

Article

Can Ammoniacal Nitrogen from Gold Mining Effluent Be a Promising Alternative for Fertilizing Boreal Forest Stands?

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Abstract: Nitrogen is an essential nutrient for plant functioning, photosynthesis, and metabolic activities. In terrestrial settings, nitrogen is not always sufficiently available because its basic form (N_2) must be fixed into other forms, such as nitrate and ammonium, to be usable by plants. Adding nitrogenous fertilizer to soils may provide a means of increasing forest productivity. Ammoniacal nitrogen ($N-NH_3$), an effluent produced during gold extraction, requires mining companies to manage its long-distance and costly transportation offsite for disposal. Applying this nitrogenous effluent, in its treated form of ammonium sulfate (ammoniacal nitrogen from mine water was converted into ammonium sulfate locally), to regional forest stands could provide a cost-effective and more environmentally sound means of managing this waste product and enhance forest productivity. Here, we conducted greenhouse- and field-based experiments to evaluate ammonium sulfate fertilization on black spruce (*Picea mariana*) and jack pine (*Pinus banksiana*) seedling growth. We assigned five treatments, varying in terms of the fertilizer concentration and presence/absence of biochar, to seedlings in greenhouse trials. We also applied various concentrations of ammonium sulfate to an 8-year-old black spruce plantation in Abitibi-Témiscamingue, Québec. We found that black spruce and jack pine seedlings experienced greater growth than the controls in terms of the stem diameter (32–44%), seedling height (21–49%), and biomass (86–154%). In the field experiment, we observed 37% greater volumetric growth in plots receiving medium-level fertilization than the control. Although nitrogen fertilization lowered the soil pH, essential nutrients increased to favor greater seedling growth. Thus, ammonium sulfate, derived from local mining effluent, appears to offer a suitable alternative for enriching nitrogen-limited boreal soils and increasing tree growth. This application could benefit both regional mining industries and forest management bodies.

Keywords: ammonium sulfate; biochar; boreal forest; productivity; sustainable forest management



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1. Introduction

Nitrogen is an essential nutrient for ensuring proper plant function and metabolic processes [1], yet its limited availability commonly restricts the growth and productivity of trees in forest ecosystems [2–4]. Synthetic nitrogen provides a non-natural nutrient source for plants. For instance, ammonium sulfate is recognized as a common and inexpensive nitrogen source for agriculture [5], having certain agronomic and environmental advantages over other nitrogenous fertilizers, e.g., ammonium nitrate. These advantages include no loss through volatilization in the form of NH_3 , reduced leaching of NO_3-N , less

denitrification, and lower emissions of greenhouse gases (NO, N₂O) [6]. Although applying such nitrogenous compounds can reduce the soil pH [7], this fertilization can, nevertheless, enhance the growth and productivity of boreal forest stands and, thus, their commercial value [8–10]. Identifying potential nitrogenous sources and their transformation into fertilizer can serve both agricultural and forestry needs.

Ammoniacal nitrogen (N-NH₃) is often present as a pollutant in mining operations. The most important sources of N-NH₃ are leachates from rock that have been in contact with explosives during periods of blasting [11,12]. N-NH₃ is a component of ammonium nitrate-based (NH₄NO₃) explosives commonly used in mining activities [13]. It can also be found in mine water through the degradation of cyanides, which produce nitrogen derivatives, including ammonia, nitrites, and nitrates [14]. These effluents are harmful to the environment, as they cause eutrophication, acidify lakes, and are toxic to many aquatic taxa; for example, concentrations of less than 1 mg/L of ammoniacal effluent can be lethal to fish [15,16]. Therefore, it is imperative to manage these effluents and transform them into innocuous forms. The removal of N-NH₃ from mine water is based on several physical (e.g., stripping, ion exchange, membrane separation) and/or biological methods (e.g., microbial processes); however, these techniques are expensive and time consuming [14]. Therefore, mining companies treat the effluent (particularly ammonia) with sulfuric acid (H₂SO₄), given its wide availability and relatively low cost. However, the long-distance transportation costs to dispose of the generated byproduct, ammonium sulfate, have made this alternative treatment somewhat questionable [17]. Therefore, suggestions have been put forward to investigate the benefits of using ammonium solution as a fertilizer for local forest plantation sites. Accordingly, it was found that an estimated 438 tons of N-NH₃ generated per year (from only one source of mining effluent coming from a gold mining company operated in the Abitibi-Témiscamingue region) could serve to fertilize at least 2190 ha of forest plantations to improve soil nutrient levels in the Canadian boreal forest [18].

A new framework has emerged to apply climate change-adapted ecosystem-based forest management to promote forest resilience [19,20]. Maintaining productivity will be a major challenge for boreal forests under future climate scenarios [21,22]. Enhancing the available nutrient levels of forest soils requires prioritizing soil fertility through natural means, such as branch cuttings, letting plant debris accumulate on the forest floor, and maintaining residual vegetation. Issues related to soil nutrition are often encountered after harvesting [23]; for example, the profitability of plantations in relation to soil quality has been an issue for intensive silviculture [24]. Although numerous strategies have been applied to ensure sustainable forestry, limited soil fertility and nutrient availability can be an issue for optimal forest growth [23,25]. In such cases of impoverished soil, fertilization could help enhance overall productivity and achieve profitability objectives in plantations and natural forests [18]. Such fertilization also benefits coniferous species, such as spruce and pine, in terms of their growth. For example, Pukkala [10] observed 15% and 5% higher timber yields for fertilized spruce and pine stands, respectively, than the nonfertilized controls. In addition, the diversity and abundance of understory vegetation also increase with fertilization [9]. Thus, forest growth can be modulated by the nitrogen content of the soil, which can be affected by several anthropogenic and natural disturbances, including fire, insects, diseases, flooding, and silvicultural practices [26,27].

Adding substrates, such as biochar, with nitrogen fertilizer can enhance the effectiveness of fertilization to improve the overall soil condition and forest productivity [17,28]. Biochar is a carbon material obtained by heating plant biomass or biomass residue under low-oxygen concentrations [29]. It is a strong carbon sequester with a high adsorptive quality and is frequently used for amending soils [30]. While using biochar as a nitrogen carrier could reduce the polluting effects of fertilizer, it can also retain nutrients through electrostatic adsorption and the retention of dissolved nutrients in water [17,29]. This strong nutrient retention relates to biochar's large contact surface, high abundance of surface functional groups, and high porosity [31,32]. The cation exchange capacity (CEC)

of biochar increases markedly once exposed to oxygen and water, leading to the oxidation of functional groups on its surface [32]. The liming effect of biochar can optimize soil fertility [33,34]. Biochar also increases the availability of nutrients, such as N, K, Ca, Mg, P, and S, which are essential for plant growth, and improves microbial activity [35,36]. In the Swedish boreal forest, the biomass of *Pinus sylvestris* trees increased between 20% and 30% after adding biochar to the forest soil [37,38].

Even if ammoniacal nitrogen is a harmful pollutant with a high environmental impact, there are limited studies on whether it can be recovered and transformed into a versatile resource to enhance tree growth and productivity. Our study aims to evaluate the impact of ammonium sulfate recovered from mining effluent on the growth of black spruce (*Picea mariana* (Mill.) BSP) and jack pine (*Pinus banksiana* Lamb.) seedlings. Specifically, we will monitor the changes in the biometric variables, i.e., the diameter, height, biomass, foliar content, and soil nutrients, after the application of fertilizer and identify the suitable amount of fertilizer to be applied for optimum growth in the boreal forest, along with the addition of biochar. Seedlings are tested in both field and laboratory settings, using ammonium sulfate or ammonium sulfate combined with biochar. We hypothesize that adding ammonium sulfate will increase seedling performance and the use of biochar will optimize the nitrogen uptake ability to heighten the overall growth and productivity of black spruce and jack pine. We also sought to determine whether ammonium sulfate provides a beneficial solution for mining and forestry needs. The incorporation of this nitrogen source into forest management practices could improve the growth and productivity of tree plantations, thereby supporting commercial forestry operations and contributing to the overall health and resilience of forest ecosystems. Ammoniacal nitrogen, particularly in the form of ammonium sulfate, will provide agronomic advantages; for example, it can cause reduced nitrogen loss through volatilization and leaching, which results in more efficient nutrient uptake by plants [6]. Moreover, the use of ammoniacal nitrogen also aligns with circular economy principles, as it transforms waste into a resource, reducing the need for synthetic fertilizers and lowering the carbon footprint associated with their production and transportation [17,39].

