the 40 Mg/ha treatment showed a 30% increase. Treatments 10, 20, 60 and 200 Mg/ha were insignificant, with growth ranging from 3 – 17%, while treatments 80, 100 and 120 Mg/ha showed reduced growth as compared to the control.

Ash additions to O.GL soil also had no significant effect on height growth of Assiniboine poplar (Table 3.2). Overall height was negatively impacted at higher loading rates relative to the control. Reduced growth was seen in all loading treatments greater than 20

Mg/ha. Ash additions to O.GL soil showed no benefit to height during the 70-day experiment. Ash did have a slight positive effect on growth in treatments 10 and 15 Mg/ha. Positive growth response in these loading rates was only 2%, which was not significant.

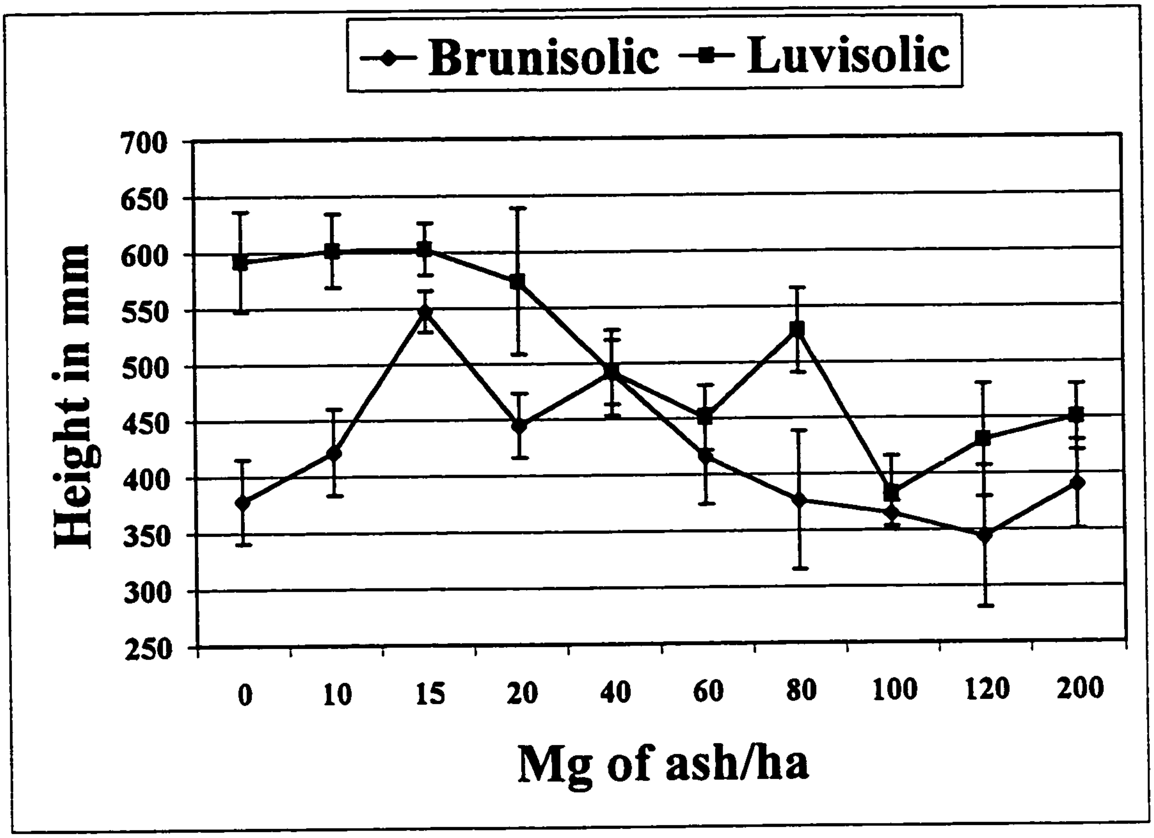
Table 3.2. Height of Assiniboine poplar (*P. deltoidies* var. Assiniboine) grown for 70 days in Eluviated Dystric Brunisol soil at various loading rates of wood ash.

Treatment Mg/ha	Brunisolic Soil Mean Height* (SE)* LSD (mm)	Luvisolic Soil Mean Height * (SE) *LSD (mm)
0	378 (37.48) cd	591.75 (44.86) a
10	421.67 (38.58) bcd	601.5 (32.47) a
15	547 (18.48) a	602.75 (23.13) a
20	444.75 (28.56) bc	573.5 (65.1) ab
40	491.25 (38.16) ab	492 (28.71) bcd
60	416 (42.24) bcd	450.67 (28.94) cde
80	376.75 (61.62) cd	528.25 (37.86) abc
100	364.75 (11.10) cd	383 (33.06) e
120	344 (62.92) d	429.75 (50.49) de
200	390 (39.47) cd	450 (29.31) cde

*SE = Standard Error based on four replicates in each treatment.
*LSD = Least Significant Difference ($p<0.05$), indicates that at least two treatments are statistically significant at the $p = 0.05$ level.
*a, b, c, d, e = treatments means with the same letter are not statistically significant from each other. The two soils were analyzed separately.

Interactions between the two soils indicate that both soil types showed an increased growth at the 15 Mg/ha treatments. There was only a slight increase in the O.GL however, at treatments greater than 15 Mg/ha tree height declined in both soil types Figure 3.1.

Figure 3.1 Is the height growth response of Assiniboine poplar grown in various loading rates of wood ash on Eluviated Dystric Brunisol and a Orthic Gray Luvisol.



3.5 Discussion

Assiniboine poplar planted in Eluviated Dystric Brunisol (E.DyB) soil had a significant beneficial growth in both height and root collar caliper during the 70-day greenhouse study. The greatest growth response of Assiniboine was in the 15 Mg/ha treatments. Similar results were found by (Weber et al., 1985; Etiegni et al., 1991; Silfverberg and Scanlon, 1989; Ferm et al., 1992). Etiegni et al. (1991) in a greenhouse experiment found that poplar increased in height by 15% and 9% in caliper in loading rates as high as 40 Mg/ha. In a field study, Ferm et al. (1992) reports a 4.6 fold increase in Scots pine volume over a 13 year period in loading rates of 20 Mg/ha. Both of these studies indicate that there is no one factor that is responsible for the increased growth, however, these studies attribute growth response to increased soil pH and available K. Etiegni et al. (1991) further attributed beneficial growth to increased soil levels of phosphorous (P) while Ferm et al. (1992) report increased soil levels of total boron (B).

E.DyB soils are classified as poor fertility soils. The texture of these soils is sandy; therefore, they will have a low cation exchange capacity compared to finer textured soils. Analyses of E.DyB soil collected from SE 5 indicated a deficiency in micronutrients such as copper (Cu) and B and have a low pH. Correction of these parameters would likely improve fertility.

Ohno (1990) studied the increased availability of nutrients in soil following wood ash application and found that additions of ash increased the levels of P, potassium (K), manganese (Mg), calcium (Ca) and sodium (Na). Kahl et al. (1996) studied ash additions to a sandy acidic forest Spodosol and found that ash additions of 6, 13 and 20 Mg/ha increased soil pH, exchangeable Ca, K, Mg, Cation Exchange Capacity (CEC) and percent base saturation in both the O and B horizons. Ash is known to be an excellent micronutrient source and liming agent (Naylor and Schmidt, 1986; Lerner and Utzinger, 1986; Naylor and Schmidt, 1989; Ohno, 1990; Campbell, 1990; Erich, 1991; Muse and Mitchell, 1995, Krejsl and Scanlon, 1996). Ash derived from Alberta-Pacific Forest Industries was very similar in physical and chemical characteristics to the above studied ashes. Alberta-Pacific's ash was also high in total zinc (Zn) and available B. Addition of ash increased soil fertility and increased growth of Assiniboine poplar planted in E.DyB soil.

Addition of ash to Orthic Gray Luvisol (O.GL) had little effect on Assiniboine poplar growth. Height response was less than 2%, however, the root collar caliper had a 13%

increase. Despite the appearance of a total volume gain there was no significant difference in woody biomass over the course of a 70-day study. Soil for this greenhouse experiment was taken from an area where the agricultural capability was a class 3, which is rated as fairly good to good arable. If these soils are used for intensive agriculture they will require high nutrient maintenance (Tawatinaw Map Sheet, 1972). This field was in a hay crop for 15 years prior to this experiment, therefore, agricultural practices likely did not deplete soil nutrients. Because of higher percent base saturation and CEC of the O.GL compared to E.DyB, the nutrients provided by the ash may not have benefited the soil fertility enough to stimulate increased growth of Assiniboine poplar. O.GL soils are typically only slightly acidic with a pH of 6.2, which is in the optimum pH range of (6-6.7) for *Populas spp.* (Peterson and Peterson, 1992). For these reason there was little benefit to O.GL soil from ash application.

Further field research is required to accurately determine if wood ash additions are beneficial to afforestation programs. Large field research programs could encompass a wider variety of soil polygons, micro-sites and tree species. Wood ash additions to O.GL soils in this experiment showed insignificant changes to growth within 70-days. A multi year study may result in compounded cumulative growth response, which could result in, significantly increased growth.

3.6 Conclusion

Additions of ash had varying degrees of success in enhancing the growth of Assiniboine poplar in 2 Alberta soil types. Assiniboine poplar planted in the E.DyB soil treated with ash resulted in significantly greater growth response in both height and root collar caliper. During the 70-day greenhouse study, Assiniboine poplar increased in height by 31% and in root collar caliper by 9% in E.DyB soil but showed no significant increase the same tree species planted in O.GL soil. Long-term studies have indicated that applications of wood ash will increase soil pH and plant available nutrient levels in agricultural and forest soils.

