

Responses of Red-Osier Dogwood to Oil Sands Tailings Treated with Gypsum or Alum

E. Redfield, C. Croser, J. J. Zwiazek,* M. D. MacKinnon, and C. Qualizza

ABSTRACT

The application of composite or consolidated tailings (CT) technology provides Alberta's oil sands industry with a means of reducing the volume of the fines fraction in extraction tailings and allows for faster reclamation and revegetation of mining sites. This study examined the effects of coagulant aids (gypsum and alum), used in the production of CT, on the ion content, growth, and survival of greenhouse-grown red-osier dogwood (*Cornus sericea* L. subsp. *sericea*). Seedlings were planted in gypsum-CT and alum-CT substrates, and compared with those planted in reclamation material (salvaged peat and till). The seedlings were bottom-watered with one of the following: (i) Hoagland mineral solution prepared in deionized water (Epstein, 1972); (ii) Hoagland solution in gypsum-based CT release water; or (iii) Hoagland solution in alum-based CT release water. Pore water of CT substrates and CT release waters had similar chemical characteristics, including salinity levels. However, plants in CT substrates had higher concentrations of ions (particularly Na and B), reduced growth, and higher mortality than plants in reclamation material and treated with CT waters. The presence of H₂S indicated low-oxygen conditions in the CT substrates, while in the reclamation materials with CT release water treatments, no evidence of sulfides was observed. Low-oxygen conditions in the CT substrate treatments may have interfered with plant exclusion mechanisms for Na and B. Therefore, substrate properties may modify responses of reclamation plants to pore water chemistry due to the effects on oxygen availability to roots.

EXTRACTION OF BITUMEN from oil sands produces large volumes of tailings that consist of solids (sand, silts, and clays), water (chemistry affected by ore, process aids, and water management), and unrecovered hydrocarbons. These materials must be reclaimed to develop sustainable aquatic or terrestrial habitats (Fine Tailings Fundamentals Consortium, 1995). Revegetation of extraction tailings is difficult due to the presence of elevated levels of salts and other potentially phytotoxic components, such as sodium, boron, and naphthenic acids (Renault et al., 2001; Apostol et al., 2002; Kama-luddin and Zwiazek, 2002). Compared with the coarse tailings sand fraction, fine tails pose more serious challenges to revegetation. Even after maturing in settling ponds, the resulting fine tails are saline aqueous suspensions (>80% water by volume) of sand, silt, clay, and residual bitumen, which settle and solidify slowly, and therefore require large geotechnically stable containment areas. To reduce the volume of this suspension, various tailings treatment options, such as consolidated

or composite tailings (CT), are currently being investigated.

In the CT process, the fines and sand fractions are treated with a coagulant aid to produce a nonsegregating mixture in which the fines are trapped with the sand fraction and low turbidity water is released (Matthews et al., 2000). Reclamation with CT would involve a process called dry capping (Fine Tailings Fundamentals Consortium, 1995). The CT deposit is covered with tailings sand, and the tailings sand is then capped with reclamation material (a mixture of salvaged peat and near-surface till materials) in which vegetation is established.

Two of the more successful coagulant aids that have been examined are gypsum (CaSO₄·2H₂O) and alum [Al₂(SO₄)₃·14.3 H₂O], both of which affect the release water composition (Matthews et al., 2000; MacKinnon et al., 2000). Gypsum is effective at dosages between 900 and 1200 g m⁻³. At these levels, increases in Ca²⁺, Na⁺, and SO₄²⁻ as well as decreases in alkalinity are observed. Alum (50% solution of aluminum sulfate) has been shown to perform comparably to gypsum at dosages of about 1000 g m⁻³. The lower required amount of alum reflects the role of the Al³⁺ ion versus the Ca²⁺ from the gypsum (MacKinnon et al., 2000). As a result, use of alum could reduce the salinity and pH of CT water compared with gypsum. However, as long as inorganic coagulant aids are used in the CT process, there will be impacts on the resulting produced waters, from both operational and reclamation aspects.

In the present study, we examined the relative performance of a reclamation plant species exposed to both kinds of CT waters and substrates. Previous studies (Renault et al., 1999, 2000, 2001) have found that the effects of CT release water and CT substrates on plants (reduced transpiration, chlorosis, stunted growth, necrosis, and mortality) vary widely between species. Red-osier dogwood (*Cornus sericea* L. subsp. *sericea*; also referred to as *Cornus stolonifera* Michx.) tolerates high salinity conditions relatively well compared with other reclamation species (Renault et al., 1999, 2001). Consequently, we used red-osier dogwood seedlings to compare their responses to alum and gypsum CT substrates and release waters.

