THE RELATIVE MERITS OF DOLOMITIC AND CALCITIC LIMESTONE IN DETOXIFYING AND REVEGETATING ACIDIC, NICKEL- AND COPPER-CONTAMINATED SOILS IN THE SUDBURY MINING AND SMELTING REGION OF CANADA[1]

by
Derek McHale and Keith Winterhalder[2]

Abstract. Soils in the Sudbury mining and smelting region have been rendered phytotoxic and barren by acidification and particulate metal contamination, but can be detoxified and revegetated by the surface application of ground limestone. On certain barren sites, plant growth is better on soil treated with dolomitic limestone than with calcitic limestone, and greenhouse experiments using mung beans (*Vigna radiata*) have shown superior root and shoot growth on certain contaminated soils when the limestone is dolomitic rather than calcitic. Results of experiments with species used in revegetation (*Agrostis gigantea* and *Lotus corniculatus*) suggest that leguminous species are more sensitive to Ca:Mg ratio than grasses, that the plant response to this ratio is greater at lower liming levels, and that the response is more marked on more toxic soils. The effects of calcium:magnesium ratio of the limestone used in revegetating acidic, metal-contaminated soils are clearly complex, interactive and difficult to interpret. Further studies are needed, but meanwhile it is recommended that the practice of using dolomitic limestone to detoxify barren Sudbury soils be continued, since there is a risk of induced magnesium deficiency at certain sites when calcitic limestone is used.

Additional Key Words: Ca:Mg ratio, liming rate, *Agrostis gigantea*, *Lotus corniculatus*

Introduction

Until recently, 10,000 ha of barren, phytotoxic land encircled the three Sudbury smelters (Coniston, Copper Cliff and Falconbridge). The barren soils, ranging in pH from 3.5 to 4.5, and with copper and nickel contents frequently exceeding $1{,}000 \mu g \cdot g^{-1}$, are the result of the effects of a century of mining and smelting activity, including SO_2 fumigation and copper, nickel, and iron particulate fallout from the smelters.

The construction of a 380m smokestack at the Copper Cliff smelter, plant closures, legislated reductions in SO_2 emissions, and improved emission control technology, have brought about a steady decline in atmospheric SO_2 and in particulate emissions from the smelters since 1972. A detectable rise in soil pH and fall in soil metal levels has occurred (Gundermann and Hutchinson 1993; Dudka *et al.* 1995), but recolonization of the barrens has been mostly confined to species that have evolved metal tolerance, such as *Deschampsia caespitosa* (Cox and Hutchinson 1980), *Agrostis gigantea* (Hogan *et al.* 1977), *Agrostis scabra* (Archambault and Winterhalder 1995), *Poa compressa* (Rauser and Winterhalder

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[2] Department of Biology, Laurentian University Sudbury, Ontario, Canada P3E 2C6.

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1985) and *Betula pumila* (Roshon 1988), and there are still areas close to the smelters that are devoid of vegetation. In these acid soils, aluminum ions are liberated from aluminosilicate minerals, as are copper and nickel ions from particulate smelter fallout. Seeds germinate, but unless the plant is from metal-tolerant stock, root growth is inhibited and the roots are unable to penetrate the soil.

Ground limestone, when applied to the surface of the toxic soil, acts as a 'Trigger Factor' (Winterhalder 1983), initiating spontaneous colonization by metal-intolerant plants from a local seed source. The stony mantle that covers these eroded, glacial till-derived soils traps limestone particles and seeds, acting as a protective mulch to the young seedlings. The soil is effectively detoxified, despite a modest pH rise of as low as one unit (e.g. from 4.5 to 5.5), allowing the seedling to develop a deep root system, protecting it against drought and frost-heaving, and enabling it to reach bases and other nutrients deeper in the profile. Ultimately, the leaf litter provides insulation, thereby reducing frost damage, while the humus helps to complex potentially toxic metals.

Winterhalder (1995) has suggested five possible components of the complex mechanism of detoxification. Precipitation of copper and nickel from solution is probably relatively rare, since a pH of above 5.5 is rarely achieved. However, the Al^{3+} ions that result from the hydrolysis of clay minerals are hydroxylated and rendered less toxic as the pH rises. Calcium ions enhance the plasma membrane integrity of root hair cells (Epstein 1961), and the abundant calcium ions reduce metal uptake by the roots by competitive exclusion (Kinraide and Parker 1987).