Successful afforestation of agricultural soils may be improved by wood ash, however, further field research in needed to determine loading rates and soil types that will best enhance growth. O.GL soil provided little benefit to the Assiniboine poplar with respect to significant increased height, however, there was a 13% increase in diameter which compounded over multiple years could ultimately result in shorter rotation time or increased yield over the same time period. Loading rates of 20 Mg/ha and less should be field tested over longer periods of time in order to adequately determine the benefits of wood ash additions to soil.

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CHAPTER FOUR

SOIL RESPONSE TO ASH ADDITION

4.1 Introduction

Wood ash is an industrial waste that is currently being investigated as a potential land applied product. Research pertaining to land-spreading wood ash is positive with respect to enhanced growth of agricultural crops (Lerner and Utzinger, 1986; Naylor and Schmidt, 1989; Campbell, 1990; Etiegni et al., 1991; Muse and Mitchell, 1995; Krejsl and Scalon, 1996; Meyers and Kopecky, 1998) and silvicultural crops (Weber et al., 1985; Silfverberg and Hotanen, 1989; Etiegni et al., 1991; Ferm et al., 1992). Increases in growth are attributed to increased percent Base Saturation (%BS), Cation Exchange Capacity (CEC), soil pH and micro/macronutrients (Kahl et al., Lerner and Utzinger, 1986; Naylor and Schmidt, 1986; Naylor and Schmidt, 1989; Ohno, 1990; Etiegni et al., 1991; Huang et al, 1993 and Krejsl and Scanlon, 1996).

The elemental compositions of ash can vary considerably between tree species and between the wood and bark of the same species. Environmental factors such as soil type and climate as well as industrial operation such as combustion temperature and ash collection systems will influence elemental composition (Campbell, 1990). Someshwar (1996) compared ash from 10 species of bark and 9 species of wood and found bark ash to have considerably higher levels of aluminum (Al) and silicates (Si). This is likely due to embedding of soil particles during logging operations. Unpublished data from Alberta-

Pacific Forest Industries Inc. (Alberta-Pacific) reports considerably higher levels of boron (B), cadmium (Cd) and zinc (Zn) in aspen (*P. tremuloides*) bark relative to the wood tissue.

Alberta-Pacific produces 45,000 m^3 or approximately 18,000 tonnes of ash per year. Landfilling is the current disposal method of these wastes, which is standard operating procedure for Alberta pulp mills and other electrical co-generation plants. Alberta-Pacific is interested in diverting this waste stream into a land-spread program in hopes of enhancing tree and cereal crop growth while expanding the life of their landfill.

4.2 Guidelines and Standards for Land Spreading

Guidelines for application rates for land spreading pulp mill solid waste currently do not exist in Alberta. Therefore, until such are available Alberta Environment Protection use and enforce application rates of heavy metals developed for other industries. Industrial application spreading rates are regulated by Alberta Tier 1 Criteria for Contaminated Assessment and Remediation. Alberta Environment Protection (AEP) recently proposed to adopt the ruling set by the Ontario Provincial Government to limit the soil concentrations of TCDD/TCDF (tetrachlorodibenzo-p-dioxin/tetrachlorodibenzofuran) to no more than 10 parts per trillion (10 pg/g). Total Toxic Equivalence (TEQ) of dioxin and furans of Alberta-Pacific's wood ash was below detection limits of 0.005 ppm by Cantest and 1.16 pg/g Alberta Research Council (Abboud and Macyk, 1994). Therefore, dioxin and furans are not a limiting factor in a land-spread program.

4.3 Limitations to Land Spreading Alberta Pacific's Fly Ash

Alberta Environment will only grant land-spreading application permits to products that will enhance soil properties and show an agronomic benefit to crops grown on the receiving soils. Parameters of greatest concern in using Alberta-Pacific's fly ash in a land-spread program are elements B, Cd and Zn as well as its high alkalinity and electrical conductivity. Alberta Environment has requested that soil parameters: SAR, EC, pH and elemental loading of B, Cd and Zn be monitored and analyzed to aid in evaluating the potential of using fly ash as land-spreading product. Alberta Environment also requested plant tissue analysis be monitored for bioaccumulation of the above-mentioned elements for the determination of safe agronomic loading rates.

4.4 Background

4.4.1 Sodium Adsorption Ratio (SAR)

Sodium adsorption ratio is the relationship of soluble sodium to soluble calcium + magnesium in water or soil solution. Soils with a SAR of 6-15 are considered slightly sodic and may reduce some crop yields by 20 - 40 % (Tisdale et al., 1993). Soils with a SAR value $\geq$ 15 are classified as sodic soils and can inhibit growth of most plants due to osmotic pressures. Because of the potential for poor crop growth of salt sensitive crops, Alberta Environment has adopted SAR 6 as a threshold in their assessments of contaminated sites.

4.4.2 Electrical Conductivity (EC)

Electrical conductivity (EC) is a measure of electrical resistance over a distance. Conductance of a solution depends directly on the amount of dissolved ions in solution and their ability to conduct electricity. Soil with an EC of 2 ds/m or greater have been known to inhibit growth of some salt sensitive agricultural crops. The accumulation of soluble salts in the soil profile inhibits crop growth by increasing osmotic potential, which can lead to plant nutrient imbalances (Tisdale et al., 1993). For these reasons the Terminology Committee of the Soil Science Society of America have suggested that soils with an EC of ≥ 2 ds/m be classified as saline. Alberta Tier 1 Criteria for Contaminated Soils has also adopted 2 ds/m as a threshold in their assessment of contaminated sites.

4.4.3 Soil pH

The use of wood ash as an alternative liming material has been studied by several investigators (Naylor and Schmidt, 1986; Silfverberg and Hotanen, 1989; Naylor and Schmidt 1989; Unger and Fernandez, 1989; Ohno, 1990; Etiegni et al., 1991; Ferm et al., 1992; Huang et al., 1992; Meiwes, 1995 and Muse and Mitchel, 1995). All of these studies showed an increased soil pH to near neutral conditions that lasted 3-5 years following ash additions. Soil pH is considered the most dominant solubility controlling factor influencing nutrient and heavy metal behavior in soil (Zahn et al., 1996). It has also been credited for much of the increased growth associated with ash land applications (Naylor and Schmidt, 1986; Etiegni et al., 1991).

4.4.4 Boron (B)

Boron (B) is 1 of 7 essential micronutrients required for normal plant growth. Boron is the most commonly deficient micronutrient for plants in coarse textured soils in humid regions (Gupta, 1993). Plants require B for cell elongation (Krueger et al., 1987). Boron requirements vary greatly among plant species and the difference between deficiency and toxic levels can be very narrow. Plant uptake of B, harvesting and removal of plant tissue, liming and leaching from the profile over time have combined to create soil B deficiencies in certain regions of Alberta.

Boron levels of the Alberta-Pacific's wood ash are very high and may be one of the trigger points that determine the maximum one-time/or maximum cumulative loading rate. Fertilizer guidelines recommend that a single application rate of B should not exceed 4.4 kg/ha. Alberta Tier One has set an upper limit of 2 ppm hot water extractable B. Therefore, Application rates of B without jeopardizing soil quality are will defined.

4.4.5 Cadmium (Cd)

Cadmium (Cd) naturally occurs in mineral soils and is associated with zinc, copper and lead minerals and phosphate rocks. A comprehensive survey of the Canadian prairies found that Ap horizons developed over glacial till derived from the Precambrian Shield had concentrations of <0.3 ppm. However, there are areas in the prairies that have Cd levels ranging from 0.8 to 3.8 ppm. These areas are relatively small and are associated with Cretaceous shale (Garrett, 1994). Some common agricultural practices like the addition of phosphate rock can add significant amounts of Cd to soil. Phosphorous commonly used in agricultural practice can contain as much as 400 ppm of Cd which is approximately 40 times that of the ash used in this study.

Alberta Tier One Guidelines were adopted to protect the soil and ensure that Alberta's soils are low in elements that could cause health risks from bioaccumulations of heavy metals and other substances that would restrict future agricultural uses. Alberta Tier One limits the total concentration of Cd to 1 ppm.

4.4.6 Zinc (Zn)

Zinc is 1 of 7 essential micronutrients that is required for normal healthy plant growth. Zinc deficiency in agricultural crops is a problem in many countries around the world. It is the most frequently encountered micronutrient deficiency (Lindsay, 1972). Zinc concentrations in Alberta soils and cereal crops are typically low (Alberta Agriculture, 1986) and can be easily adjusted by Zn fertilizers (Tisdale, 1993; Alberta Agriculture, 1992).

Ash characterization studies in the literature have not found Zn concentrations to be a limiting factor. Campbell (1990), Muse and Mitchell (1995), Zhan et al. (1996) and Meyers and Kopecky (1998) all report Zn concentration in various wood and bark species to range between 35.2-1250 ppm. Characterization of 25 ash samples from Alberta-Pacific indicates that there is great variability in Zn concentration. Concentrations range from 86-2969 ppm with and average of 1542 ppm. The average Zn concentration of Alberta-Pacific's ash is greayer than the maximum range found in the literature.

Zinc is a heavy metal that is regulated by Alberta Tier One. Total Zn allowed in the soil is 120 ppm. Alberta-Pacific may be restricted to a maximum one-time application of ash to soil due to the accumulation of Zn.

4.5 Objectives

The objective of this study was to determine beneficial agronomic loading rates of wood ash that will not jeopardize soil quality with respect for Alberta Tier One thresholds. Treatment levels for this experiment were determined in two ways:

1. First by considering the biomass results reported in Chapter 2. Treatment levels that were agronomically beneficial by increasing barley biomass ranged from 10 Mg/ha and 100 Mg/ha of ash.