2. Materials and Methods

2.1. Materials

White birch residues collected from the Abitibi-Témiscamingue region, Québec, were milled (<6 mm) and converted into biochar through fast pyrolysis CarbonFX technology (Airex Energy Inc., Bécancour, QC, Canada), at 425 °C, in a low-oxygen environment, at the Centre Technologique des Résidus Industriels (CTRI) laboratory (Rouyn-Noranda, QC, Canada). Details related to the process and furnace conditions are presented in the work by Braghiroli et al. [40]. The stripping technique for separating ammoniacal nitrogen from mine water involves volatilizing the water to release ammonia gas, which occurs at specific pH and temperature (40–80 °C) conditions in terms of the wastewater [41]. By increasing the pH (up to 8.5) through the addition of lime, the chemical equilibrium is shifted to the right, which favors the formation of ammonia gas [42]. The released ammonia is then captured using an acid trap, which converts the volatile NH_3 into NH_4^+ . Finally, the ammonia is introduced into an air stream and, subsequently, absorbed by washing with sulfuric acid, which results in the production of ammonium sulfate. This conversion process effectively transforms the ammoniacal nitrogen from mine water into ammonium sulfate, without a significant impact on the environment. For our experiment, the prepared ammonium sulfate salt solution (20%) was provided by Valorex (Warwick, QC, Canada).

2.2. Greenhouse Trials

For the greenhouse experiments, we used 25 black spruce and 25 jack pine seedlings provided by the Trécesson nursery (Pépinière forestière de Trécesson, Amos, QC, Canada). We selected black spruce and jack pine because of their wide distribution in the boreal region and the commercial benefits of these species in the forestry sector. We followed

the work by Boivin [43] to ensure that the selected seedlings were as similar as possible in terms of their size and color. Each seedling was then transplanted into a container with a diameter of 25 cm that was filled with soil, approximately 17 cm from the rim. The potting soil was recovered from a spruce stand on an organic deposit in the municipality of Beaudry (Centre Jardin Lac Pelletier, Rouyn-Noranda, QC, Canada) and was air dried and sieved with a 2 mm mesh, before being placed in the containers. The bottom of the container was lined with geotextile fabric to prevent soil loss during watering. The greenhouse conditions (temperature, humidity, and light availability) were controlled and we monitored the growth every 10th, 20th, and 30th week of fertilization. For each species, we assigned five treatments with five replicates, considering the optimal recommended nitrogen doses according to Pukkala [10], for each species (Figure 1). The treatments were as follows:

1. N0B0: control, without fertilization;
2. N0B4: 12.8 g of anhydrous biochar (corresponding to $4090 \text{ kg} \cdot \text{ha}^{-1}$; according to Hart and Luckai [44], this represents the maximum concentration of biochar in the natural boreal forest in North America). The biochar used was chemically composed of C (75.4%), H (3.5%), N (0.9%), S (0.5%), and O (19.7%);
3. N2B0: 14.4 mL of 21% ammonium sulfate solution (corresponding to $200 \text{ kg N} \cdot \text{ha}^{-1}$);
4. N4B0: 28.8 mL of 21% ammonium sulfate solution (corresponding to $400 \text{ kg N} \cdot \text{ha}^{-1}$);
5. N2B4: 12.8 g of biochar and 14.4 mL of 21% ammonium sulfate solution.

For each plant, a treatment was randomly assigned. The biochar was incorporated into the first few centimeters of the soil [45] and the ammonium sulfate solution was applied using a syringe to achieve optimum evenness of the fertilizer on the soil.



Figure 1. Illustration of a treatment allocation for 25 jack pine and 25 black spruce seedlings in the greenhouse at the CÉGEP de l’Abitibi-Témiscamingue (Rouyn-Noranda, Québec).

2.2.1. Plant Monitoring

We measured the height and collar diameter of each seedling before fertilization. To ensure better accuracy, we used a digital Vernier caliper to make two collar diameter measurements perpendicular to each other. We repeated these measurements at the 10th, 20th, and 30th weeks after fertilization. The plant vases containing seedlings were watered every week to the full capacity [46]. We placed individual trivets under each plant to minimize fertilizer loss through drainage and returned the drained water and sediments to the container [47]. The reapplication of the drained sedimental water ensured no loss of fertilizer, while also keeping the soil as humid as possible. Once a week, the seedlings were rotated in the greenhouse to avoid any location-specific effects on plant growth [43] and to prevent uneven light [48]. Because of excessively hot conditions in the greenhouse during early summer, we placed the seedlings outside the greenhouse for the last ten

weeks of the experiment. After the experimental period, we cut the seedlings at the collar and dried them in an oven at 70 °C for 48 h. The needles and branches were weighed separately to obtain the measurements of the dry biomass at the end of the experiment. The needle samples were then crushed for foliar analyses of the nitrogen, phosphate, and potassium contents.

2.2.2. Soil Measurements

We recorded the pH, at the end of the experiment, of the soil in each container to determine whether ammonium sulfate had acidified the soil (Intellical™ pH PHC 101, Rouyn-Noranda, QC, Canada). The distilled water extraction method was used for the measurement of the pH and conductivity of the soil. The sample weight was brought in contact with an identical weight of water (1:1 ratio). The solution was shaken for 5 min at room temperature; the solid portion was left to settle and, then, the pH of the liquid portion was finally measured. Eight weeks after fertilization, plant root simulators (PRS™, Western Ag Innovations Inc., Saskatoon, SK, Canada) were installed in three replicates in the controls (N0B0) and the nitrogen-fertilized vases (N2B0 and N4B0), a total of nine samples were measured for one week [49].

The total Kjeldahl nitrogen (TKN) and total phosphorus in the soil were measured using a BUCHI K-435 digestion unit (BUCHI Corporation, New Castle, DE, USA) and an ammonia selective electrode, according to the recommendations from the Québec Center of Expertise in Environmental Analysis (CEAEQ) [50]. Similarly, the concentration of heavy metals in the leachate and soil was measured using microwave plasma atomic emission spectroscopy (MP-AES 4200, Agilent 183 Technologies, Mississauga, ON, Canada), according to the CEAEQ's recommendations [51].

2.3. Field Plantation Experiment

2.3.1. Study Area and Experiment Setup

Our field plots were located in the Abitibi-Témiscamingue region (QC), Canada, with latitudinal and longitudinal coordinates ranging from 28.253100° N to 28.259314° N and 78.693998° W to 78.712780° W, respectively. The regional geology supports diversified mining activities, contributing significantly to the regional and provincial economy. The site is located within the clay belt of the boreal region, dominated primarily by lacustrine deposits of organic and mineral soil. The site is characterized by a very fine texture, ranging from sandy clay loam to loamy sand, that has a high water-holding capacity [52]. We used regional forest maps to locate a recovered black spruce slash-and-burn plantation dating back to 2007 (Figure 2). We selected this eight-year-old plantation to avoid the site adaptation period of the plants, which can last at least four to five years.