In the present study, we compared seedlings grown for three months directly in gypsum CT, alum CT, or reclamation material. The plants were bottom-watered

E. Redfield, C. Croser, and J.J. Zwiazek, Dep. of Renewable Resources, 442 Earth Sciences Bldg., Univ. of Alberta, Edmonton, AB, Canada T6G 2E3. M.D. MacKinnon, Syncrude Canada Ltd., Edmonton Research Centre, 9421 17 Avenue, Edmonton, AB, Canada T6N 1H4. C. Qualizza, Environment Dep., Syncrude Canada Ltd., P.O. Bag 4009, M.D. 0078, Fort McMurray, AB, Canada T9H 3L1. Received 5 July 2002. *Corresponding author (janusz.zwiazek@ualberta.ca).

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Abbreviations: AA, alum consolidated tailings substrate treated with alum consolidated tailings release water; AH, alum consolidated tailings substrate treated with Hoagland solution; CT, consolidated tailings; EC, electrical conductivity; GG, gypsum consolidated tailings substrate treated with gypsum consolidated tailings release water; GH, gypsum consolidated tailings substrate treated with Hoagland solution; RA, reclamation material substrate treated with alum consolidated tailings release water; RG, reclamation material substrate treated with gypsum consolidated tailings release water; RH, reclamation material substrate treated with Hoagland solution.

with Hoagland solution made with water from one of the following three sources: deionized water, alum-based CT release water, or gypsum-based CT release water. These treatments mimicked the release and rise of water from CT as it solidifies, and avoided flushing salts from the system when adding water to compensate for evapotranspiration. This approach allowed a comparison of the effects of both the substrates and the release waters on ion accumulation, growth, and survival of the seedlings.

MATERIALS AND METHODS

Red-osier dogwood (*Cornus sericea* L. subsp. *sericea*) seedlings were grown for one year under greenhouse conditions from seed collected approximately 60 km north of Fort McMurray, Alberta (57°05.95' N, 111°38.90' W). At one year in age, each dogwood plant was transplanted into a 4-L pot containing one of the following substrates: (i) reclamation material, (ii) gypsum produced CT (1000 g m⁻³ of CT mix), or (iii) alum produced CT (1000 g m⁻³ of CT mix). Following planting, a 3-cm layer of peat moss was placed on top of the surface of all substrates to prevent desiccation and crusting of the CT deposit.

The plants were then assigned one of three treatment solutions: (i) Hoagland mineral nutrient solution made with deionized water (Epstein, 1972); (ii) Hoagland solution made with alum-based CT release water (for seedlings in alum CT); or (iii) Hoagland solution made with gypsum-based CT release water (for seedlings in gypsum CT). This produced a total of seven treatments, each applied in a plastic basin containing four pots, with three replicated basins, resulting in a total of 12 plants per treatment. The treatments were as follows:

- RH: Reclamation material supplied Hoagland solution with deionized water.
- RA: Reclamation material supplied Hoagland solution with alum CT water.
- RG: Reclamation material supplied Hoagland solution with gypsum CT water.
- AH: Alum CT supplied Hoagland solution with deionized water.
- AA: Alum CT supplied Hoagland solution with alum CT water.
- GH: Gypsum CT supplied Hoagland solution with deionized water.
- GG: Gypsum CT supplied Hoagland solution with gypsum CT water.

The plants were bottom-watered, and the solution levels in the basins were maintained at approximately 2-cm depths by topping them up with deionized water as needed. Ion and nutrient levels were maintained by replacing the solutions monthly. The treatments continued for 3 mo in the greenhouse under day–night temperatures of 25 and 18°C and a 16-h photoperiod supplemented by 400-W high pressure sodium lamps (Lumalux; GTE Sylvania, Drummondville, PQ, Canada).

Before and after the experiment, substrate samples were removed from several locations of randomly selected pots and analyzed for elemental contents with an inductively coupled plasma optical emission spectrometer (ICP–OES) (Vista-PRO RL; Varian Analytical Instruments, Victoria, Australia) on saturated paste extracts. Saturated paste extracts were also used to measure pH and electrical conductivity (EC). Plant survival and height were recorded weekly. At the end of the experiment, plants were removed from the substrates and washed in deionized water, and dry weights of roots and shoots

were determined. Ion concentrations in shoot and root samples were then measured using ICP–OES. The data were analyzed with a general linear model procedure by one-way analysis of variance (ANOVA) (SPSS, 1998). The means were compared with the Duncan’s multiple range test.