2. Secondly a maximum one-time application rate of Zn equal to a soil concentration. The limiting application rate was 120 ppm was calculated at 67 Mg/ha of ash. Therefore, the ranges for ash application rates for this experiment were set between 10 Mg/ha and 67 Mg/ha.

4.6 Material and Methods

4.6.1 Soils

Eluviated Dystric Brunisol (E.DyB) soil was collected from an agricultural field near the Alberta Pacific pulp mill located 35 km northwest of Grassland, Alberta. E.DyB soil was collected from the Ap horizon of a Nestow soil series from quarter section SE 5-69-19 W 4th. The soil was air dried at 22°C and ground to pass through a 2 mm sieve. Soil was analyzed for physical and chemical parameters, which are presented in table form in Appendix 1.

4.6.2 Ash

Bark and wood waste derived fly ash was collected from the power boiler at Alberta-Pacific Forest Industries Inc. near Grassland, Alberta. Bark and wood waste is comprised of 80% hardwood and 20% softwood. Ash was air dried at 22° C and ground to pass through a 2 mm sieve. Fly ash from Alberta-Pacific is routinely collected and sent to Enviro-test Laboratories® in Edmonton, Alberta. Ash was characterized for total metals nutrients, available metal and nutrients and physical parameters and is presented in table form in Appendix 1.

4.6.3 Design

Dry ash and soil were premixed in a cement mixer for 5 minutes per loading rate to ensure a thorough blend. Four loading rates: control, 20, 40 and 60 Mg/ha equivalence were potted in 6-inch leak proof pots. Each loading rate was replicated 6 times. All pots were seeded with five seeds of Harrington barley (*Hordeum Vulgare*) and thinned back to three after germination.

Pots were placed in a greenhouse with a diurnal cycle of 16-day light hours and 8 dark. Room temperature was maintained between 21-24° C. Pots were watered every other day with even amounts of water. Deionized water (18.0 megohm cm^{-1}) was used to eliminate additional nutrient and trace element inputs.

Pre and post treatment soil samples were collected on various days throughout the experiment. Analysis included total metals, pH(1:2 $CaCl_2$ suspension), EC and SAR. Summarized in Table 4.1 is the sample schedule of what was sampled and when.

Table 4.1. Soil was extracted and analyzed for the following parameters on the corresponding days.

Day Sampled	Total Elements	Detailed Salinity	Alkalinity	pH CaCl2	EC	Selected Available Elements
0	*	*		*	*	*
1	*			*	*	
5		*	*	*	*	
10				*	*	
15				*	*	
20		*	*	*	*	
30				*	*	*
50		*	*	*	*	
70				*	*	
90		*	*	*	*	

* = Sampled for corresponding parameter.

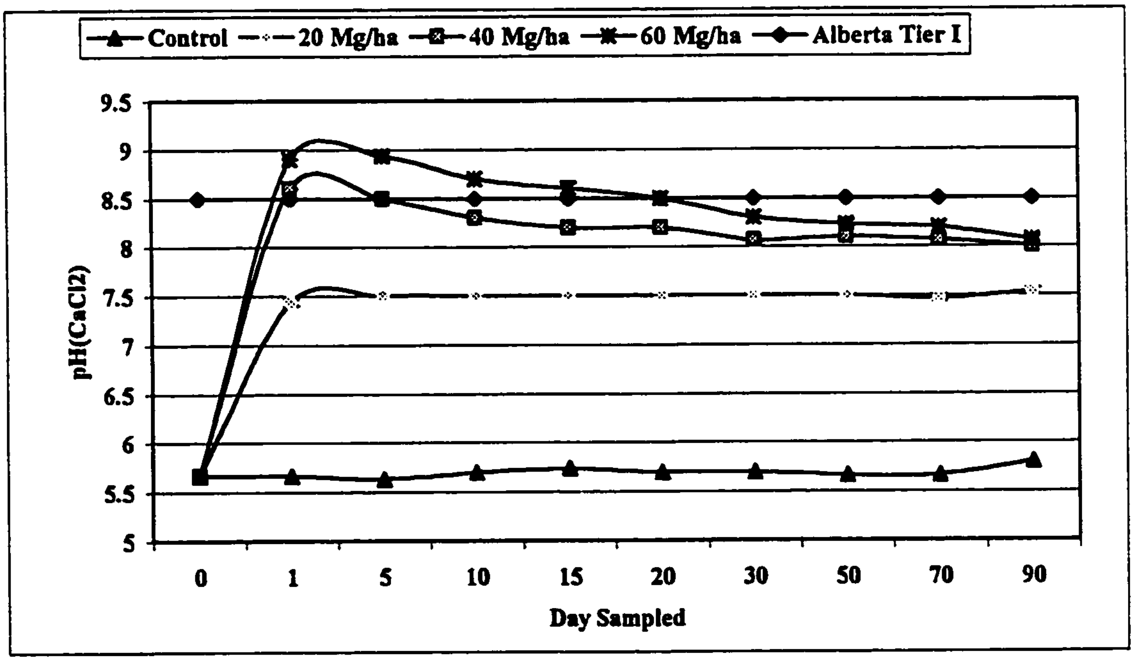
4.6.4 Statistical Analysis

Statistical analysis of the data collected for heavy metal uptake was determined using the Fisher's Protected Least Significant Difference (LSD). LSD can only be completed if the Probability >F (Prop>F) calculated from the ANOVA is ≤ 0.05.

4.7 Results

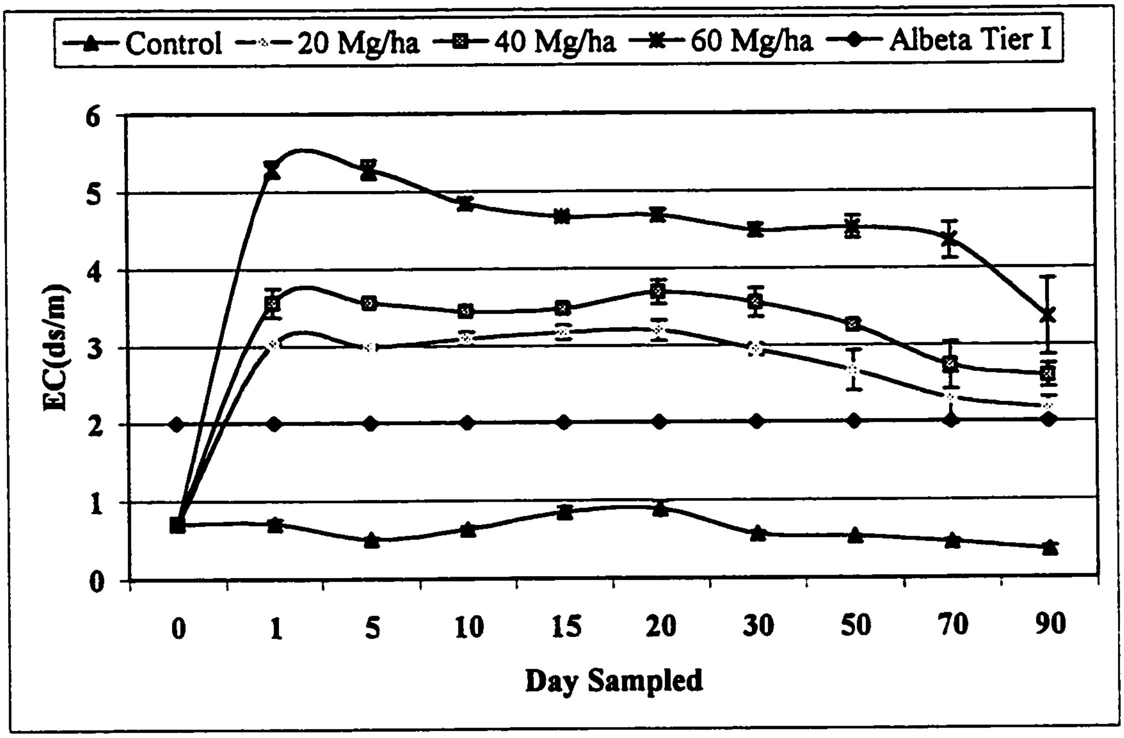
Additions of wood ash had an immediate effect on soil pH($CaCl_2$) (Figure 4.1). The 20 Mg/ha treatments raised the soil pH to 7.5 with in a 24-hour period and the soil pH remained constant at 7.5 throughout the 90-day experiment. The 40 and 60 Mg/ha treatments increased soil pH($CaCl_2$) within 24 hours to levels over Alberta Tier One Guidelines for Contaminated Sites. However, after 5 days the soil buffering capacity lowered the soil pH to 8.5, which is the upper limit of Alberta Tier 1. After 20 days the soil buffering capacity also lowered the 60 Mg/ha treatments to the upper limit of Alberta Tier One. By day 30 the pH in the 2 high-end treatments stabilized at pH of 8.2 for the remainder of the 90-day experiment. The control remained constant at pH 5.6 to 5.4 throughout the experiment.

Figure 4.1. Eluviated Dystric Brunisolic soil pH($CaCl_2$) responses to wood ash addition at various loading rates over a 90-day period.