We defined three 30 × 50 m blocks, aligned in an east–west orientation, located to consider the watercourses, ecotones, and the edge effect of adjacent stands; we left a minimum inter-block spacing equivalent to the height of the trees. Each block was subdivided into three 15 × 30 m experimental plots and a 2 m border was delimited between each plot to prevent treatment overlap. To control the variability in the site conditions, such as soil texture and moisture, we ensured that all blocks were located on relatively homogeneous terrain within the plantation zone. In addition, the treatment blocks were not distantly located, which limited the possibility of high variability in the climatic factors, like temperature and precipitation. Treatments were, then, randomly assigned to the experimental (rectangular) plots within each block: (i) N0 was the control, without fertilization; (ii) N1.5 was fertilization equivalent to 150 kg N·ha⁻¹; and (iii) N3 was fertilization equivalent to 300 kg N·ha⁻¹. The ammonium sulfate solution (20% volume) was applied manually at the beginning of summer and as evenly as possible, with a sprayer using garden watering cans (avoiding spraying the plant foliage).

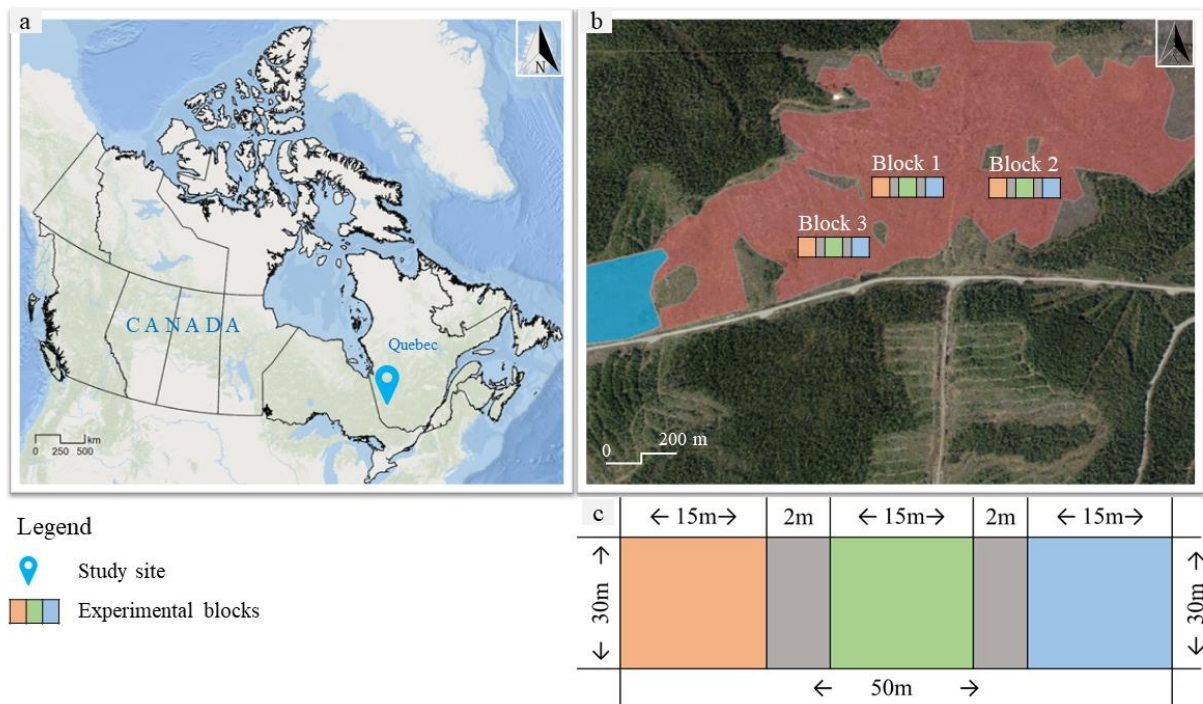


Figure 2. (a) Location of the study site in the Abitibi-Témiscamingue region, Québec, Canada; (b) location of three blocks in a pre-established black spruce plantation site showing the experimental plots; (c) magnified illustration of an experimental block, with the dimensions of each plot.

2.3.2. Measurements and Monitoring

All dendrometric measurements in the field were taken from 25 plants per experimental plot, like the seedlings in the greenhouse. Sampling always began in the southwestern corner of the plot. We sampled needles from the seedlings in the spring from 2015 to 2020 (collected around the same date each year) for foliar analyses.

In the first year (2015), we collected soil samples before and after the fertilization experiment, i.e., one time in spring 2015 and the other time at the onset of autumn 2015 (at the end of the growing season). From 2016 to 2020, the soil was sampled during the spring. With the use of a manual soil auger, a soil sample was collected from the organic and mineral horizons (0–15 cm) from two locations in each plot, within the first and second thirds along a southwest–northeast transect, i.e., at approximately 11 m and 22 m from the sampling site in the southwestern corner. The collected soil samples were air dried, the mineral soil was sieved with a 2 mm sieve, and then transferred for pH, conductivity, and metal content measurements (using the same methods used in the greenhouse trials). Six weeks after the fertilization treatments were applied, we installed PRS probes, in four sections in terms of the N0 and N3 treatments with three replicates, for two weeks to determine the ionic concentrations in the organic and mineral layers [49]. These probes, installed in the field and greenhouse settings, measured the anions and cations available to the seedlings.

2.4. Statistical Analyses

The effects of various treatments on biometric variables, such as the diameter, height, biomass, foliar content, and soil nutrients, were analyzed using a one-way analysis of variance (ANOVA) test. The ANOVA was conducted using the `aov` function in R, with treatment as the main factor. Following the ANOVA, Tukey's honestly significant difference (HSD) post hoc test was performed to identify specific treatment pairs with statistically significant differences [53]. To further explore the differences among the treatment groups, we employed the `emmeans` package, which allowed us to visualize, compare, and identify

treatment pairs with significant differences at a 95% confidence level ($\alpha = 0.05$). For the analysis of the plantation seedlings, we implemented a linear mixed-effects model (LMM) using the lme4 package [54]. In this model, the fixed effects were the treatment and height of the seedlings, while the tree and year were treated as random effects to account for repeated measures and potential temporal variability. This approach allowed us to model the dependency within the data, due to repeated measurements on the same trees over time. To ensure the robustness of our models, we conducted several diagnostic checks. We visually inspected residual plots to assess the assumptions of normality and homoscedasticity. The normality of the residuals and random effects was further confirmed using the Shapiro–Wilk test. Additionally, we checked for the absence of any discernible patterns in residual plots, ensuring that there were no violations of the model assumptions, such as trends in the residuals or heteroscedasticity. All the statistical analyses were performed in R version 4.2.0 [55].

3. Results

3.1. Greenhouse

3.1.1. Growth

Ammonium sulfate enhanced the growth of spruce and pine seedlings in terms of the diameter and height in the greenhouse experiments (Table S1). The increase in the height of the black spruce seedlings was significantly greater than that of jack pine seedlings, reaching more than double that of jack pine at the 30th week (Figure 3); however, at the beginning of the experiment, those seedlings had comparable heights. Throughout the experiment, seedlings from the three treatments (N2B0, N2B4, and N4B0) experienced a mean 9.5 cm height increase every 10 weeks (Table 1). This growth was up to 32% greater than the treatments without nitrogen supplements (N0B0 and N0B4 showed an average height gain of 4.2 cm per 10 weeks). At the 30th week, the height growth was significantly different between the seedlings that received nitrogen and those that did not. At the end of the greenhouse experiment, the nitrogen-treated pine seedlings were 21% taller (height difference from 24.7 to 41.4 cm) than the seedlings that received no nitrogen (height increase from 24.7 to 34.4 cm).