RESULTS

Substrate Properties

The CT substrates had higher EC and pH than the reclamation material at both the beginning and the end of the experiment. The initial electrical conductivities of the paste saturates ranged from 1.2 dS m⁻¹ for the reclamation material to 3.9 and 5 dS m⁻¹ for the alum and gypsum CT, respectively. Hoagland solution changed the average EC of treatments to 1.44 dS m⁻¹ (RH), 5.14 dS m⁻¹ (AH), and 4.96 dS m⁻¹ (GH). The addition of CT release waters to the substrates caused large increases in EC, with the reclamation material (RA and RG) displaying EC values of 4.35 and 7.67 dS m⁻¹, respectively, the alum CT (AA) increasing to 5.58 dS m⁻¹, and the gypsum CT (GG) increasing to 8.19 dS m⁻¹. The preexposure pH of the substrates ranged from 6.3 for the reclamation material to about 8.5 for both CT materials. After 3 mo of exposure to the treatment solutions, the pH of reclamation material in all treatments fell to 5.2 to 5.4, and in the CT substrates, the pH declined to about 6.0.

The concentrations of Na⁺, Cl⁻, and B were similar in alum and gypsum CT release waters used in treatment solutions (Table 1). However, gypsum CT release water had higher concentrations of SO₄²⁻, Mg²⁺, and Ca²⁺ than alum release water and slightly higher EC values (Table 1).

Substrates treated with the Hoagland solution (RH, AH, and GH) tended to have lower concentrations of potentially toxic ions (Na⁺, Cl⁻, SO₄²⁻, and B) than those treated with CT release waters (particularly gypsum CT water); however, differences were not statistically significant in every case (Fig. 1). Reclamation materials treated with alum CT (RA) and gypsum CT (RG) release waters had slightly elevated concentrations of Ca²⁺, Mg²⁺, Na⁺, Cl⁻, B, and SO₄²⁻ compared with those present in treatment RH (Fig. 1). These ion levels were comparable with those in the CT substrates treated with the Hoagland solution (AH and GH) (Fig. 1). Reclamation material treated with gypsum release water (RG)

Table 1. Representative ion concentrations and electrical conductivity (EC) of gypsum and alum consolidated tailings (CT) release waters used in treatment solutions.

Ion	Alum CT release water	Gypsum CT release water
	μg L ⁻¹	
B	4	3.6
Mg	10	30
Ca	10	40
SO ₄	625	1300
Cl	800	725
Na	975	1125
EC, dS m ⁻¹	4.1	5.0