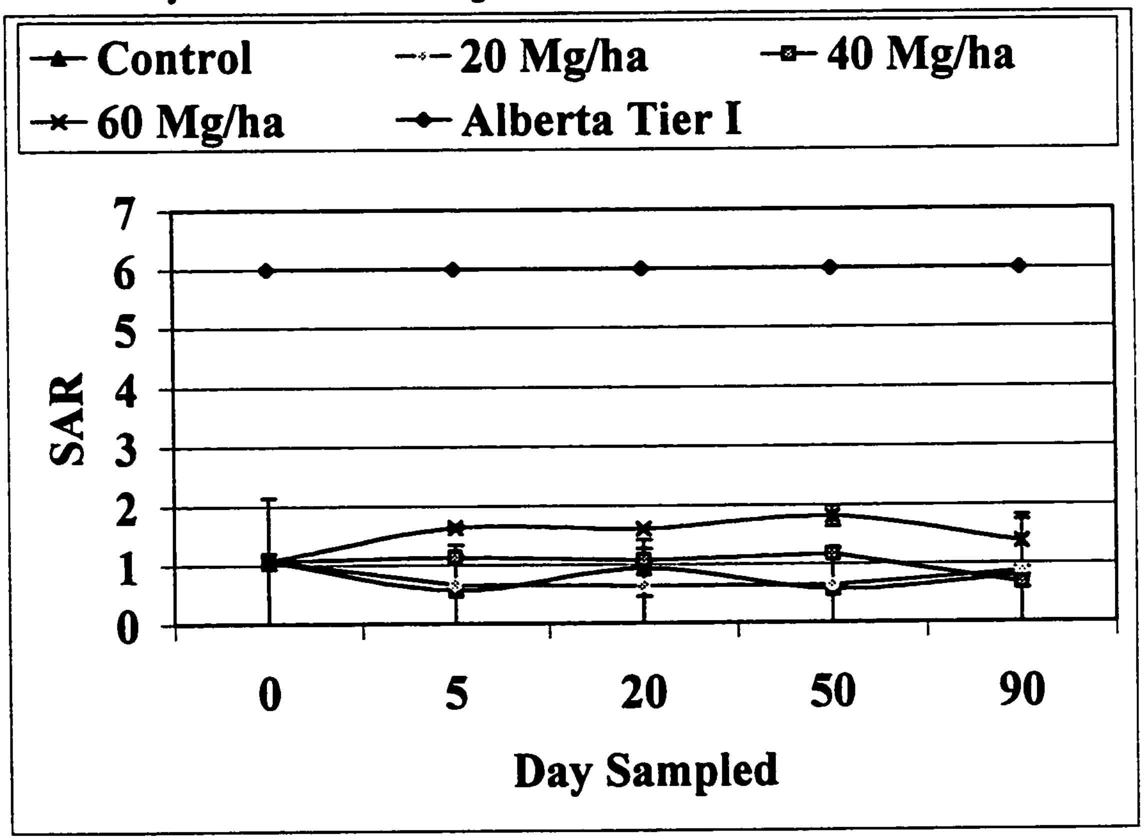
Electrical conductivity was greatly affected by the addition of fly ash. All 3 treatments increased soil EC over Alberta Tier One limits (Figure 4.2). Greater loading rates had a corresponding order of magnitude increase in EC. After 90-days of sampling the EC had subsided but still remained over Alberta Tier One. The control remained relatively constant fluctuating slightly between 0.5 and 0.8 ds/m.

Figure 4.2 Electrical conductivity response of Eluviated Dystrict Brunisolic soils following the additions of fly ash at various loading rates.



SAR was not greatly affected by the addition of fly ash to the soil (Figure 4.3). SAR slightly increased in all ash loading rates as compared to the control. The increase remained constant over the first 50 days of the experiment but by the 90th day the SAR in all treatments was similar to the control. The 60 Mg/ha treatments had the greatest increase on the SAR; however, even at this loading rate the increase in SAR did not approach the Alberta Tier One limit of 6.

Figure 4.3. SAR response of Eluviated Dystric Brunisolic soils following the addition of fly ash at various loading rates.



Elemental loading of the 3 elements of concern B and Zn did not exceed soil-loading limits set by Alberta Tier One Guidelines (Table 4.2). However, B and Zn concentrations in E.DyB soil significantly increased between each loading rate and demonstrate a trend of increasing concentration with increasing ash additions.

Cadmium was more variable within each loading as well as between each loading rate and does not show a clear trend between incremental loadings. Cadmium levels in the soil exceeded Alberta Tier One Limits in the 20 Mg/ha loading. Total Cd in the 40 and 60Mg/ha treatments were on average lower than that of the control. Non-detection (<0.5 ppm) was recorded in the Cd analysis in the 0, 40 and 60 Mg/ha loading rates. There was likely a laboratory error in the analysis that resulted in such large variation of total Cd in soil. This made statistical analysis irrelevant for the purpose of this study.

Table 4.2. Available B, total Cd and Zn content following wood ash application to Eluviated Dystrict Brunisolic soils.

Treatments	Available B ♦(SE) ♣LSD	Total Cd (SE) LSD	Total Zn (SE)LSD
Mg/ha	ppm		
Alberta Tier One Limits	2	1	120
Control	♦0.440 (0.023) ♥d	0.767 (0.218)	45.7 (0.926) d
20	0.847 (0.035) c	1.100 (0.251)	66.0 (0.436) c
40	1.330 (0.033) b	0.667 (0.167)	84.2 (0.600) b
60	1.800 (0.058) a	0.533 (0.210)	112.3 (1.453) a

♦SE = Standard Error based on four replicates in each treatment.
♣LSD = Least Significant Difference (p<0.05), indicates that at least two treatments are statistically significant at the p = 0.05 level.
♥a, b, c, d = treatments means with the same letter are not statistically significant from each other.
♦Mean value for treatment n=3

4.8 Discussion

Wood ash was characterized and compared to Alberta Tier One Guidelines for Contaminated Sites (Appendix 1). From this characterization it was determined that soil parameters pH, EC, SAR and elements B, Cd and Zn would have to be monitored in order to determine a safe loading rate at which to apply wood ash generated from Alberta-Pacific Forest Industries Inc. Alberta Environment has agreed that wood ash can be applied to agricultural soil provided that all the above soil parameters are monitored and the soil environment shows no negative impact with respect to Alberta Tier One.

4.8.1 Electrical Conductivity (EC)

Electrical conductivity of E. DyB soil was greatly affected by the addition of fly ash (Figure 4.2). All loading rates 20, 40 and 60 Mg/ha increased the EC of the soil over Alberta Tier One limits of 2 ds/m. By definition the addition of wood ash to soil at these loading rates created saline soil conditions and should have produced unfavorable soil conditions for vegetation (Tisdale et al., 1993).

Loading rates chosen for this experiment were based on beneficial growth response of Harrington Barley (*Hordeum Vulgare* var. Harrington) and Assiniboine poplar (*P. tremuloidies* Var. Assinboine) to ash additions as outlined in Chapters 1 and 2. Electrical conductivity had little effect on the growth of barley, as there was a significant increase in above ground tissue in these same loading rates. Assiniboine poplar showed reduced growth with increased loadings over 15 Mg/ha. Reduced growth may have been a result of saline soil conditions.

Figure 4.2 shows EC decreasing over the course of the study. As the ash in the soil weathers, site conditions may improve for *Populas* spp. Field studies following wildfires in north-central Alberta showed more favorable seedbed conditions and tree survival of highly salt sensitive jack pine (*Pinus banksiana*) after 2 years of leaching soluble salts out of the seed germination zone (Thomas and Wein, 1993).

4.8.2 Sodium Adsorption Ratio (SAR)

The SAR of the receiving soils in this experiment were not affected by the addition of wood ash. SAR slightly increased in all ash loading rates as compared to the control as shown in Figure 4.3. This increase remained constant over the first 50 days of the experiment and by the 90th day all loading rates were similar to the control. The 60 Mg/ha loading had the greatest increase in SAR, however, even at this loading rate the increase in SAR did not exceed a value of 2. Thacker (1995) in a field study with an application of 50 Mg/ha and also reported no significant increase in SAR.

4.8.3 Soil pH

The addition of wood ash increased the pH of Eluviated Dystric Brunsolic (E.DyB) soils (Figure 4.1). Soil pH increased immediately after wood ash application in all treatments. The 20 Mg/ha treatment increased from $pH_{(CaCl2)}$ 5.7 to 7.4 where it stabilized for the remainder of the 90-day experiment.

Treatments 40 and 60 Mg/ha increased pH rapidly and exceeded Alberta Tier One limits of $pH_{(CaCl2)}$ 8.5. After 5-days of incubation the buffering capacity of the soil lowered the 40

Mg/ha treatments below pH 8.5. Meyers and Kopecky (1998) found a similar result following 78 days of incubation the soil pH in 40 Mg/ha treatments lowered from the initial spike to a pH 8.2. The 60 Mg/ha loading was used to test the soils capacity to buffer a high application of ash. Twenty-days after application the pH was reduced to levels below Alberta Tier One limits. Naylor and Schmidt (1989) used loading rates as high as 50 Mg/ha and found that 150-days after the application the soil pH was 7.4.

4.8.4 Boron (B)

Alberta Agriculture has defined soils with less than 0.4 ppm of hot water extractable B to be deficient in B (Alberta Agriculture, 1992). E.DyB soils collected for this experiment were coarse textured soils under cultivation in the sub humid regions of north central Alberta. Therefore, it was of little surprise to find these soils bordering on B deficiency (Table 4.2).

The addition of ash increased B availability of the soil in all treatments. Boron concentrations increased from near deficit concentrations of 0.44 ppm (hot water extractable) in the control, to 0.8, 1.3 and 1.8 ppm. The treatment concentrations are all under Alberta Tier one limits of 2 ppm and improved the soil fertility rating to adequate with respect to B (Alberta Agriculture, 1992). High B concentrations of Alberta -Pacific's ash are consistent with characterizations of ash from several tree species (Campbell, 1990; Muse, 1995, Meyers, 1998). Meyers (1998) also reported on the increased available B following land application ash.