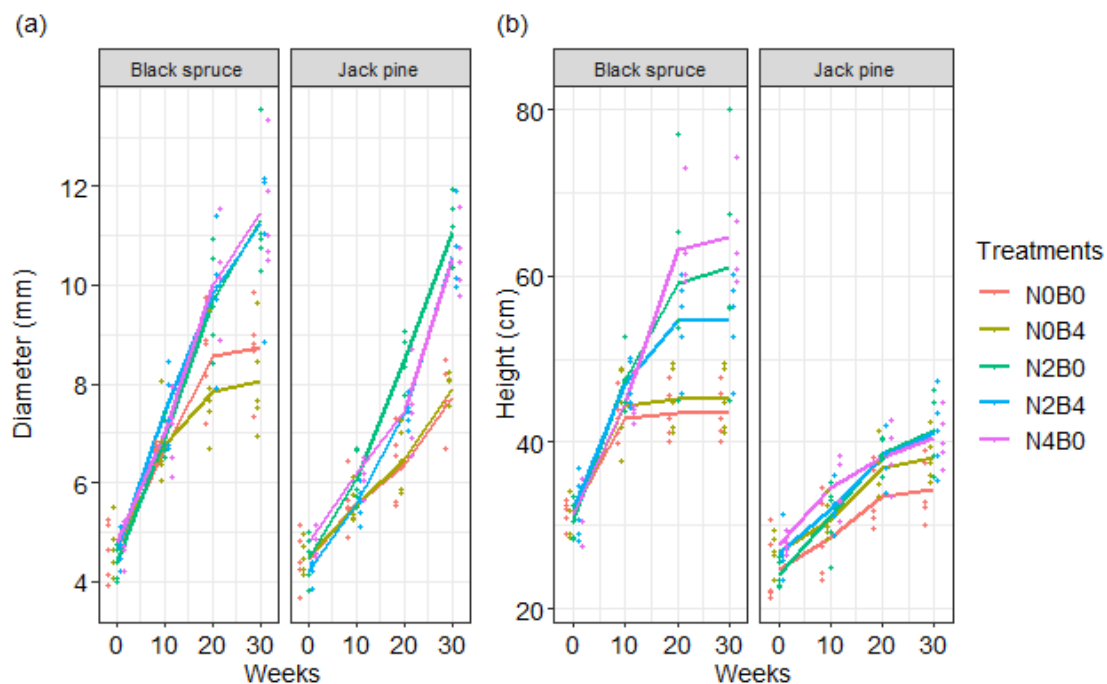


Figure 3. (a) Diameter; (b) height growth in relation to the nitrogen treatments received during greenhouse experiments. N0B0: control; N0B4: 12.8 g of anhydrous biochar; N2B0: 200 kg N·ha⁻¹; N2B4: 200 kg N·ha⁻¹ and 12.8 g of anhydrous biochar; N4B0: 400 kg N·ha⁻¹.

Table 1. Average change in growth and biomass of spruce and pine seedlings after the application of ammonium sulfate relative to the untreated seedlings. N0B0: control; N0B4: 12.8 g of anhydrous biochar; N2B0: 200 kg N·ha⁻¹; N2B4: 200 kg N·ha⁻¹ and 12.8 g of anhydrous biochar; N4B0: 400 kg N·ha⁻¹.

| Species | Week | Treatment | Diameter (mm) | Height (cm) | Biomass (g) | | Change (%) Compared to Control in | | |
|--------------|------|-----------|---------------|-------------|-------------|--------|-----------------------------------|--------|---------|
| | | | | | Needle | Branch | Diameter | Height | Biomass |
| Black spruce | 0 | N0B0 | 4.62 | 31.5 | - | - | - | - | - |
| | 10 | N0B0 | 6.66 | 42.98 | - | - | - | - | - |
| | 10 | N0B4 | 6.75 | 44.40 | - | - | 1.35 | 3.30 | - |
| | 10 | N2B0 | 6.91 | 47.28 | - | - | 3.75 | 10.00 | - |
| | 10 | N2B4 | 7.43 | 46.92 | - | - | 11.56 | 9.17 | - |
| | 10 | N4B0 | 7.01 | 44.52 | - | - | 5.26 | 3.58 | - |
| | 20 | N0B0 | 8.56 | 43.50 | - | - | - | - | - |
| | 20 | N0B4 | 7.84 | 45.20 | - | - | -8.41 | 3.91 | - |
| | 20 | N2B0 | 9.69 | 59.16 | - | - | 13.20 | 36.00 | - |
| | 20 | N2B4 | 9.83 | 54.66 | - | - | 14.84 | 25.66 | - |
| | 20 | N4B0 | 9.99 | 63.08 | - | - | 16.71 | 45.01 | - |
| | 30 | N0B0 | 8.73 | 43.58 | 6.23 | 8.26 | - | - | - |
| | 30 | N0B4 | 8.04 | 45.26 | 5.56 | 7.95 | -7.90 | 3.85 | -10.80 |
| | 30 | N2B0 | 11.32 | 61.02 | 11.60 | 18.57 | 29.67 | 40.02 | 86.06 |
| | 30 | N2B4 | 11.25 | 54.68 | 10.81 | 15.77 | 28.87 | 25.47 | 73.39 |
| 30 | N4B0 | 11.49 | 64.76 | 11.48 | 17.78 | 31.62 | 48.60 | 84.12 | |
| Jack pine | 0 | N0B0 | 4.45 | 24.74 | - | - | - | - | - |
| | 10 | N0B0 | 5.59 | 28.46 | - | - | - | - | - |
| | 10 | N0B4 | 5.55 | 30.80 | - | - | -0.72 | 8.22 | - |
| | 10 | N2B0 | 6.08 | 31.16 | - | - | 8.77 | 9.49 | - |
| | 10 | N2B4 | 5.59 | 32.04 | - | - | 0.00 | 12.58 | - |
| | 10 | N4B0 | 6.17 | 34.44 | - | - | 10.38 | 21.01 | - |
| | 20 | N0B0 | 6.35 | 33.46 | - | - | - | - | - |
| | 20 | N0B4 | 6.47 | 36.94 | - | - | 1.89 | 10.40 | - |
| | 20 | N2B0 | 8.49 | 38.56 | - | - | 33.70 | 15.24 | - |
| | 20 | N2B4 | 7.42 | 38.10 | - | - | 16.85 | 13.87 | - |
| | 20 | N4B0 | 7.43 | 38.26 | - | - | 17.01 | 14.35 | - |
| | 30 | N0B0 | 7.72 | 34.36 | 9.32 | 4.64 | - | - | - |
| | 30 | N0B4 | 7.90 | 38.10 | 9.62 | 4.75 | 2.33 | 10.88 | 3.21 |
| | 30 | N2B0 | 11.08 | 41.40 | 23.69 | 10.97 | 43.52 | 20.49 | 154.19 |
| | 30 | N2B4 | 10.59 | 41.20 | 21.68 | 9.76 | 37.18 | 19.91 | 132.58 |
| 30 | N4B0 | 10.54 | 40.44 | 19.98 | 9.35 | 36.53 | 17.69 | 114.37 | |

Although height growth exhibited a species-specific pattern, the collar diameter exhibited somewhat similar trends for both species. After the 30th week, for both species, we observed a significant difference between the diameter growth of the seedlings that received ammonium sulfate and those that did not (Figure S1). At the final sampling, the black spruce seedlings that received a high nitrogen dose (N4B0) increased in diameter by 6.7 cm on average (from 4.6 cm to 11.5 cm), whereas the mean diameter of the N0B0 seedlings increased by only 4.1 cm. For the jack pine seedlings, seedlings grown with biochar alone (N0B4) grew slightly taller than the controls; although, we did not observe a significant effect on the diameter growth for biochar alone. The combination of nitrogen and biochar increased the diameter growth, i.e., the seedlings attained an 11 cm diameter from an initial 4.5 cm. We observed a slight slowing in the growth of the jack pine diameter after the initial weeks; however, this trend was reversed after 20 weeks, with growth for the biochar (N2B4) and the high dose of nitrogen (N4B0) increasing to eventually match the height of the seedlings supplied with the moderate dose of nitrogen (N2B0).