The amelioration of metal toxicity by calcium is well documented. Increased calcium can reduce the detrimental effects of aluminum on soybean (Lund 1970; Brady *et al.* 1993) and nickel toxicity in corn (Robertson 1985). Calcium-nickel interactions can be complex, and Knight and Crooke (1956) found that the calcium uptake of oat plants, but not of tomato plants, was higher in nickel-contaminated soils .

Quite fortuitously, the limestone that has been used in Sudbury's Regional Land Reclamation Program has been dolomitic, with a Ca:Mg ratio of approximately 2:1.

Calcium and magnesium levels in Sudbury soils are within the normal range for soil in a temperate region (Hazlett *et al.* 1983), despite a century of acid leaching, probably because both Ca and Mg are components of the emissions of the Copper Cliff smelter, at rates of over 300 $kg \cdot day^{-1}$. It therefore seems unlikely that plants on Sudbury soils would suffer from a simple deficiency of calcium or magnesium. Nevertheless, Winterhalder (1983) suggested that the beneficial effects of liming Sudbury barren soils involved both neutralization and a calcium nutrition effect. Later (Winterhalder 1993), it was suggested that calcium:magnesium balance might play a critical role in the nutrition of plants in limed Sudbury soils.

Tan *et al.* (1992), when examining a variety of sorghum genotypes which had different sensitivities to acid soils, found that the benefit of liming is increased when there is an adequate magnesium supply. It is therefore possible that the exclusive use of calcitic limestone on Sudbury soils would bring about a calcium-magnesium imbalance and consequent induced magnesium deficiency, in which the superabundance of calcium ions would exclude magnesium uptake by competing on the root exchange sites, since magnesium is less competitive than calcium and aluminum under acid conditions (Sumner *et al.* 1991). In addition to its nutritional role, possible beneficial effects of magnesium include interaction with potentially toxic metals. Proctor and McGowan (1976) showed a clear alleviation of Ni toxicity by Mg in oats, and Robertson (1985) showed that both calcium and magnesium provided protection to corn roots against nickel poisoning, although at higher levels Mg itself became toxic unless alleviated by the appropriate Ca balance.

Previous studies on the effects of Ca and Mg have focused on agricultural plant species growing in soils which are acidic and therefore contain elevated levels of soluble aluminum, or else on serpentine soils which have a neutral to basic pH, high levels of nickel, chromium and magnesium, and low levels of calcium (Johnston and Proctor 1981). The Sudbury area soils are unique in being acidic like the former but metal-contaminated like the latter. The purpose of this paper is to examine the relative merits of dolomitic and calcitic limestone, with respect to the Ca:Mg ratio within the limestone, for detoxifying and revegetating acidic, nickel- and copper-contaminated soils in the Sudbury region.

Plant Growth Experiments

At the beginning of the investigation, several exploratory experiments were carried out to test the hypothesis that the two limestone types could have differential ameliorative capacities. Two examples follow.

1. Mung bean (*Vigna radiata*) on semi-barren soil. Mung bean seedlings were grown in soil collected from a semi-barren birch woodland 6 km south-west of the Falconbridge smelter and 6 km

north of the Coniston smelter (Maki Road). The four treatments were: no limestone (control), calcitic limestone, 50:50 calcitic:dolomitic limestone, and dolomitic limestone. A total of 1.5 g of limestone was mixed into 150 g of soil in each pot. As shown in Figure 1, both root and shoot growth were significantly greater in the dolomitic containing treatments than in the calcitic or control treatments.

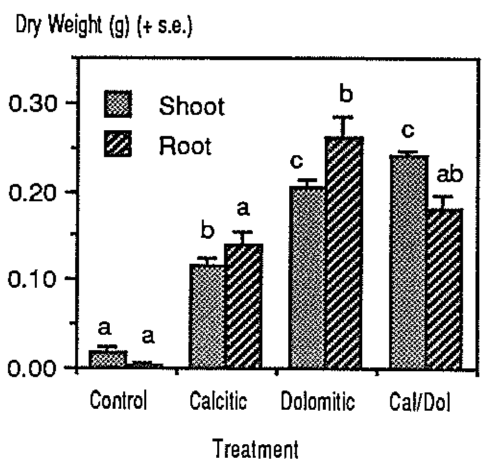


Figure 1. Mean *Vigna radiata* shoot and root dry weights in Maki Road soil under different limestone treatments (vertical bars represent standard error). Means for shoots or roots with the same superscript are not significantly different ($p<0.05$).