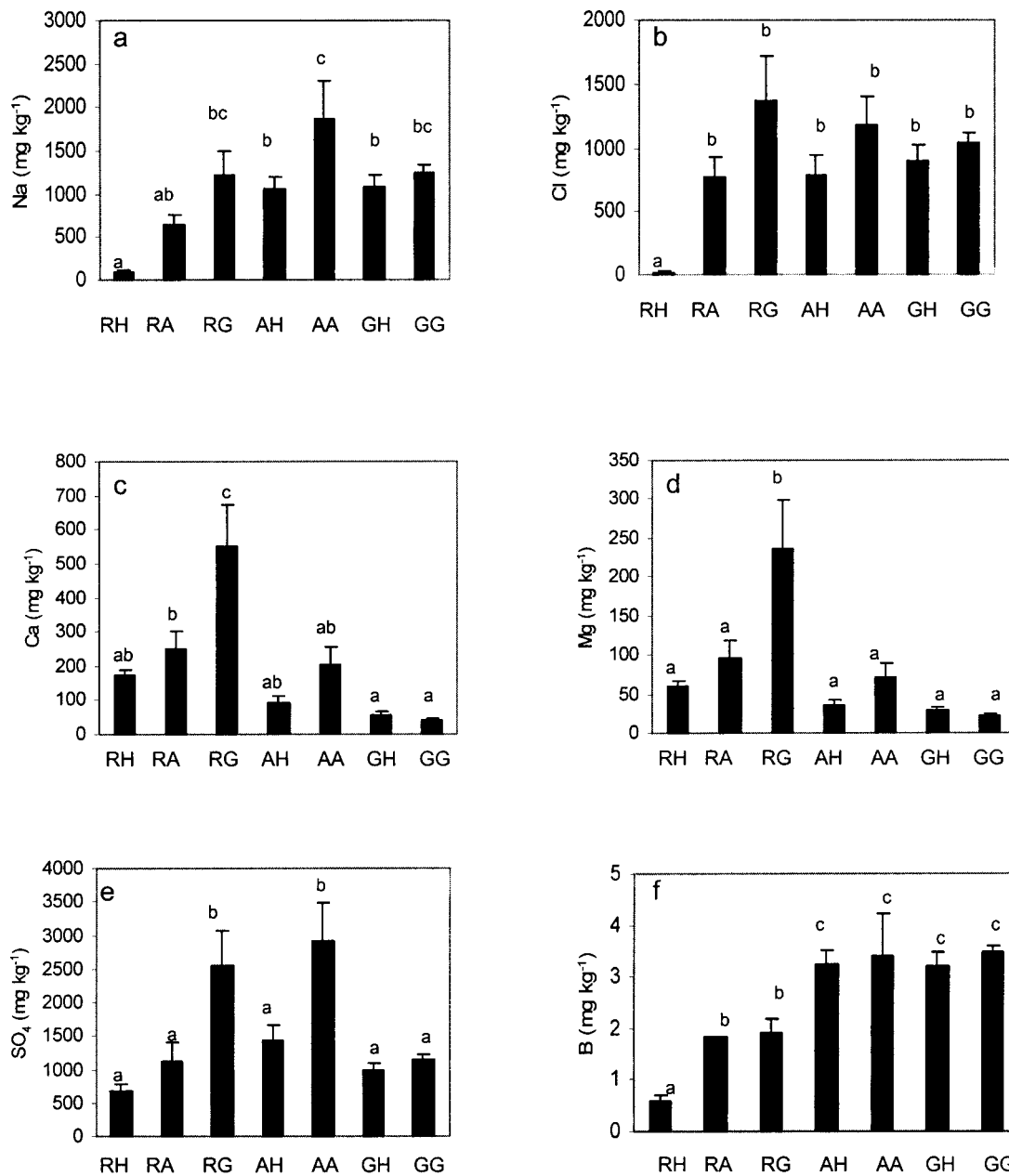


Fig. 1. Ion concentrations in saturated paste extracts after 3 mo of treatment. Different letters indicate statistically significant differences at $P \leq 0.05$. Means ($n = 5$) \pm standard errors are shown. RH, reclamation material supplied Hoagland solution with deionized water; RA, reclamation material supplied Hoagland solution with alum consolidated tailings (CT) water; RG, reclamation material supplied Hoagland solution with gypsum CT water; AH, alum CT supplied Hoagland solution with deionized water; AA, alum CT supplied Hoagland solution with alum CT water; GH, gypsum CT supplied Hoagland solution with deionized water; GG, gypsum CT supplied Hoagland solution with gypsum CT water.

had higher levels of Ca²⁺ and Mg²⁺ than those found in the gypsum (GG) CT substrate treated with the gypsum release water. Boron concentrations were elevated in all treatments (Fig. 1f). However, B was present in significantly higher concentrations in CT substrates than in the reclamation materials ($P \leq 0.001$).

Although oxygen levels were not measured, the strong H₂S odor of CT substrates, detected at the end of the experiments, indicated the presence of sulfate-reducing processes characteristic of low-oxygen conditions.

Seedling Survival and Shoot Length

There was a 100% survival rate for seedlings in the RH, RA, and RG treatments (Fig. 2a). In CT substrate treatments (AH, AA, GH, and GG), however, survival was significantly lower ($P \leq 0.001$) and ranged between 30 and 70% by the end of three months, depending on the treatment (Fig. 2a).

Shoot length of seedlings growing in reclamation material increased over the course of the experiment (Fig. 2b), with no significant differences between RH, RA, and RG. In the CT substrate treatments, there was no