3.1.2. Biomass

Biomass at the end of the experiment differed significantly between the controls (6.23 g for black spruce, 9.32 g for pine) and nitrogen treatments (10.8 to 23.7 g) for both species (Table 1). However, the biochar treatment (without added nitrogen) did not increase the branch and needle biomass relative to the control (Figure 4). Whereas a significant biomass increase was observed for the three fertilizer treatments, compared with the nonfertilized

seedlings. We observed an approximate 4.6 to 5.4 g and 7.5 to 10.3 g increase in the needle and branch biomass, respectively, for black spruce seedlings in relation to the control. For jack pine, these increases in the needle and branch biomass, relative to the controls, ranged from 10.7 to 14.4 g and 4.6 to 5.1 g, respectively. In terms of the needle and branch biomass, spruce seedlings that received nitrogen fertilization weighed more than double that of the control plants. We observed an apparent increase in biomass (86% for spruce, 6.23 to 11.6 g; and 154% for pine, 9.6 g to 23.7 g), when the ammonium sulfate solution was added as a fertilizer.

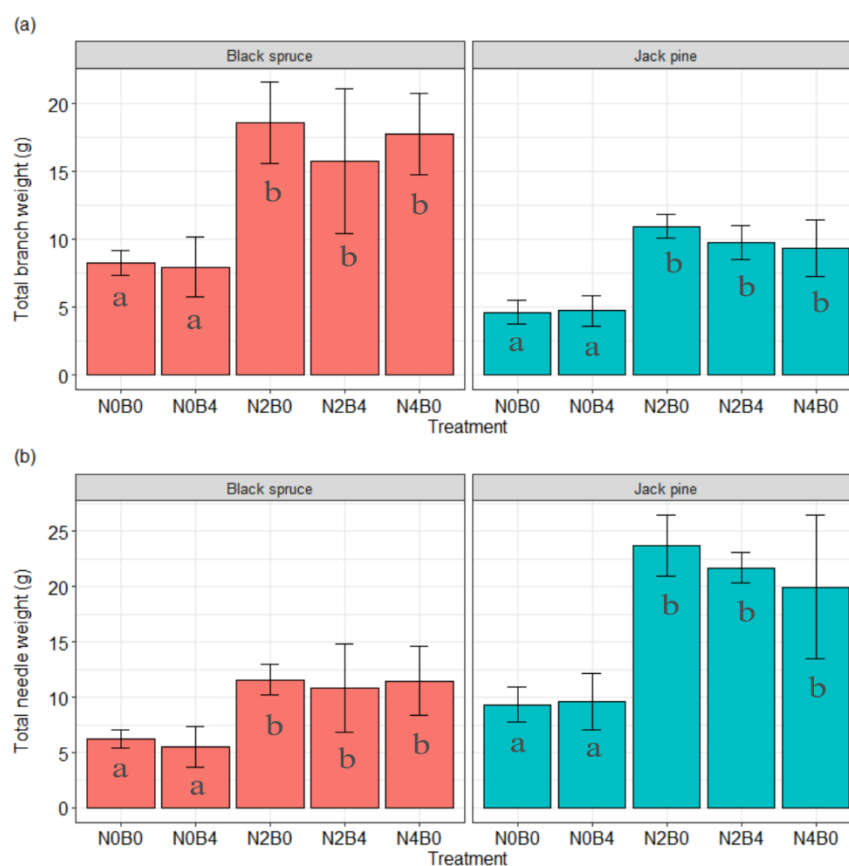


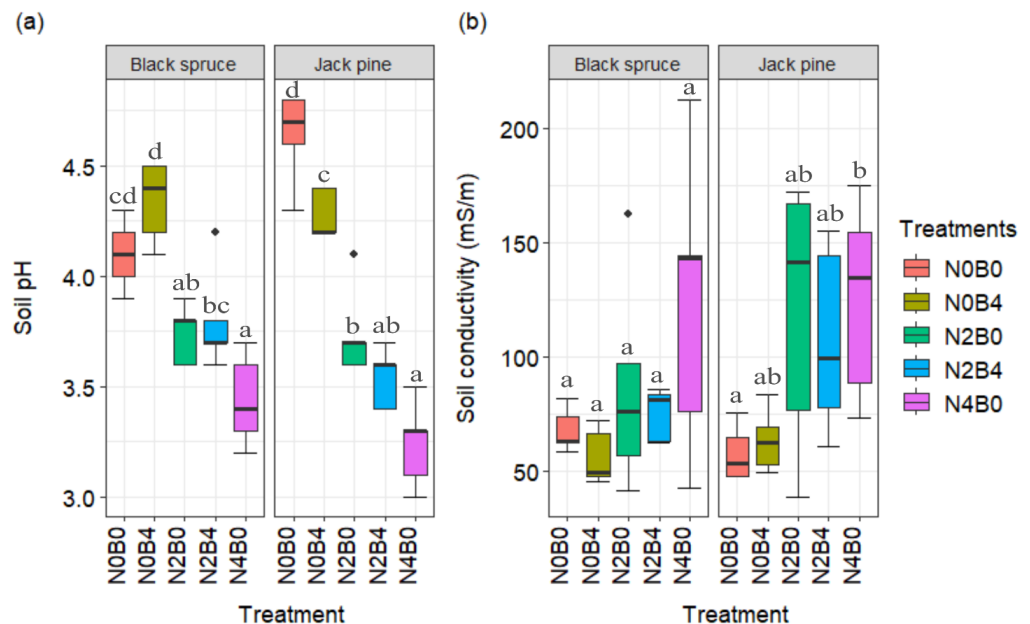
Figure 4. (a) Biomass of branches; (b) needles for black spruce and jack pine seedlings; error bars represent the 95% confidence interval and the significance difference between the treatments are represented by different letters, following $b > a$. Treatment N2B0/N2B4/N4B0 differ significantly from the control (N0B0) and the biochar alone (N0B4). N0B0: control; N0B4: 12.8 g of anhydrous biochar; N2B0: 200 kg N·ha⁻¹; N2B4: 200 kg N·ha⁻¹ and 12.8 g of anhydrous biochar; N4B0: 400 kg N·ha⁻¹.

3.1.3. Soil Properties

The PRS probes installed after nine weeks of fertilization for the three treatments recorded changes in the soil element composition (Table 2, Figure S2). The most significant change was the total nitrogen content for the specified treatments; higher nitrogen concentrations (887.84 mg·m⁻² per two weeks for N4B0) were obtained in soils with the highest fertilizer dose. Moreover, the elemental concentrations of Cu, Fe, K, P, and Pb decreased as the nitrogen fertilizer increased. Adding fertilizer caused the soil to become more acidic (pH lowered from 4.1 to 3.4 for black spruce seedlings, 4.6 to 3.2 for jack pine seedlings) (Figure 5a). There was significantly different and high soil conductivity in regard to the jack pine seedlings compared to the black spruce seedlings (Figure 5b).