2. Redtop (*Agrostis gigantea*) on barren soil. Mung bean is a convenient experimental species, but has no relevance to the revegetation program. Therefore seedlings of redtop, a major revegetation grass species, were grown in barren soils from 10 km northeast of the Copper Cliff and 2 km south of the Falconbridge smelter, respectively, using the same protocol as above, except that 1.0 g of limestone was mixed into 150 g of soil. In neither soil did shoot biomass show significant response to dolomitic limestone, and the only significant root response was that seen in Copper Cliff soil (Figure 2), where the response was once again significantly greater in dolomitic treatments than in the control and calcitic treatments.

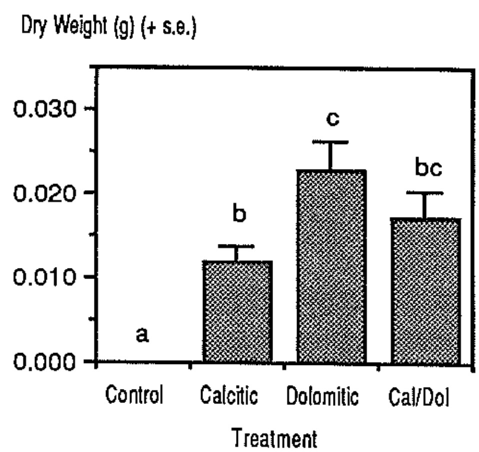


Figure 2. Mean *Agrostis gigantea* root dry weights in Copper Cliff soil under different limestone treatments (vertical bars represent standard error). Means with the same superscript are not significantly different ($p<0.05$).

Two additional, more comprehensive experiments were then carried out, the first looking at early seedling root and shoot growth under two lime types, and the second at longer term growth under widely varying Ca:Mg ratios.

3. Petri dish bioassay using redtop (*Agrostis gigantea*) on three barren soils. Three barren soils from the vicinity of each of the three smelters (Table 1) were used in a Petri dish root/shoot growth bioassay.

Table 1. Chemical characteristics of soils used in Petri dish bioassay (Mean ± standard deviation for three samples at each smelter). Within rows, data followed by the same letter are not significantly different at the 5% level (Tukey Multiple Range Test). O.M. = Organic Matter, W.S. = Water Soluble, Ext. = Weak Acid Extractable, Tot. = Total.

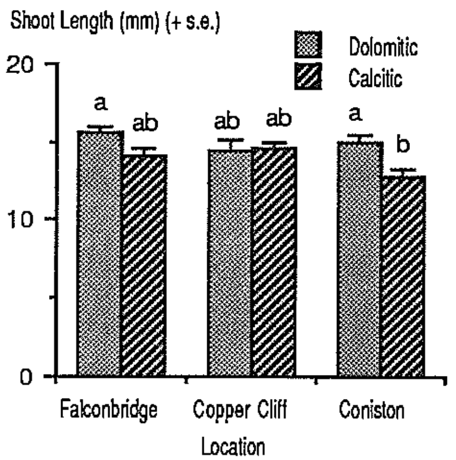
	Falconbridge	Copper Cliff	Coniston
pH	3.75a	4.00b	3.85ab
O.M.	6.95%a	7.86%a	10.74%b
W.S. Ni	3.6±3.7a	11.9±5.5b	16.2±21.6c
Cu	2.95±1.4a	7.9±3.4b	16.8±4.5c
Al	11.0± 8.3a	29.24±9.1ab	64.8±60.4b
Ca	31.8±41.2a	42.35±41.4a	60.3±72.8a
Mg	4.1±2.7a	4.2±1.70a	6.2±6.8a
Ext. Ni	8.6±8.34a	20.0±12.0a	17.8±26.5a
Cu	71.0±32.5a	105.3±28.8a	176.3±76.2b
Al	381.3±230.4a	981.2±364.6b	1215.6±283.2b
Ca	74.0±77.7a	76.7±34.4a	105.2±110.7a
Mg	5.9±3.9a	6.9±5.5a	8.0±7.5a
Tot. Ni	410.3±191.0a	506.9±259.0a	611.6±314.2a
Cu	450.8±128.0a	484.3±131.9a	806.6±431.1b
Al (%)	4.3±0.3a	5.7±0.7b	5.3±0.8b
Ca (%)	2.9±3.2a	3.5±0.7a	2.7±1.7a
Mg (%)	2.0±0.3a	3.5±0.4b	3.6±1.7b

Values in $\mu g \cdot g^{-1}$ except where indicated.