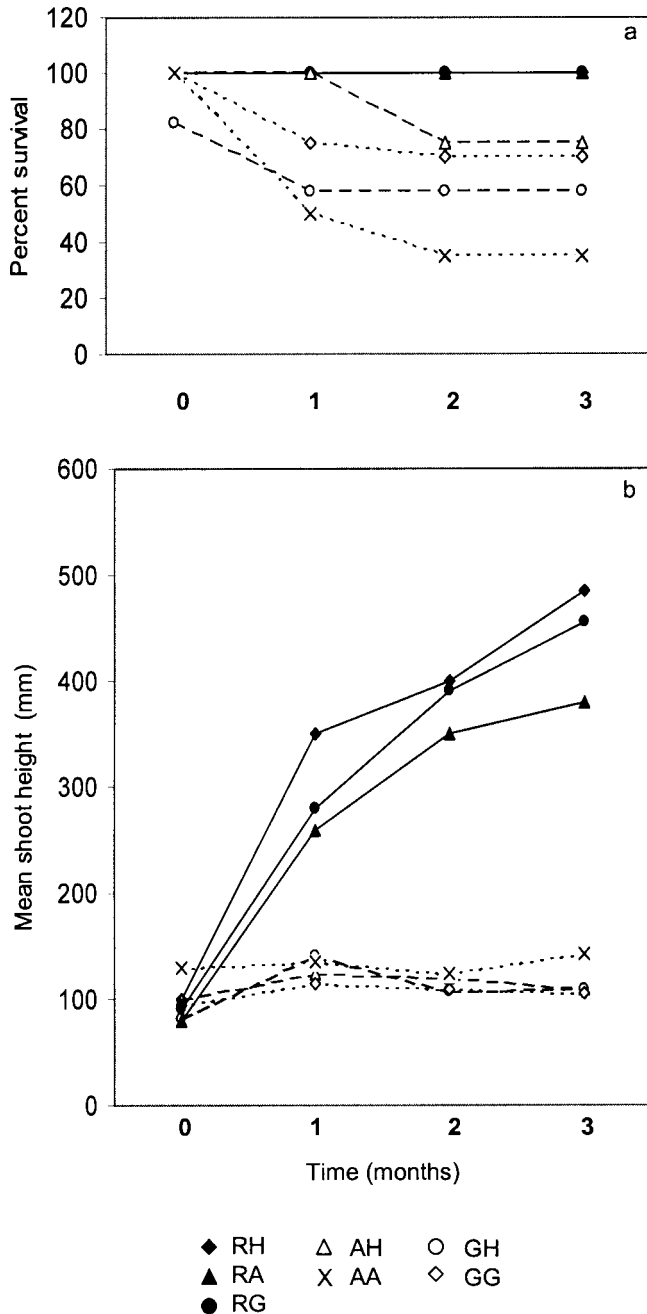


Fig. 2. Survival (a) and shoot height (b) in dogwood seedlings subjected to different treatments as explained below. Means ($n = 12$) are shown. Survival was significantly lower ($P \leq 0.001$) for all consolidated tailings (CT) substrate treatments. Treatment designations are explained in Fig. 1.

significant increase in shoot length over the course of the experiment (Fig. 2b). Growth fluctuations and small decreases in shoot length were due to leaf necrosis.

Dry Weight

The dry weight of RH seedlings was significantly greater ($P = 0.043$) than that of those grown in RA treatment, but not those in RG ($P = 0.924$). In all CT substrate treatments, total dry weights were significantly

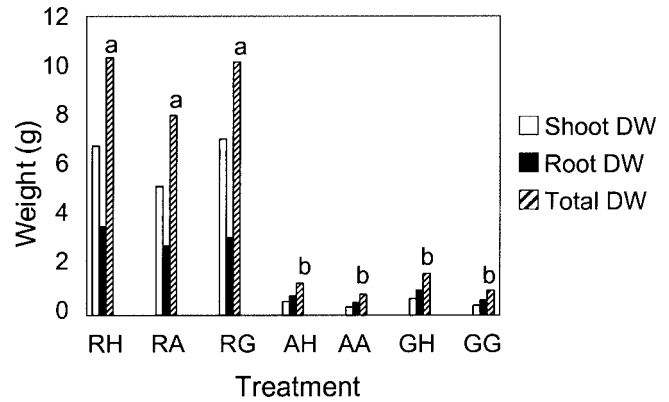


Fig. 3. Shoot, root, and total dry weight (in grams per plant) of dogwood seedlings after three months of treatments. Means ($n = 12$) are shown. Different letters indicate statistically significant differences at $P \leq 0.05$. Treatment designations are explained in Fig. 1.

lower ($P \leq 0.001$) than in the corresponding reclamation substrate treatments, with no differences between the CT substrates (Fig. 3).

Root to shoot ratio differed between plants grown in the reclamation material and CT substrates. In all the reclamation material treatments, shoot dry weight was consistently greater than root dry weight. However, in CT substrates the root dry weight was greater than the shoot dry weight (Fig. 3).