Table 2. Soil ion (cation and anion) concentrations for greenhouse samples, as recorded by PRS probes. N0B0 control; N2B0: 200 kg N·ha⁻¹; N4B0: 400 kg N·ha⁻¹.

| | Soil Ions | Treatment | | |
|--|-------------------------------|-----------|--------|---------|
| | | N0B0 | N2B0 | N4B0 |
| Elemental composition (mg/m ² of membrane/two weeks) | Total N | 3.04 | 645.53 | 887.84 |
| | NO ₃ ⁻ | 3.03 | 373.97 | 331.58 |
| | NH ₄ ⁺ | 0.01 | 271.55 | 556.26 |
| | Ca ²⁺ | 481.37 | 857.67 | 908.62 |
| | Mg ²⁺ | 167.98 | 301.52 | 318.58 |
| | K ⁺ | 71.15 | 43.89 | 41.37 |
| | PO ₄ ³⁻ | 0.49 | 0.44 | 0.19 |
| | Fe ²⁺ | 12.32 | 11.63 | 6.41 |
| | Mn ²⁺ | 3.03 | 25.87 | 22.86 |
| | Cu ⁺ | 1.07 | 1.1 | 0.66 |
| | Zn ²⁺ | 0.8 | 1.53 | 1.34 |
| | BO ₃ ³⁻ | 0.03 | 0.03 | 0.01 |
| | S ²⁻ | 279.23 | 718.14 | 1048.48 |
| | Pb ²⁺ | 0.1 | 0.02 | 0.03 |
| | Al ³⁺ | 14.9 | 40.34 | 30.75 |
| | Cd ²⁺ | 0 | 0.01 | 0.01 |

**Figure 5.** (a) Soil pH; (b) conductivity among the five treatments for spruce and pine seedlings. The letter corresponds to the significance of the difference between the treatments, following $d > c > b > a$. N0B0: control; N0B4: 12.8 g of anhydrous biochar; N2B0: 200 kg N·ha⁻¹; N2B4: 200 kg N·ha⁻¹ and 12.8 g of anhydrous biochar; N4B0: 400 kg N·ha⁻¹.

3.2. Field Experiment

3.2.1. Growth

The linear mixed-effects model explained 70% of the variation in the diameter of black spruce in the field experiment plots (Table S2). The average diameter increased by 1.14 cm per year (2015–2020), with the other variables being constant. Nitrogen at medium or high doses increased the diameter growth by 1.0 cm (84% diameter increment) and 0.7 cm (61% diameter increment) per year, respectively. We also observed a significant difference in the diameter growth between all the treatments. The observed growth was highest for the medium nitrogen dose (Figure 6). However, all three field treatments (N0, N1.5, and N3) exhibited very similar patterns of height increase. We observed significant differences only between the controls and the seedlings that received a medium nitrogen dose. The growth differences between the medium-dose seedlings and the controls produced seedlings with

a reduced taper index (height/diameter, from 48 to 55) (Table S3); therefore, the fertilized seedlings were less slender and more robust. The differences in the slopes of the diameter and height between the treatments, especially after the first year of fertilizer application, confirmed the effectiveness of fertilization (Figure 6). This difference was visible as a greater stem volume for black spruce seedlings treated with ammonium sulfate. The average volumes in 2018 were 52% higher than those of the controls, whereas in 2020, this difference was 37% greater (relative to the control) for seedlings that received the medium dose of nitrogen (Table S3).

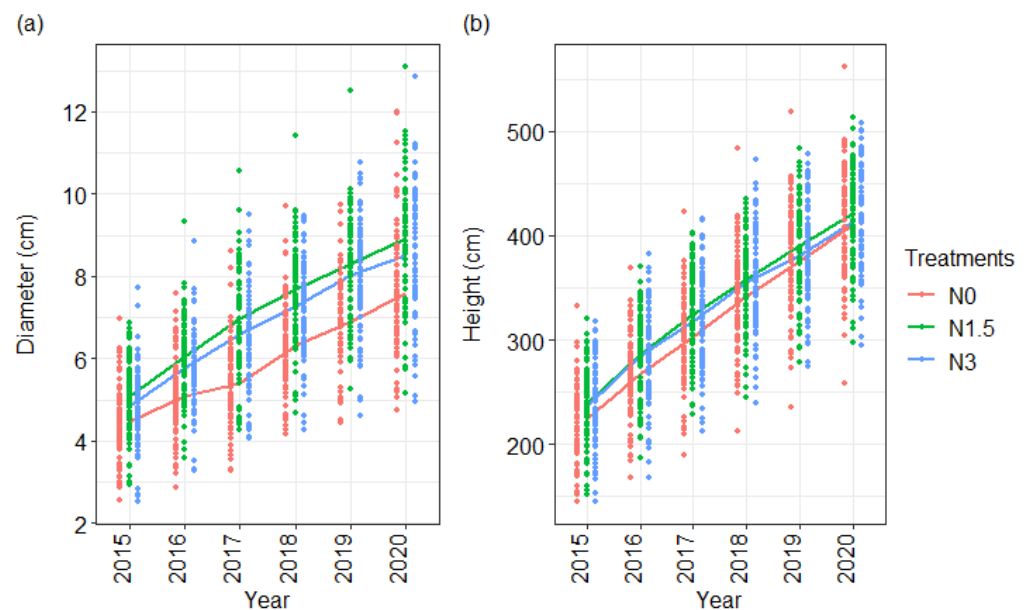


Figure 6. Trends in (a) diameter, (b) height growth for black spruce seedlings during the five years of the field experiment. N0: control; N1.5: 150 kg N·ha⁻¹; N3: 300 kg N·ha⁻¹.

The amount of nitrogen was higher than that of phosphorus and potassium in all three treatments (Figure 7); however, no significant difference was observed between the foliar compositional ratios of N, P, and K between the treatments.

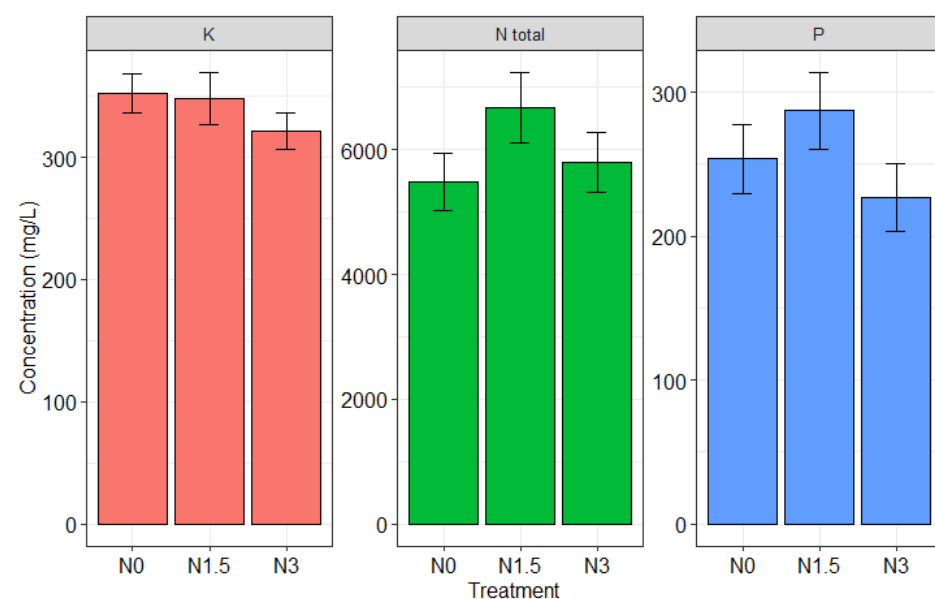


Figure 7. Foliar composition of the black spruce needles. Note that there are different scales on the y-axis. N0: control; N1.5: 150 kg N·ha⁻¹; N3: 300 kg N·ha⁻¹.

3.2.2. Soil Properties

The amount of soil nutrients available to black spruce differed between our experimental plots. In 2018, the nitrogen concentration in the control plots seems to be lower for the organic layer and higher for the mineral layer, compared to the plots treated with the fertilizer (Table S4). Fifteen weeks after fertilization, in 2019, the treated plots had lower nitrogen levels in the soil solution than the control plot (3.2% vs. 0.84% and 1.4% for the respective organic layers; Table 3). The treated plots also had higher levels of available Ca, Mg, Al, Mn, Zn, and S. The concentrations of C, H, and N increased to a greater extent in the organic soil layer than in the underlying mineral horizon (Figure S3).