The addition of wood ash in higher treatments of 40 and 60 Mg/ha exceeded the single application rate of 4.4 kg/ha recommended by fertilizer guidelines. Loading rates of 40 and

60 Mg/ha of ash were the equivalent of applying 5.52 and 8.28 kg of B/ha, respectively. Increasing soil pH can inversely affect the availability of B (Gupta, 1985). Therefore, the low background concentration of E.DyB soils and increased soil pH may limit the availability of B in the short term. As the soil begins to buffer the increased pH effect of the ash, however, more B could become available and the higher loading rates could then exceed Alberta Tier One limits and crops may start to show B toxicity symptoms over the long term. More research is needed in this area before loading rates as high as 60 Mg/ha can be prescribed.

4.8.5 Cadmium (Cd)

Cadmium levels in this study were quite variable both within each of the treatments as well as across the treatments. Statistical analysis of Cd could not be done in this experiment because most of the treatments had one or more values below the detection limit of 0.5 ppm. The variable background level of soil combined with the variable composition of wood ash may account for some inconsistency in total Cd as reported in Table 4.2.

Characterization of 25 samples of ash collected from Alberta-Pacific showed Cd ranging from 7.3-19 ppm. Variability in Alberta-Pacific's ash is consistent with other ash-Cd concentrations found in the literature. Campbell (1990), Muse (1995), Zhan (1996) and Meyers (1998) found Cd concentrations ranging from 0.18-26 ppm depending on tree species and location grown.

Analytical spikes of Cd found in the control and 20 Mg/ha treatments may also be an effect of residual phosphorous fertilizer from previous farming practices. Characterization of rock phosphorous commonly used in agricultural practice can contain as much as 400 ppm of Cd which is approximately 40 times the concentration of wood ash. Eluviated Dystric Brunisolic soils require high fertilizer inputs in order to be productive, therefore, Cd may have accumulated over time through fertilizer inputs. Background characterization of Cd in the soil is high with respect to crustal abundance of <0.3 but is well within the expected range (Krauskopf, 1979).

4.8.6 Zinc (Zn)

The addition of ash to the soil showed an incremental increase with every increase in ash application; however, Zn never exceeded the Alberta Tier One even at the highest loading rate of 60 Mg/ha (Table 4.2). Zinc is a heavy metal that is controlled under Alberta Tier One. Total zinc allowed in the soil under these guidelines is 120 ppm. Mathematical calculations indicate that Zn will be a limiting factor to the maximum one time application rate of ash to soil. Our calculations indicate that Zn will exceed Alberta Tier One at approximately 67 Mg/ha.

Ash application of 60 Mg/ha did approach Tier One levels (Table 4.2). Comparing the literature for Zn concentration Alberta-Pacific's ash is considerably higher in Zn than ashes reported by (Campbell, 1990; Muse, 1995; Zhan, 1996 and Meyers and Kopecky, 1998). The range of Zn concentrations for their studies were between 35.2-1250 ppm. Characterization of 25 ash samples from Alberta-Pacific indicates that the variability in Zn is between 86- 2969 ppm with an average concentration of 1542 ppm and a standard error of 245.5 ppm. The average Zn concentration of Alberta-Pacific's ash is higher than the maximum range found in the literature.

4.9 Conclusion and Recommendations

With the exception of SAR, all the soil parameters (EC, pH, B, Cd and Zn) investigated in this experiment increased with the addition of wood ash. The EC and Cd levels exceeded the regulatory limits with respect to Alberta Tier One Guidelines for Contaminated Soils in this closed pot greenhouse experiment. Under field conditions soil is interacting with several environmental factors like climate, microbial activities and leaching processes that would dilute wood ash response.

Cadmium levels in the 20 Mg/ha loading exceeded Alberta Tier One but in the higher loading rates of 40 and 60 Mg/ha total Cd levels were lower than the control. Data analysis is likely vary suspect. Soil pH increased in the 20 Mg loading to pH 7.5 while the higher loading rates exceeded Alberta Tier One of pH 8.5. Soil buffering capacity was able to correct the soil pH after 20 days. Boron and Zn increased in soils with incremental loading but did not exceed regulatory limits.

If ash is to be used as a land-spreading product, background analysis of the above mentioned soil parameters should be routinely done to ensure the environmental sustainability of the soil is not compromised. More research needs to be done focusing on loading rates with a tighter window around 20 Mg/ha. At this treatment level there was minimal environmental impact on the soil. Electrical conductivity increased over Alberta Tier One limits but appeared to be declining rapidly towards the end of the 90-days. Background Cd levels was too variable and may need to be one of the prescreening criteria for land-spreading wood ash on agricultural land.

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CHAPTER FIVE

ELEMENTAL UPTAKE

5.1 Introduction

The addition of fly ash to agricultural soils is not a new concept. In Mexico the native Minas used to clear the land of trees and burn the slash and spread the ashes on the land to increase the growth of crops. As the soil would slowly become less productive the nomadic tribes would then move to a new location and start the process over again. In Canada, settlers cleared much of the northern agricultural land less than sixty years ago. Many older generation farmers can testify to crops growing better for years, where the burn piles were. Hopkins (1910) reports of field trials being conducted as early as 1891-1904 showing increased growth rates of wheat, oats and barley. Wood ash has long been regarded as a source of potash and the practice of using wood ash was replaced with cheaper forms of potassium fertilizers.

Much of the previous scientific research is focused on the increased yields of silvicultural and agricultural crops and micro and macronutrients provided by ash that contributes to growth. In Finland, soil treated with fly ash has shown to increase growth of trees by 25 fold when compared to trees grown in untreated soils (Silfverberg and Hotanen, 1989). Similar results by other researchers also show increased growth in silvicultural crops (Weber et al., 1985; Etiegni et al., 1990; Ferm et al., 1992). Agricultural trials in the United States used loading rates of 30 Mg/ha and demonstrated a 45% increased crop growth (Krejsl and Scalon, 1996). Other similar studies have also shown beneficial growth of agricultural crops in fly ash treated soils. These growth increases were attributed to the increased soil pH and available P, K, Ca and other micro nutrients provided by the ash (Unger and Fernandez , 1990;; Etiegni et al., 1991a; Etiegni et al., 1991b; Ohno, 1990; Meiwes, 1995; Naylor and Schmidt, 1986; Erich, 1991; Meyers and Kopecky, 1998; Naylor and Schmidt, 1989 and Lickacz et al., 1999).

Stricter guidelines on landfill designs have increased the cost of land filling industrial waste. This has forced industry to search for alternative uses for industrial byproducts in hopes of reducing operating costs. Alberta Environmental Protection (AEP) is concerned that fly ash producing industries would begin using agricultural and silvicultural land as waste sites. After reviewing the ash characterization from Alberta-Pacific Forest Industries Ltd., AEP had concerns as to the bioaccumulation and potential toxicity from boron (B), cadmium (Cd) and zinc (Zn). At their request, a greenhouse study was designed to address these concerns before field-testing of wood ash started.

The concentration of B, Cd, and Zn in plant tissue has been studied in greenhouse and field trials. In either method, tissue concentrations of Cd and Zn in cereal grains and forages grown in loading rates as high as 20 Mg/ha showed no significant increase over the control (Meyers and Kopecky, 1998; Lerner and Utzinger, 1986). Krejsl and Scalon (1996) found that concentrations of Zn actually decreased in oat and bean crops at loading rates as high as 50 Mg of ash/ha. All three of these studies found tissue concentrations of B to significantly increase in ash-amended soils. However, none of the tissue concentration levels reached toxic or near toxic levels for any of the plant species investigated.

Uptake of B, Cd and Zn by plants is highly specie specific and soil pH dependant (Gupta, 1971; Kabata-Pendias and Pendias, 1984; Bailey et al., 1996; Zhan et al., 1994). Zhan et al. (1994) studied the solubility of heavy metals of three different wood ashes at pH's ranging from 3.8 to 7, and reported that at pH > 6.5 both Cd and Zn were not very soluble. He further showed that pH levels < 6 significantly increased the solubility of both Cd and Zn and by pH 4, 80 - 100% of Cd and 70 - 90 % of Zn is soluble. Gupta (1971) showed similar results, with B plant toxicity increased with diminishing soil pH. Zhan et al. (1994) speculated that with time following liming with wood ash, that soil pH will decrease and plant availability and uptake of soluble Cd and Zn by plants will increase.

5.2 Background

5.2.1 Boron

Boron is 1 of 7 essential micronutrients required for normal plant growth. Boron is the most common deficient micronutrient for plants in coarse textured soils in humid regions (Gupta, 1993). Boron is needed for cell elongation (Krueger, 1987) and facilitates the availability of UDPG-pyrophosphorylase for cellulose synthesis (Dugger, 1980).

Boron requirements vary greatly from specie to specie and the window between deficiency and toxicity can be very narrow. Barley plants may show B deficiency symptoms at <3 ppm at the silage stage. Deficiency symptoms appear as stunted growth and chlorotic leaves. Boron is not translocated easily within the plant, therefore, actively growing plants can have healthy older leaves but new leaves may have irregular shaped lesions and pale yellow chlorosis (Alberta Agriculture, 1992). Boron toxicity under field conditions generally occurs around 200 ppm in plant tissue (Gupta, 1985), but critical B toxicity is specie dependent. Boron toxicity can occur in barley plants at tissue concentration above 40 ppm at the boot stage (Reuter, 1986)

5.2.2 Cadmium

Cadmium (Cd) is a heavy metal naturally found in soil. Accumulation and distribution of Cd in plant tissue is specie dependant and varies among cultivars of the same specie (Chang, 1982). Plants do not require Cd for metabolic reasons; however, Cd tends to accumulate in plant tissue in higher concentration than soil solution (Cutler, 1974). Plants can accumulate high levels of Cd without showing any adverse effects in growth (Kuboi, 1986). Cereal and legumes appear to be the most sensitive to Cd toxicity. Outridge (1994) found that yields of cereal grains were reduced at soil concentrations >1.8 ppm. Deciduous and coniferous trees are more tolerant as reduced growth does not occur until soil concentrations are > 100 ppm (Kelly, 1979).