40 g of each soil were placed in a filter paper lined perforated plastic Petri dish which was then placed in a larger glass Petri dish. The treatments were: no limestone (control), calcitic limestone, and dolomitic limestone, the limestone being applied by sprinkling 0.2 g onto the soil surface. Glass fibre filter paper was placed over the soil, and the soil was wetted from below. Twenty-five seeds of *Agrostis gigantea* were placed on the surface of each glass fibre filter paper.

The seeds were kept in darkness for 4 days, then in a 12 hour light/dark cycle for a further 12 days, after which the roots and shoots were measured to the nearest millimetre. In the case of the unlimed soils (data not shown), a gradient existed in the toxicity of the unlimed soils with respect to both the root and shoot growth of *Agrostis gigantea*. The roots in the Falconbridge soil were significantly longer than those in the other two soils. The shoot lengths were significantly the longest in the Falconbridge soil and the shortest in the Coniston soil, with the shoots in the Copper Cliff area soil being intermediate in length, but not significantly different from the others. In all cases there was a significant difference between the controls and the lime treatments.

In the limed treatments (Figure 3), there was no significant difference in root growth between limestone types for any of the soils, but the fact that the Coniston soil was the most inhibitory to root growth, even after liming, was clear .



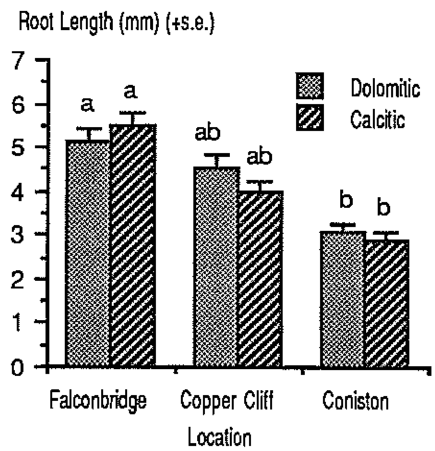


Figure 3. Mean shoot lengths (above) and root lengths (below) of young *Agrostis gigantea* seedlings grown on three Sudbury soils under different limestone treatments (vertical bars represent standard error). Means with the same superscript are not significantly different ($p<0.05$).

However, shoot growth showed a significant response to the dolomitic treatment on the Coniston soil, but not on the Falconbridge or Copper Cliff soil .

4. Ca:Mg Ratio Pot Experiment. Seeds of redtop and birdsfoot trefoil (*Lotus corniculatus* - a major leguminous species in revegetation) were sown in two contaminated soils (Coniston and Falconbridge) and a control soil (Cartier, 44 km northwest of the Copper Cliff smelter), each of which had been treated with finely-powdered calcium carbonate-dolomitic limestone combinations to give equal neutralization, but with Ca:Mg ratios of 1:0, 192:1, 96:1, 48:1, 24:1, 8:1, 4:1 and 2:1, respectively. Two neutralization levels were employed (0.2 g limestone•pot^{-1}, giving a pH of approximately 4.0, and 0.8 g limestone•pot^{-1} giving a pH of approximately 5.5). The experiment was replicated five times, and after 2 months of growth, roots and shoots were harvested and dry weights determined.

Results for the two plant species were analyzed separately, as were the two lime rates (moderate and low). Ca:Mg ratio had no significant effect on root or shoot growth of *Agrostis gigantea* at either lime rate on any soil, and for *Lotus corniculatus* there was no significant difference between treatments at the moderate lime rate for any soil. However, at the low lime rate, Ca:Mg ratio significantly influenced shoot mass in *Lotus corniculatus* in the Coniston area soil, with significantly higher shoot mass at Ca:Mg ratios of 4:1 or less (Figure 4).

Leaf chlorosis was consistently present in both contaminated soils at the low lime rate, but more intense under the higher calcium regimes.