Plant Tissue Ion and Elemental Analysis

Plants grown in RA, RG, AA, and GG, had significantly elevated ($P \leq 0.001$) root Na^+ concentrations compared with the seedlings watered with Hoagland solution (RH, AH, GH) (Fig. 4a). The difference in Na^+ concentrations between roots and shoots was greatest in those plants grown in the reclamation material and treated with CT release waters (RA and RG) (Fig. 4a). In seedlings of all CT substrate treatments, no significant differences occurred between concentrations of Na^+ in roots and shoots (Fig. 4a)

Boron concentrations in RA and RG seedlings were similar to those in RH seedlings (Fig. 4b). A sharp increase was observed in plants of all treatments grown in the CT substrates with higher concentrations present in plants also treated with CT release water ($P \leq 0.001$). No significant differences occurred between alum and gypsum CT substrates (Fig. 4b). In all treatments, similar concentrations of B were present in shoots and roots (Fig. 4b).

Calcium concentrations showed an opposite trend to B (Fig. 4c). In all treatments, when the plants were grown in reclamation material (RH, RA, RG), Ca^{2+} concentrations in plant tissues were similar, with higher Ca^{2+} levels being present in the shoots than in the roots (Fig. 4c). Regardless of solution treatment, in plants grown in CT substrates (AH, AA, GH, GG), shoots and roots contained similar concentrations of Ca^{2+} . Shoot Ca^{2+} concentrations of CT-grown plants were lower ($P \leq 0.001$) than those growing in reclamation