Canadian soils are among the lowest in the world with respect to Cd levels. Historically, increases to Cd levels to agricultural soils have occurred with the addition of phosphate fertilizers and animal manure (Bailey et al., 1996). More recent increases are attributed to human and industrial waste applications. Heavy metals entering the soil-plant-human food chain are a human health problem. Approximately 95% of Cd uptake in Canadian diets occurs in cereal, fruits and vegetables (Bailey, 1996). In extreme cases, long-term high ingestion of Cd induces kidney failure and liver damage (Friberg, 1985). Because of these concerns Alberta Environmental Protection has imposed soil limits for Cd and other heavy metals.

5.2.3 Zinc

Zinc is 1 of 7 essential micronutrients required for normal healthy plant growth. It is adsorbed through the root by diffusion and is partly mobile within the plant (Alberta Agriculture, 1992; Lindsay et al., 1972). Zinc is involved in the same enzymatic functions as Mn and Mg, but is the only component of the plant that regulates carbonic anhydrase activity within the plant (Brown et al, 1993; Jones et al., 1991; Ohki, 1976). Plant deficient symptoms are observed at leaf concentrations below 12 ppm. Plant sufficiency ranges from 15-150 ppm and phytotoxicities range from 300 to 1000 ppm depending on plant species (Tisdale et al, 1993; Jones et al., 1991).

Zinc deficiency in agricultural crops is a problem in many countries around the world. It is the most frequently encountered micro nutrient deficiency (Lindsay, 1972). Zinc concentrations in Alberta soils and cereal crops are typically low (Alberta Agriculture, 1986) and can be easily adjusted by Zn fertilizers (Tisdale, 1993; Alberta Agriculture, 1992). Symptoms of Zn deficiency are specie dependant. Cereal crops will show chlorotic stems and leaves in the middle section of the plant. More severe symptoms are failure to elongate and shorter stems with fan shaped clusters of leaves at the top.

Zinc is an important element in the diets of humans and other animals. Zn plays a role in the prevention of heart and skin diseases, skeletal development and male fertility (Welch, 1993; Alberta Agriculture, 1992, 1986; Lindsay, 1972). Alberta Agriculture (1990) recommends that 70% of daily Zn requirements for domestic farm animals should come from vegetation with the remaining 30% from feed additives (Welch 1993).

Zinc is a heavy metal and is regulated by Alberta Environmental Protection under Alberta Tier One Guidelines for Contaminated Sites. Wood ash has a high concentration of Zn and is one of the limiting factors when considering total acceptable accumulative loading of ash.

5.3 Objective

The objective of this research was to determine potential loading rates of wood ash that will not increase elemental levels of boron, cadmium and zinc to toxic or near toxic levels within any component of the plant tissue. Ash application to soil has been shown to increase growth rates of cereal crops. Therefore, in order to determine safe environmental and agronomic loading rates further research is required to determine the extent of bioaccumulation of the above mentioned elements.

5.4 Material and Methods

5.4.1 Soils

Eluviated Dystric Brunisol (E.DyB) soil was collected from an agricultural field near the Alberta Pacific pulp mill located 35 km northwest of Grassland, Alberta. E.DyB soil was collected from the Ap horizon of a Nestow soil series from quarter section SE 5-69-19 W 4th. The soil was air dried at 22°C and ground to pass through a 2 mm sieve. Soil was analyzed for physical and chemical parameters, which are presented in table form in Appendix 1.

5.4.2 Ash

Bark and wood waste derived fly ash was collected from the power boiler at Alberta-Pacific Forest Industries Inc. near Grassland, Alberta. Bark and wood waste is comprised of 80% hardwood and 20% softwood. Ash was air dried at 22° C and ground to pass through a 2 mm sieve. Fly ash from Alberta-Pacific is routinely collected and sent to Enviro-test Laboratories® in Edmonton, Alberta. Ash was characterized for total metals nutrients, available metal and nutrients and physical parameters and is presented in Appendix 1.

5.4.3 Design

Dry ash and soil were premixed in a cement mixer for 5 minutes per loading rate to ensure a thorough blend. Four loading rates control, 20, 40 and 60 Mg/ha equivalence were potted up in 6-inch leak proof pots. Each loading rate was replicated 6 times. All pots were seeded with five seeds of Harrington barley and thinned back to three after germination.

Pots were placed in a greenhouse in complete randomization with a diurnal cycle of 16-day light hours and 8 dark. Room temperature was maintained between 21-24°C. Pots were watered every other day with even amounts of water. Deionized water (18.0 megohm cm^{-1}) was used to eliminate additional nutrient and trace element inputs.

Silage samples were collected after 70 days of growing. Silage was collected by completely harvesting barley tissue from 3 out of 6 pots/treatment. Tissue samples were oven dried at 55°C for 72 hours, ground and sent to Enviro-Test Laboratories® for analysis.

Grain and straw samples were allowed to grow until ripened naturally between 90 to 95 days. Grain was separated from the straw by hand thrashing and both the straw and grain were oven dried to 55°C for 72 hours. Grain and straw samples were ground and sent to Enviro-Test Laboratories® for analysis.

Plant samples were digested using 2 parts concentration HNO3 and 1 part concentration HClO4 to white fumes of HClO4 (Bock, 1979). Metal analysis was conducted by ICP MS according to Environmental Protection Agency (EPA) Method 6010A.

5.4.4 Statistical Analysis

Statistical analysis of the data collected for heavy metal uptake was determined using the Fisher's Protected Least Significant Difference (LSD). LSD can only be completed if the Probability >F (Prop>F) calculated from the ANOVA is ≤ 0.05.

5.5 Results

Changes in boron concentration in the straw, seed and whole plant tissue were very consistent across the treatments (Tables 5.1.- 5.3). Boron levels had no significant increase over the control in the 20 Mg/ha loading rate in any of the treatments. There was no increase in B concentration in barley seed in the 40 Mg/ha treatments. However, significant increases ($p \leq 0.05$) in B concentration were detected in the straw and whole plant tissue at the 40 Mg/ha treatments. Increased ($p \leq 0.05$) was noted in all 3-plant components at the 60 Mg/ha treatments.

Cadmium levels in the whole plant tissue and the seed analysis had no significant increase over the control across all treatments. However, the straw component of the analysis did show an increase ($p \leq 0.05$) in the 40 and 60 Mg/ha treatments over the control.

Zinc concentration in the total plant tissue significantly decreased ($p \leq 0.05$) in the 20 Mg/ha treatments as compared to the control. Zinc concentration significantly increased ($p \leq 0.05$) in the 40 and 60 Mg/ha treatments of the total plant tissue. Zinc concentration in the straw shows a similar pattern with a non significant reduction in the 20 Mg/ha loading and a significant increase ($p \leq 0.05$) in the greater two treatments compared to the control. Analysis of the seed shows a linear significant increase ($p \leq 0.05$) in each of the treatments.

Table 5.1. Elemental analysis of the total above ground plant tissue of Harringtong barley (*Hordeum Vulgare*) collected at day 70. (Silage stage of growth or at the soft dough stage).

Treatment Mg/ha	Total B [♦] (SE) [•]LSD ppm	Total Cd (SE) LSD Ppm	Total Zn (SE) LSD ppm
Control	[•]15.67 (0.51) [♥]c	0.29 (0.02) a	36.37 (0.48) c
20	15.67 (0.19) c	0.37 (0.04) a	28.57 (0.39) d
40	24.67 (1.58) b	0.43 (0.02) a	43.50 (0.90) b
60	43.67 (1.95) a	0.38 (0.01) a	60.03 (1.20) a

[♦]SE = Standard Error based on four replicates in each treatment.
[•]LSD = Least Significant Difference ($p<0.05$), indicates that at least two treatments are statistically significantlt different at the $p = 0.05$ level.
[♥]a, b, c, d = treatments means with the same letter are not statistically significant from each other.
[•]Mean value for treatment n=3

Table 5.2. Elemental analysis of Harrington barley (*Hordeum Vulgare*) seed (grain) portion. Harvest at full maturity at day 95 of growth.

Treatment Mg/ha	Total B [♦] (SE) [•]LSD ppm	Total Cd (SE) LSD ppm	Total Zn (SE) LSD ppm
Control	[•]2.67 (0.19)[♥] b	0.115 (0.017) a	34.7 (0.58) d
20	1.67 (0.19) b	0.125 (0.011) a	42.9 (2.57) c
40	2.67 (0.19) b	0.163 (0.011) b	51.6 (2.25) b
60	4 (0.34) a	0.187 (0.008) b	61.7 (0.15) a

[♦]SE = Standard Error based on four replicates in each treatment.
[•]LSD = Least Significant Difference ($p<0.05$), indicates that at least two treatments are statistically significant at the $p = 0.05$ level.
[♥]a, b, c, d = treatments means with the same letter are not statistically significant from each other.
[•]Mean value for treatment n=3

Table 5.3. Total elemental analysis of leaf and straw tissue minus the seed of Harrington barley (*Hordeum Vulgare*). Harvested at full maturity at day 95.