Table 3. Cations and anions measured in the soil, according to the treatment in respect of the organic and mineral horizon (2019 data). N0: control; N1.5: 150 kg N·ha⁻¹; N3: 300 kg N·ha⁻¹.

| Treatment | Soil Horizon | Ca ²⁺ | K ⁺ | Mg ²⁺ | PO ₄ ³⁻ | S ²⁻ | pH | C | H | N |
|-----------|--------------|---|----------------|------------------|-------------------------------|-----------------|------|-------|------|------|
| | | (mg/m ² of Membrane/2 Weeks) | | | | | | (%) | | |
| N0 | Organic | 2378.33 | 1300.50 | 740.33 | 313.83 | 0.20 | 3.43 | 26.21 | 2.58 | 3.16 |
| | Mineral | 2538.67 | 1574.33 | 937.50 | 121.15 | 0.05 | 3.48 | 4.97 | 0.40 | 2.99 |
| N1.5 | Organic | 2535.83 | 1055.50 | 1103.83 | 337.70 | 0.17 | 2.93 | 27.46 | 2.92 | 0.84 |
| | Mineral | 2314.60 | 377.00 | 655.60 | 154.20 | 0.16 | 2.98 | 2.82 | 0.39 | 0.19 |
| N3 | Organic | 2819.40 | 1906.00 | 437.00 | 404.20 | 0.71 | 3.10 | 44.25 | 4.79 | 1.40 |
| | Mineral | 1510.25 | 234.25 | 2267.25 | 74.48 | 0.09 | 3.58 | 2.29 | 0.34 | 0.13 |

4. Discussion

The greenhouse and field experiments demonstrate that ammoniacal nitrogen from mining effluent can be a good source of fertilizer to optimize forest productivity in black spruce- and jack pine-dominated boreal stands. Nitrogen fertilization can improve tree growth and overall forest yield [4,10]. We also observed increased growth and biomass for both black spruce and jack pine after applying nitrogen at both greenhouse and plantation sites. Moreover, soil nutrient concentrations were higher for the nitrogen-treated seedlings than for the untreated seedlings, which increased the uptake of macronutrients essential for plant metabolic activities [1,43]. The use of ammoniacal effluent from regional mining sites can offer a novel means of reducing the environmental impacts of nitrate accumulation around mining locations [11,17,56]. Moreover, this fertilization could heighten forest productivity, often limited in the circumboreal region by low concentrations of available nitrogenous nutrients, benefiting the regional forest economy.

The added nitrogen derived from mining effluent led to increased seedling growth, because increased nitrogen availability enhanced the photosynthesis process [57]. Three nitrogen treatments in the greenhouse experiment (N2B0, N2B4, and N4B0) enhanced seedling growth and biomass accumulation. Under the conditions of optimal nitrogen availability, greater growth could have been achieved because of the higher photosynthetic rates [58,59]. Nonetheless, jack pine seedlings lagged behind in terms of their height growth relative to that observed for black spruce seedlings. Because the soil used for the greenhouse experiment came from a natural black spruce stand with an organic-rich substrate, the growth of spruce seedlings may have been favored. As the soil acidity level changed with the application of the fertilizer (Figure 5), it could also possibly interfere with pine growth, which usually grows in slightly acidic conditions. These edaphic factors and climatic conditions could explain the observed height disparity between the seedlings of black spruce (49% more vertical growth than the controls) and jack pine (21% more vertical growth than the controls) [60]. Nevertheless, similar diametric growth (approximately 30–35% greater than the controls for both species) suggests that species-specific factors most likely reduced the height of the jack pine. For instance, as excessive moisture can limit taproot development in jack pine, inadequate drainage can limit seedling growth, even under conditions with appropriate soil nutrients [61]. For the greenhouse trials, however, we note that a longer growing season may affect all growth gains, as the greenhouse plants

emerged from dormancy in mid-January compared with early mid-June for field seedlings. This resulted in a 30-week growing period for the greenhouse samples, compared with approximately 14 weeks for the field samples. Moreover, the biochar-alone treatment (N0B4) produced slightly taller jack pine seedlings than the control for pine seedlings; however, this effect was not significant [33]. Because jack pine is an early-successional species associated with fires, biochar could be a priori favorable for its growth, as biochar is similar to charcoal generated from natural forest fires, which promotes tree growth [45]. The slower growth observed for combined biochar and nitrogen treatments relative to the treatments with nitrogen alone could also be caused by the retention of nitrogen by biochar [29]. Indeed, most biochar has a porous structure, as well as numerous functional oxygenated groups (e.g., carboxylic acids, hydroxyls, and ketones) that can adsorb various nutrients (including phosphate, ammonium, and potassium) and reduce losses of soil nitrogen [62,63]. However, the change in growth at the end of the experimental period could reflect nitrogen losses. As the plants were placed outdoors for a few weeks to avoid excessive heating in the greenhouse, the soils likely experienced nitrogen loss through leaching, probably because of changes in the environmental conditions. These losses were likely less for the N2B4 treatment because of the nitrogen retention by the biochar.

In the field experiment, the diametric growth of black spruce responded better than the height growth when treated with a medium dose of nitrogen [45]. In natural settings, height is modulated by light availability, tree density, and competition [64]. As growth can be modulated by complex interactions occurring in the forest ecosystem [65], the effects of fertilization on black spruce vary depending on site-specific qualities [66]. Nevertheless, the added nitrogen could boost nutrient loading in the spruce seedlings through the accumulation of nitrogenous compounds [47]. In plantation plots, Sheedy [67] showed that nitrogen fertilization strongly stimulates growth, i.e., 25% over ten years, which supports our observations of increased tree volume for black spruce (37%) in 5 years. These results confirm that nitrogen is the element that can optimize tree growth in boreal forests [3]. Low nitrogen concentrations in boreal forest soils can occur because of the wildfires typical in this biome. During fires, significant amounts of nitrogen can be lost through volatilization [26]. The increased growth of seedlings reflects the role of nitrogen-rich fertilizers in enhancing growth and productivity in natural forests. The strong short-term growth (5 years) confirms that fertilization improves seedling nutrition, growth, and productivity.

In our field-based experiments, a medium dose of nitrogen ($150 \text{ kg N}\cdot\text{ha}^{-1}$) was more effective than a high dose of nitrogen ($300 \text{ kg N}\cdot\text{ha}^{-1}$) in promoting tree growth and productivity. The limited growth observed in plots treated with high nitrogen doses is likely due to the lower decomposition rate of nitrogen in the eastern Canadian boreal forest, which limits the availability of nitrogen to trees [68]. High nitrogen doses can possibly induce the deficiencies of other macro- and micronutrients [69]. Furthermore, applying higher nitrogen doses can lead to leaching and runoff, which can decrease the tree's retention of the available nitrogenous nutrients [18,70]. Such mechanism can potentially lower tree nutrient uptake, resulting in limited growth [71]. In addition, high nitrogen doses can negatively affect soil microorganisms and reduce growth and wood quality [18]. Higher doses can alter the response of forest-dependent communities; therefore, medium doses of nitrogen can be applied to limit the negative consequences, while enhancing tree production [9] and profitability from the forest [10].

Biochar influences the microbial activity involved in soil C–N recycling [72], likely affecting seedling growth and biomass. The lower needle biomass observed for black spruce seedlings indicates that these seedlings likely lost more needles, which were then broken down by microorganisms. This process of needle decomposition could be favored by the presence of biochar, which explains why the soil organic matter content was slightly higher for this treatment. Fertilization increased spruce growth, causing more needles to be produced and shed for soil microorganisms to decompose. We observed a greater decomposition of organic matter when the biochar was combined with nitrogen, as the needles on the ground decomposed well due to the presence of biochar. More significant

decomposition occurred because of the enhanced soil–microbiota interactions supported by the presence of biochar [29]. Biochar can also improve seedling resistance to pathogenic microorganisms by suppressing disease [73].