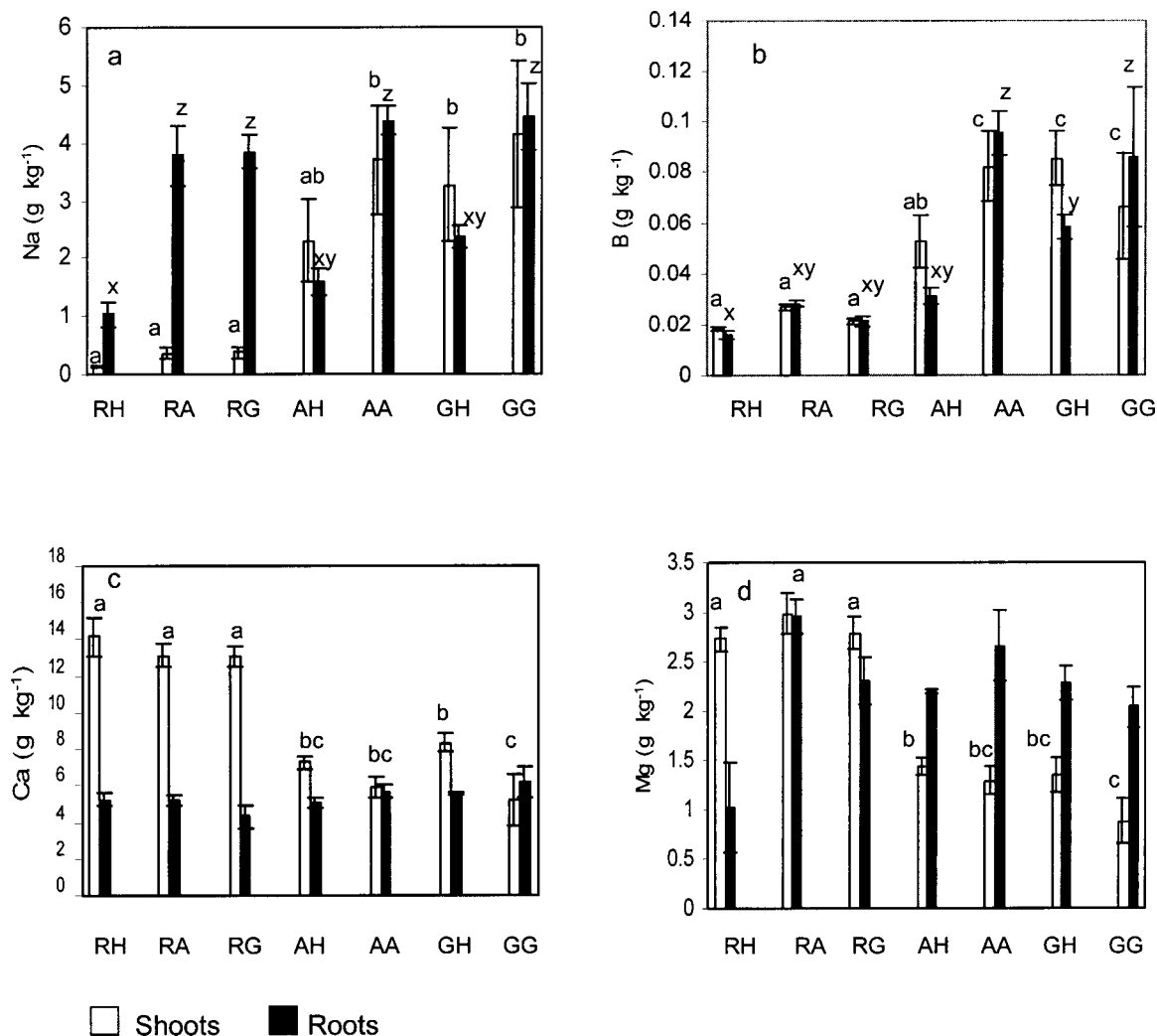


Fig. 4. Ion contents (on dry weight basis) in shoots and roots at the end of the experiment. Letters a, b, and c indicate differences among shoot tissue, while x, y, and z indicate differences among root tissues ($P \leq 0.05$). Means ($n = 5$) \pm standard errors are shown. Treatment designations are explained in Fig. 1.

material (Fig. 4c). There were no significant differences between all treatments in root Ca^{2+} concentrations ($P = 0.099$).

The Mg^{2+} content of plants in CT substrates was affected regardless of the type of substrate and release water to which these seedlings were exposed (Fig. 4d). Plants grown in the RH treatment had a ratio of shoot to root Mg^{2+} of about 2.5 to 1. When the plants were watered with alum and gypsum CT release waters (RA, RG), Mg^{2+} concentrations remained similar in the shoots, but the root Mg^{2+} concentrations increased almost threefold, becoming nearly 1 to 1 (Fig. 4d). In all CT substrate treatments, shoot Mg^{2+} concentrations were reduced and those in the roots remained high compared with the corresponding reclamation material treatments (Fig. 4d). No significant differences were found between treatments in root concentrations of Mg^{2+} ($P = 0.082$).

DISCUSSION

Our results suggest that there was little difference between alum- and gypsum-based CT, both in terms of

the effects of release waters and substrates on red-osier dogwood. The responses of plants to the different substrates and treatment solutions included increased tissue salt contents. However, there was not a simple direct relationship between ion concentrations in tissues and their concentrations in the pore (paste saturate) water or the treatment waters (CT release waters) to which the plants were exposed. In our study, shoots of the plants grown in reclamation material contained lower concentrations of Na^+ than those grown in the CT substrate, regardless of treatment solution (Fig. 4a). The elevated concentrations of Na^+ in shoots of plants in CT substrate treatments suggests that the ability of roots to store Na^+ was reduced compared with plants in reclamation material.

Salt concentrations vary between roots and shoots of plants subjected to salinity (Cheeseman, 1988). Prevention of Na^+ transport into the shoots is among the most effective salt resistance mechanisms used by plants (Greenway and Munns, 1980; Shannon et al., 1994; Renault et al., 2001). Sodium and Cl^- are toxic, and even in halophytes, which can survive high external levels

of these ions, and Na^+ and Cl^- are stored away from sensitive cytoplasmic enzymes and membranes (Jeffrey, 1987). It has been suggested that Na^+ is removed from the xylem at the top portion of the root system by xylem parenchyma cells (Khan and Ashraf, 1988), hence preventing Na^+ from entering the shoot. Sodium may then be transferred to the phloem and moved back into the roots (Greenway and Munns, 1980), where it may be released into the root medium by nonmetabolic means or ion exchange (Jacoby, 1979), or sequestered into woody tissues of the roots (Ziska et al., 1991). In salt-tolerant and halophytic species, Na^+/H^+ antiport systems are believed to exist in the plasmalemma and tonoplast, which drive Na^+ out of the cytoplasm and into the vacuole (Garbino and DuPont, 1989). In corn (*Zea mays* L.), Na^+ exclusion at the plasmalemma level of root cells has been indicated in salt-tolerant cultivars (Schubert and Lauchli, 1990). Hypoxic conditions have been reported to greatly reduce the Na^+ exclusion capabilities of corn (Drew and Lauchli, 1985) and wheat (*Triticum aestivum* L.) (Barnett-Lennard et al., 1999). By affecting metabolically controlled membrane transport processes and root integrity, hypoxia may increase the entry of Na^+ into roots and allow more Na^+ to move into the xylem apoplastically (Barnett-Lennard et al., 1999).