Treatment Mg/ha	Total B ♠ (SE) ♣LSD ppm	Total Cd (SE) LSD Ppm	Total Zn (SE) LSD ppm
Control	♦16.3 (1.02) ♥c	0.21 (0.03) b	26.43 (1.49) c
20	21 (1.76) c	0.34 (0.02) ab	25.43 (1.46) c
40	32 (0.67) b	0.44 (0.01) a	43.5 (0.93) b
60	39.67 (1.17) a	0.43 (0.002) a	58.77 (2.39) a

♠SE = Standard Error based on four replicates in each treatment.
♣LSD = Least Significant Difference ($p<0.05$), indicates that at least two treatments are statistically significant at the $p = 0.05$ level.
♥a, b, c = treatments means with the same letter are not statistically significant from each other.
♦Mean value for treatment n=3

5.6 Discussion

Fly ash additions to soil did increase tissue concentrations of B, Cd and Zn in various parts of the barley plant tissue.

5.6.1 Boron

Fly ash increased plant B concentration levels of barley silage, straw and seed components in the 40 and 60 Mg/ha treatments and showed no significant increase in the 20 Mg/ha treatments. Plant B concentration levels in the 40 and 60 Mg/ha treatments did approach and exceed B toxicity levels but no toxicity symptoms were noted. Reuter,

(1986) found that B toxicity symptoms in barley could occur at concentrations of 40 ppm. Nable et al. (1990) reported that concentration of B in plant tissue grown in greenhouse experiments could exceed toxicity levels without showing any symptoms. Nable explained that high evapotranspiration rates of greenhouses could increase the amount of water and soluble B extracted from soil and deposited on the leaf surfaces through transpiration processes, which then contributes to higher B concentration. Concentration levels of unwashed leaves can be 10 to 50 fold higher than that of the stem.

Nable et al.(1990) showed that spraying or watering greenhouse plant experiments to simulate rainfall lowers B concentrations of leaf tissue analysis. A literature review comparing B uptake in a variety of plants and loading rates of wood ash is consistent with Nable et al. (1990) conclusions. Consistently B levels in plants grown in greenhouses are reported to be higher than field trials in similar loading rates (Naylor and Schmidt, 1989; Etiegni et al., 1991; Muse and Mitchell, 1995; Krejsl and Scalon,1996; Meyers and Kopecy,1998) . Another explanation for the higher B levels in greenhouse experiments is that the roots are confined to the pot and incorporated ash. Agricultural plants grown in field studies will have roots reaching depths of 1 meter and greater depending on plant species. In field studies Naylor and Schmidt, (1989) and Muse and Mitchell, (1995) reported no increase in B concentration in plant tissue.

5.6.2 Cadmium

Fly ash additions showed no significant increase in Cd tissue concentrations in the 20 Mg/ha treatments. Cadmium levels increased in all plant components over their respective controls, however, only the straw and seed components in the 40 and 60 Mg/ha treatments were significantly higher. This is supported by previous research that claims

Cd levels do not significantly increase in plant tissue at loading rates as high as 20 Mg/ha (Meyers and Kopecky, 1998; Lerner and Utzinger, 1986). Increases at the higher loading rates may be explained by Nable's et al. (1990) explanation of increased mineral deposition on leaf surfaces in greenhouse studies and the fact that roots are confined to the incorporation zone. However, wood ash can change environmental factors that increase the potential for Cd uptake. Because of potential health risks associated with Cd bioaccumulation, the environmental factors need to be addressed in order to determine safe loading limits.

Environmental factors influencing Cd solubility are: increased soil concentration of Cd, an increase in competing ions for sorption sites, salinization of soils and soil pH.

Table 4.1 shows that there was no significant increase in total soil Cd concentration between treatments, even though Cd levels in wood ash are in measurable concentrations (Appendix 1). The solubility of Cd in the soil can be expected to increase with increases in total soil concentration. (Grant et al., 1996). Wood ash also adds several divalent cations like Zn and Ca, which compete for sorption sites and can displace Cd into soil solution (Christensen, 1984). It is this increase in soil solution concentration that increases the availability of Cd to plants and the potential to increase its concentration in plant tissue.

Salinization of soil can also increase soluble Cd concentration. Wood ash supplies additional anions like Cl, which remain suspended in soil solution. Anions in solution compete with cation exchange sites, which can increase Cd concentration in solution (Garcia – Miragaya and Page, 1976). Figure 4.2 of this study shows that the soil became saline following wood ash treatment. Coupled with the potential increased Cd concentration, this may have resulted in the higher Cd concentration in plant tissue.

In all publications on the subject, soil pH is considered the most dominant solubility-controlling factor for Cd. Cadmium becomes most soluble in soil between pH 4.5 and 5.5 and decreases rapidly with increase soil pH. Using fly ash as a liming agent, Zhan (1994) reported that at pH levels greater than 7 - 8 (soil texture dependant) almost no Cd is in solution. Figure 4.1 of this study shows that soil $pH_{(CaCl2)}$ increased and stabilized at 7.5 to 8.5 (treatment dependant). Therefore, despite all other environmental influences affecting Cd solubility, soil pH has likely limited the solubility of Cd (i.e. availability) resulting in small increases in tissue concentration across the treatments.

Zhan et al (1994) further states that as the buffering capacity of soil begins to lower soil pH, it is conceivable that there will be an increase in heavy metal uptake by plants grown in soils treated with wood ash. Therefore, loading rates of wood ash should be made with the knowledge of total metal concentration in soils prior to treatment and not on short-term results on heavy metal uptake by plants.

5.6.3 Znic

In the 20 Mg/ha treatments, Zn showed a slight reduction in tissue concentration in the silage and straw component. The seed portion significantly increased linearly across all treatments. Lower plant tissue concentrations of Zn in pot trials are also reported by Meyers and Kopecky, (1998) and Lerner and Utzinger, (1986) at loading rates of 20 Mg/ha. Environmental factors controlling Cd solubility are the same for Zn, therefore, much of what was said for Cd holds true for Zn. Zhan et al.(1994) reported that Zn is not released from wood ash at soil pH greater than 7. The decrease in Zn concentrations in plant tissue within the 20 Mg/ha loading rate may have resulted from a reduction of Zn solubility with increased soil pH or competition from additional divalent cations added with the ash.

Significantly increased tissue concentrations of Zn in the 40 and 60 Mg/ha treatments are likely a result of higher Zn concentration of the soil (Table 4.1) and soil salinization (Figure 4.2). Total Zn concentration of the soil increased significantly between each treatment and linearly across the treatments. By the 60 Mg/ha treatments total Zn concentration in the soil is almost contaminated according to Alberta Tier One Guidelines for Contaminated Sites. Zinc uptake by plants is linear with respect to increasing soil concentrations when all other environmental factors remain constant (Thorseby, 1979). Pierzynski (1993) reports that plants grown in Zn contaminated soils decreased Zn tissue concentration following a liming treatment. This is supported by Zhan et al. (1994) showed that increasing soil pH with wood fly ash decreases the solubility of Zn. However, the total effect of increased sorbtion by increasing soil pH is likely overwhelmed or saturated by the additional Zn concentration and resulted in an increased tissue concentration.

The maximum concentration of Zn peaked in the seed component of the plant at 61.7 ppm. Feed quality guides from Alberta Agriculture (1992) and Jones et al. (1991) report that Zn concentration of barley is sufficient between 15 and 70 ppm. Therefore, the ash treatment improved the feed quality of barley with respect to Zn.

In the short term the higher ash treatments improved the feed quality of barley. However, as the buffering capacity of soil slowly returns the pH to levels less than 7, Zn will become more soluble (Zhan et al., 1996). With the reduction of soil pH it is expected that Zn uptake by plants would increase which may cause toxicity problems in extreme cases. Care must be taken to not overload the soil with Zn in order to prevent tissue increases of this magnitude. If ash is land applied at higher loading rates, a long-term management plan may be required to insure Zn does not increase to toxic levels. One possibility is to maintain neutral pH with Ag-lime, which does not add any additional metals to the soil.

5.7 Conclusion

The 20 Mg/ha loading rate had little impact on elemental uptake by barley plants. Boron and Cd levels were relatively unchanged from the control and only the grain significantly increased in Zn. The Zn increase had an agronomic benefit by increasing feed quality.

The 40 and 60 Mg/ha loading rate significantly increased tissue concentration of B, Cd and Zn and pushed the environmental envelope on soil and feed quality. The addition of ash increased tissue concentrations of B, Cd and Zn in barley. Boron concentration is high enough to cause toxicity effects at the highest loading rate, although no symptoms were seen. Cadmium concentrations increased slightly with increasing loading rates. Zinc concentrations increased significantly across the treatments and improved the feed quality of the barley from low-adequate to high-adequate. Part of the total elemental increase may be attributed to the high evapotranspiration rates of greenhouse as explained by Nable (1990) and the fact that roots are confined to the incorporation zone. However, wood ash can change soil environmental factors, which influence solubility of these elements.

Environmental factors like increasing elemental concentration and salinity can increase solubility and plant uptake potential of these elements. Wood ash is an effective soil-liming agent, which decreases plant availability of these elements. Soil pH being the dominant solubility-controlling factor, may delay plant uptake of heavy metals. Soil buffering capacity will lower soil pH over time, resulting in an increased soil concentration and increased plant availability and uptake.

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CHAPTER SIX

SUMMARY

6.1 Summary

Fly ash was added to two Alberta forest soils at ten different treatment levels in order to evaluate the growth response of hybrid poplar (*P. deltoidies* var. Assiniboine) and barley (*Hordeum Vulgare* var. Harrington) and monitor soil environmental factors that may contribute to bioaccumulation of heavy metals in barley. The objectives of the study were to: 1) determine an application rate that will enhance the growth of barley on two Alberta forest soils; 2) determine an application rate that will enhance the growth of hybrid poplar on two Alberta forest soils; 3) determine an environmentally safe application rate that will not significantly increase elements in the soil as regulated by contamination criteria; and 4) determine the uptake of elements of concern in agricultural plant tissue.