In the natural environment, soil organic matter is a plant's main nitrogen source. The nitrogen in the organic matter must be degraded by microorganisms into ammonium (NH_4^+) to be better assimilated by plants [74]. Whether NH_4^+ comes from decomposed organic matter or fertilizer, its fate can involve several pathways: use by microorganisms (immobilization), plant uptake, or volatilization. Adding nitrogenous fertilizer to raw humus leads to immobilization via its use by microorganisms, followed by accelerated degradation of the organic matter. This process provides a mechanism for conserving added nitrogen, reducing leaching [2], and explains the observed variations in the nitrogen concentrations.

Indeed, nine weeks after fertilization, the concentrations of Ca, Mg, Mn, and Zn were higher in the N2B0 and N4B0 treatments because of the decomposition by larger microorganisms. We did not observe increased potassium and phosphorus, as plants benefit from these nutrients. The sulfur concentration increased noticeably with the addition of the nitrogen fertilizer and reached a maximum level with the high nitrogen dose. Excessive sulfur levels in the soil can impair the growth of seedlings, so special consideration should be taken when using nitrogen-based fertilizer. However, in general, ammonium sulfate promotes seedling growth, and larger seedlings consume more nutrients for rapid development; on the other hand, nitrogen also promotes the decomposition of soil organic matter [75]. Thus, we would expect less organic matter in the nitrogen treatments (N2B0, N2B4, N4B0) relative to the controls.

Ammonium sulfate acidified the soils and decreased the abundance of alkaline cations [8,57]. At high doses of nitrogen, leaching may have increased the pH, to approach optimal growth conditions, and offered a greater residual nitrogen supply. For the jack pine vases, the added biochar also acidified the soil. This acidification occurred despite biochar's liming power, as biochar favored jack pine growth, increasing the seedlings' nutrient uptake and demand for cationic nutrients. Trees generally require more cationic nutrients than anionic nutrients, especially in cases where the available nitrogen is predominantly ammoniacal. The uptake of basic cations must be compensated by a release of protons (H^+), which acidifies the soil [2,18]. However, we did not observe such a pattern for black spruce seedlings because the decomposition of the organic matter made more cations available to reverse the phenomenon and maintain a less acidic soil [2].

In our field plots, the soil solution contained higher concentrations of N, Ca, Mg, Al, Mn, Zn, and S. Increases in the concentrations of the first five elements probably come from an improved recirculation of nutrients, as increased microbial decomposition led to the rapid metabolism of nitrogen and the decomposition of organic matter, which released nutrients into the soil solution [13]. Most sulfur in the soil solution likely came from the sulfate portion of ammonium sulfate. Interestingly, the high concentrations of NH_4^+ relative to NO_3^- observed in the plots were consistent with previous studies in boreal forests, possibly related to differences in nitrogen mineralization rates between the nitrogen-applied plots [76,77].

Despite an important result from the greenhouse and field experiments, this study has several limitations that affect the generalizability of the positive outcomes. The short duration of the experiments may not fully capture the long-term effects of ammoniacal nitrogen on tree growth, soil health, and ecosystem dynamics in field sites. For example, while our findings showed a significant increase in seedling growth over the short term, it remains uncertain whether these benefits would persist over the lifespan of the trees, which can live for more than 200 years. Furthermore, the specific site conditions, including the soil type, disturbances, and micro-climate in our trials, might be less variable among the blocks, but can differ in other regions in the boreal forest, which can potentially lead to different outcomes. To address these limitations, future studies should extend the monitoring period and include a variety of site conditions to better understand the broader applicability of ammoniacal nitrogen as a fertilizer in boreal forests. There is a need for long-term assess-

ments of tree growth, associated biodiversity, and environmental impacts in response to ammoniacal nitrogen treatments, including considerations on the sustainability, economic viability, and potential benefits for forest ecosystems. Future research should also apply experimental and modeling approaches in multiple provenance trials to confirm whether other plantation sites and species experience similar growth responses as those observed here. A detailed cost–benefit analysis is also required to evaluate whether the economic benefit obtained from enhanced tree growth outweighs the cost of transforming effluent into fertilizer. This assessment must include the overall carbon footprint, a life-cycle analysis [78], and the valuation of this residue at the regional scale. Kytä et al. [79] applied a life-cycle analysis to show that recycled fertilizers reduce energy consumption and greenhouse gas emissions relative to mineral fertilizers. The environmental footprint of applying this effluent must also be established. Agricultural fertilization produces a series of negative environmental impacts, including water contamination caused by runoff and leaching, the eutrophication of waterways, the disturbance of aquatic and terrestrial ecosystems, greenhouse gas emissions, and the consequences for human health [80]. Nevertheless, it should also be noted that nitrogenous fertilization could increase acidity, while also activating the accumulated heavy metals in the soil; this can potentially have negative effects on the environment. In forest ecosystems, soil microorganisms quickly mobilize nitrogen and, therefore, the associated risks are expected to be lower. The accelerated decomposition of organic matter promotes the recirculation of nutrients to favor greater plant nutrition, leading to a more significant release of carbon dioxide by the soil [81]. These effects could be similar to those encountered in agricultural ecosystems. Combining nitrogen fertilization with biochar could overcome these undesirable consequences [80]. Moreover, recent studies have indicated an increasing risk to tree growth and productivity in boreal forests, so adding nitrogenous fertilizer could minimize such risk in changing climate scenarios [20]. However, particular attention must be paid to the fate of applied nitrogen in forest sectors to minimize environmental impacts and maximize the ecological benefits.

5. Conclusions

Our field and greenhouse experiments with black spruce and jack pine seedlings demonstrated that nitrogenous fertilization enhances seedling biomass and growth. The greenhouse experiments suggest that ammonium sulfate could be a suitable alternative fertilizer to favor the growth of spruce and jack pine seedlings. Adding this nitrogen fertilizer, with or without biochar, significantly improves seedling growth and biomass. Comparatively, black spruce seedlings have a better growth response to ammonium sulfate fertilization than jack pine; we observed the same patterns at our plantation site for black spruce. Medium doses (150–200 kg N·ha⁻¹) of ammonium sulfate are sufficient to produce higher black spruce seedling growth and productivity. Therefore, to limit acidification and the release of heavy metals in the soil, we suggest not exceeding the 200 kg N·ha⁻¹ limit when applying nitrogenous fertilization in forest stands. Considering the approach of fertilizer generation, the output observed in terms of the seedling growth is remarkable and can unequivocally provide an alternative solution for minimizing environmental contamination. Thus, the use of recovered ammoniacal nitrogen from mining effluent appears to provide a promising option as a fertilizer for forest plantations and could help solve mining issues, while achieving sustainable forest management goals.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/su16177683/s1>, Figure S1: Black spruce and jack pine seedling (a) diameter, (b) height after 30 weeks of fertilization for the greenhouse experiments; Figure S2: Concentration of soil cations and anions from the greenhouse treatments; Figure S3: Concentration of soil cations and anions from the field experimental plots; Table S1: Summary of black spruce and jack pine growth in relation to the experimental treatments in the greenhouse experiments; Table S2: Linear mixed-effects model estimates of diameter growth in black spruce; Table S3: Average growth of black spruce in field experiments for each treatment for 5 years and the change in volume relative to the control in that particular year; Table S4: Cations and anions measured in the soil, according to

the treatment in relation to the organic and mineral layers, for the field experiments between 2016 to 2018.

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