Since ion membrane transport is often energy-dependent, any reduction in oxygen available for respiration could seriously alter the plant metabolism and interfere with energy-requiring processes, such as ion pumping. Consequently, plants with roots growing in low-oxygen (hypoxic) conditions are likely to have a reduced capacity to exclude salts from their shoots. The presence of H_2S in the CT substrates suggests that such reducing, low-oxygen conditions were probably among the factors affecting plant responses to salt in our study. Therefore, differences in physical properties between the CT material and reclamation material, such as poor aeration due to low substrate porosity of CT, may have affected the efficiency of Na^+ transport and storage within the plants.

Accumulation of other ions from the studied substrates and treatment solutions as well as mineral deficiencies that may develop under saline conditions may have also contributed to the observed results. Nutrient imbalance resulting from depression in uptake and/or shoot transport and impaired internal distribution of Ca^{2+} and other essential nutrients is a major factor affecting plant growth in saline substrates (Marschner, 1995). Calcium plays an important role in maintaining membrane integrity and protects plants against salinity stress (Shannon et al., 1994). Consequently, the lower levels of Ca^{2+} found in tissues of plants grown in CT substrates compared with reclamation materials could be a significant factor that contributed to plant injury and growth inhibition.

The transport of Na^+ to the shoot that occurred in plants growing in alum and gypsum CT could be also affected by the presence of relatively high B concentrations in these substrates since B can interfere with the regulation of Na^+ (Apostol et al., 2002). Concentrations of B increased with increasing levels of B in the substrate

pore water. However, similarly to other studies, the concentrations of B were similar in shoots and roots (Wallace and Romney, 1977; Apostol et al., 2002).

At low levels, B is a micronutrient; however, B concentrations that are considered harmless or beneficial to some plant species are toxic to other plants (Huber, 1980). At high concentrations that are often associated with salinity, B is toxic, causing stem dieback, leaf necrosis, and disruption of chloroplast membranes (El-Motaium et al., 1994). The phytotoxic threshold for B in red-osier dogwood is not known, but tissue concentrations in CT substrate grown seedlings were comparable with B concentrations that cause injury in other vegetation (Gupta, 1984). Grattan et al. (1997) found that at moderate salinity (EC of 2–6 dS m^{-1}), similar to this experiment, plant sensitivity to B toxicity increased. Our ongoing studies examining the effects of hypoxia on plants exposed to salinity and boron will shed more light on plant responses to these combined factors.

In the present study, the RA and RG treatments closely paralleled actual operational reclamation conditions. Since in most areas seedlings will not be planted directly into CT, but into a cap of reclamation material and tailings sand placed over the CT deposit (Fine Tailings Fundamentals Consortium, 1995), most of the roots will likely not penetrate into the CT deposit. However, temporary hypoxic conditions in some reclamation areas are expected due to flooding and diffusion of methane from tailings into the root zone (MacKinnon et al., 2000).

Our results strongly suggest that the effects of either of the CT waters can be severely aggravated by the presence of low-oxygen conditions in the root zone. Therefore, the factors that produce low-soil-oxygen conditions, including low soil porosity and high water content, should be considered among the important factors that can profoundly affect oil sands revegetation efforts.

CONCLUSIONS

Concentrations of Na^+ and Cl^- in the pore waters of reclamation material substrates treated with CT waters were not very different from those in CT substrates (Table 1). However, this similarity in pore water chemistry was not reflected in final ion concentrations within plant tissues from the various treatments (Fig. 4). Consequently, it is unlikely that simple exposure to these ions was the primary cause of plant stress. In our study, regardless of treatment solution chemistry, the seedlings that were planted in reclamation material showed also greater survival and shoot growth compared with the plants grown in either the alum or gypsum CT substrate. Although many of the seedlings planted in CT substrates survived, their growth was almost completely inhibited.

We suggest that the hypoxia due to low porosity of the CT substrates was responsible for aggravating salt effects by affecting metabolic processes and ion exclusion mechanisms. The different CT solutions applied to the substrates and the different (gypsum vs. alum) CT formulations had no significant effect on most studied parameters, which further implies that substrate struc-

ture was a major factor in reducing plant growth and survival.

Our results suggest that red-osier dogwood should be considered for planting in well-aerated soils on reclamation areas that are affected by salinity. Therefore, the factors that affect soil oxygen levels, including the cap thickness, remain an important consideration in salt-affected areas.

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