The Ap horizon of a Eluviated Dystric Brunisolic (E.DyB) and an Orthic Gray Luvisolic (O.GL) were collected from two agricultural fields near Grassland, Alberta. Fly ash from waste wood and bark of aspen (*P. tremuloidies*) was added and thoroughly mixed with ten treatment levels consisting of a control, 10, 15, 20, 40, 60, 80, 100, 120 and 200 Mg/ha equivalence. Four pots of each treatment and soil type were seeded with barley and four were planted with rooted poplar cuttings and randomly placed in a greenhouse. Poplar whips were cut after 70 days and measured for height and root collar diameter. Barley was harvested after 90 days, oven dried and weighed. Soil was sequentially sampled thoughout the 90 days from the control, 20, 40 and 60 Mg/ha treatments of the E.DyB soil and analyzed for Sodium Adsorption Ratio (SAR), Electrical Conductivity (EC), $pH_{(CaCl2)}$ and elements boron (B), cadmium (Cd) and zinc (Zn).

Height and root collar measurements revealed significantly increased growth of poplar grown in E.DyB soil in the 15 Mg/ha loading and poorer growth at higher treatments levels when compared to the control. There was no significant increase in poplar growth in the O.GL soil at any treatment level.

Barley significantly increased in above ground biomass in both soil types. In the E.DyB there was significant growth in all treatment levels compared to the control. The greatest increase was seen in the 100 Mg/ha treatments with a 50 % increase in above ground biomass. The O.GL soil also showed increased growth at all treatment levels as compared to the control. The greatest increase was seen in the 40 Mg/ha treatments with a 48 % increase over the control.

Soil analysis revealed SAR and Cd levels were not affected by ash applications up to 60 Mg/ha. Soil parameters EC, $pH_{(CaCl2)}$, boron (B)and zinc (Zn) increased incrementally with each incremental treatment. All treatments increased in EC over Alberta Tier One limits of 2 ds/m, creating saline soil conditions. The change in EC appears to be transient as the trend following the initial increase was in a downward direction, however, the EC did not return below Alberta Tier One limits after the 90-day experiment. Soil $pH_{(CaCl2)}$ also increased incrementally with ash additions. The 40 and 60 Mg/ha treatments temporarily increased pH over Alberta Tier One limits of pH 8.5, however, both returned below criteria limits within 5 and 20 days, respectively. Soil concentrations of B and Zn increased in all treatments but never over Alberta Tier One limits. Comparing soil concentration levels of B and Zn to micronutrient requirements recommended by Alberta Agriculture, both elements were elevated from moderately sufficient to adequate, which may have contributed to the increased growth of poplar and barley.

Results from this study confirm the findings of other studies with respect to elemental uptake of B, Cd and Zn following ash application. The 20 Mg/ha loading rate had little impact on elemental uptake by barley. Boron and Cd levels were relatively unchanged from the control and only the grain significantly increased in Zn. Higher treatments of 40 and 60 Mg/ha increased the tissue concentration of B, Cd and Zn. Neither element were in high enough concentration to cause toxicity symptoms in fact Zn was increased to highly sufficient nutrient states. However, as soil pH decreases heavy metals like Cd and Zn become more soluble and could increase in tissue concentration with time.

From the results of this greenhouse trial it can be concluded that application rates of 20 Mg/ha could be safely applied to soil without causing significant environmental impact to the soil and barley. Increased growth rates and nutrient quality of barley grown in ash treated soil could have significant agronomic benefits for landowners. Further research is required before wood ash should be marketed for large-scale applications.

6.2 Limitations to the greenhouse study

Limitations to this study are that it was a greenhouse study and the soil and plants are not interacting with the natural environment. Greenhouse studies can increase the concentration elements in plant tissue through increased exposure of the roots to wood ash in the incorporation zone as well as the higher evapotranspiration rates of the plants. The O.GL was atypical in that most luvisolic soil have pH lower than 6.2. Therefore, the growth estimates in this study, with respect to volume increases of poplar on, may not truly reflect the majority of O.GL.

6.3 Recommendations for Further Research

1. Loading rates suggested from this research need to be field-tested.
2. A tighter window of loading rates surrounding 20 Mg/ha should be field tested in order to determine maximum growth potential with minimal environmental risk for cereal crops.
3. More experiments need to be done on determining the effectiveness of ash for enhancing poplar growth.
4. More varieties of cereal, forages and oil seed crops should be tested in order to gain more information about the effects of fly ash on soil quality.
5. Longer-term field research should be done in order to confirm the risk of heavy metal uptake by agricultural crops with decreasing soil pH over time.
6. Field testing of ash loading rates need to be done in order to determine any benefit to Orthic Gray Luvisolic soils.

Appendix 1

Table A.1. Physical parameters and available nutrients of E.DyB (SE 5-69-19 W4th) and O.GL (SW36-68-19 W4th) and ash collected from Alberta-Pacific Forest Industries Inc. Alberta Tier One limits are also listed for controlled elements. Included are the elemental loading contributed by the ash at each incremental loading.

n=4	Physical Analysis							Available Nutrients													
	Bulk Density g/cm3	Texture	pH(CaCl2)	EC ds/cm	%Total Carbon	C/N Ratio	SAR	Ammonia-N	Nitrate-N	Aluminum	Phosphorus	Potassium	Calcium	Magnesium	Sodium	Copper	Iron	Manganese	Zinc	Boron	Sufate-Sulfur
Sample: Soil								ppm													
SE 5-69-19 W4th	1.0	S.loam	5.5	0.26	1.2	13	1	3	3.8	210	40	90	1150	100	7.5	0.6	45	40	3.2	0.2	1.8
SW 36-68-19 W4th	1.2	loam	7.3	0.46	2.6	15	1	5	4.5	205	33	90	2450	230	13	0.5	45	72	8.0	0.3	2.4
Ash n=25	0.4 8	27	13.3	70.0	13.6	320	0.025	4	28	1	3.0	38000	96000	380	3500	0.6	1.0	0.2	0.7	27	6359
Alberta Tier One			8.5	2			6													2.0	
								Additional Nutrients Added (kg/ha) With Each Treatment													
20 Mg/ha Eqiv.								0.0 8	0.56	0.02	0.06	760	1920	7.6	70	0.01 2	0.0 2	0.00 4	0.01 4	0.54	12.71
40 Mg/ha Eqiv.								0.1 6	1.12	0.04	1.02	1520	3840	15.2	140	0.02 4	0.0 4	0.00 8	0.02 8	1.08	25.44
60 Mg/ha Eqiv.								0.2 4	1.68	0.06	1.08	2280	5760	22.8	210	0.03 6	0.0 6	0.01 2	0.04 2	1.62	38.15

Table A.2. Total nutrients of E.DyB (SE 5-69-19 W4th) and O.GL (SW36-68-19 W4th) and ash collected from Alberta-Pacific Forest Industries Inc. Alberta Tier One limits are also listed for controlled elements. Included are the elemental loading contributed by the ash at each incremental loading.

N=4	**Total Nutrients**									
	Nitrogen	Phosphorus	Calcium	Magnesium	Iron	Sodium	Copper	Manganese	Zinc	Boron
Sample: Soil	%					ppm				
SE 5-69-19 W4th	0.09	453	0.30	0.20	1.3	71	6	469	45	13
SW 36-68-19 W4th	0.17	557	0.66	0.23	1.5	70	9	538	56	15
Ash n=25	0.03	0.60	21.00	1.82	0.17	3700	15	780	1760	148
Alberta Tier One							80		120	
	Additional Nutrients Added (kg/ha) With Each Treatment									
20 Mg/ha Eqiv.	6(E-6)	1.2(E-4)	0.42	0.027	3.4(E-5)	0.185	0.3	15.6	35.2	2.96
40 Mg/ha Eqiv.	1.2(E-5)	2.4(E-4)	0.84	0.055	6.8(E-5)	0.37	0.6	31.2	70.4	5.92
60 Mg/ha Eqiv.	1.8(E-5)	3.6(E-4)	1.26	0.082	1.02(E-4)	2.22	0.9	46.8	105.6	8.88

Table A.3. Total metals of E.DyB (SE 5-69-19 W4th) and O.GL (SW36-68-19 W4th) and ash collected from Alberta-Pacific Forest Industries Inc. Alberta Tier One limits are also listed for controlled elements. Included are the elemental loading contributed by the ash at each incremental loading.

	Total Metals															
	% Aluminum	Arsenic	Barium	Beryllium	Cadmium	Cobalt	Chromium	Lead	Mercury	Nickel	Selenium	Silver	Strontium	Tin	Titanium	Vanadium
		ppm														
SE 5-69-19 W4th	0.85	2.6	111		<0.25	6	13	5	0.019	16	<0.5	<2	11	<5	19	20
SW 36-68-19 W4th	0.90	2.8	161		<0.25	7	15	6	0.015	15	<0.5	<2	27	<5	17	21
Ash	0.22	<5	202	<1	11.3	4	<2	1	0.003	7	<10	<2	659	<5	23	6
Alberta Tier One		10	600	5	1	20	100	50	0.20	40	2					100
	Total Metals Added (kg/ha) With Each Treatment															
20 Mg/ha Eqiv.	4.4(E-5)	Na	4.04	Na	0.23	0.08	Na	0.02	6(E-5)	0.02	Na	Na	13.18	Na	0.46	0.12
40 Mg/ha Eqiv.	8.8(E-5)	Na	8.08	Na	0.46	0.16	Na	0.04	1.2(E-4)	0.04	Na	Na	26.36	Na	0.92	0.24
60 Mg/ha Eqiv.	1.32(E-4)	Na	12.12	Na	0.69	0.24	Na	0.06	1.8(E-4)	0.06	Na	Na	39.54	Na	1.38	0.36

Na = not applicable