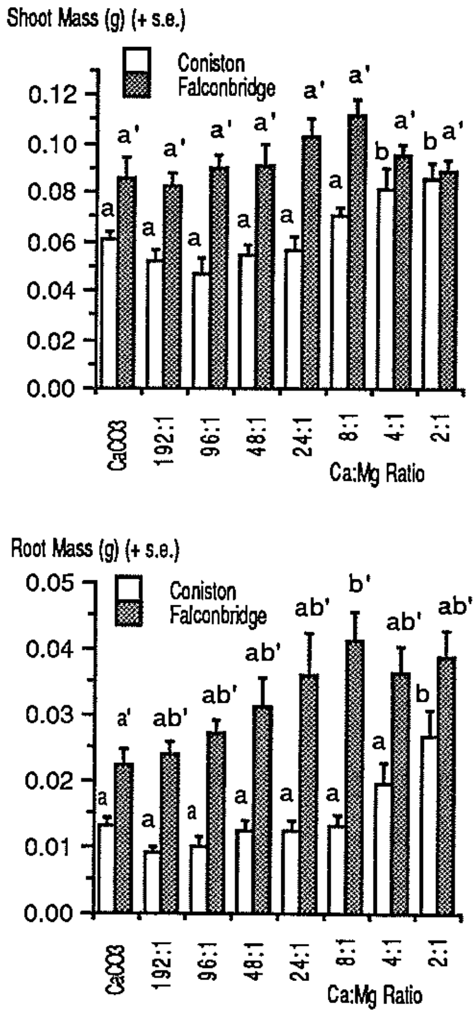


Figure 4. The effect of Ca:Mg ratio at low liming rate on the growth of birdsfoot trefoil shoots (above) and roots (below) on Coniston and Falconbridge soils (vertical bars represent standard error). Means with the same superscript are not significantly different ($p<0.05$).

Also at the low lime rate, Ca:Mg ratio significantly influenced root mass in *Lotus corniculatus* in both Coniston and Falconbridge soils (Figure 4). The uncontaminated Cartier soil showed no response to either the lime rate or the calcium-magnesium ratio for either species.

Discussion

The dramatic response of the mung bean seedlings to the dolomitic limestone treatment in the first experiment strongly supported the hypothesis, based on previous anecdotal evidence, that Ca:Mg balance can play an important nutritional and/or anti-toxic role on certain Sudbury area soils. However, redtop did not give the same consistent results on two different toxic soils in the second experiment. Indeed, the fact that the only significant response of redtop was that shown by roots in the Copper Cliff soil, whereas in the Maki Road soil the greater response of mung bean was evident in the shoots, suggests that the phenomenon is species and/or soil dependent.

There was a clear gradient in the toxicities of the three un-limed Sudbury area soils, with Coniston being the most toxic, followed by Copper Cliff, and finally Falconbridge. The Coniston area soil consistently had higher levels of copper and nickel, and a higher percentage of organic matter. Although all of the contaminated soils were barren when collected, they possessed different degrees of toxicity. It is still not clear which of these factors is interacting with the limestone to produce the differential responses.

In the third experiment, a contradictory situation arose, in which there was no significant root response to dolomitic limestone in any of the soils, but a definite shoot response on the Coniston soil, which also appeared to be the most toxic. The difference here could have been soil-related. At higher soluble metal concentrations, the competition for exchange sites could reduce the magnesium available to the shoots, resulting in a lime induced magnesium deficiency, but at slightly lower metal levels the ameliorating effects on the metal toxicity in the rhizosphere would be the over-riding factor. Furthermore, the Coniston soil had the highest organic matter content. Although organically bound copper is less rhizotoxic than the ionic form, it is possible that it could be taken up by the plants and translocated where metal ions could not, as shown in the case of EDTA-chelated copper in *Triticum aestivum* by Taylor and Foy (1985). Alternatively, the difference could be the result of the fact that the organs measured were radicles and first leaves, rather than mature root and shoot systems, as well as the fact that length, rather than biomass, was measured.

In the fourth experiment, an interesting difference between the behaviour of the grass (redtop) and the legume (birdsfoot trefoil) appeared, in that there was no significant response to dolomitic limestone in redtop at any liming level in any soil, whereas birdsfoot trefoil shoots showed a significant response at the low liming level. Similar results were found by Edmunds *et al.* (1983) in a study on the effect of calcitic limestone on ryegrass and white clover grown on a New Zealand yellow-brown loam. After examining yield and internal magnesium concentrations, they showed that magnesium concentration in the grass decreased in proportion to increasing yield, suggesting a dilution effect. In the clover, however, magnesium concentration decreased with plant yield reduction, suggesting that the calcitic limestone had an antagonistic effect on magnesium uptake. One consistent feature in the Sudbury and New Zealand experiments has been the apparent greater sensitivity to Ca:Mg ratio of leguminous species, compared to grasses, possibly due to the lower ability monocot roots have to bind cations such as calcium (Rorison and Robinson 1984).

McNaught *et al.* (1973) were able to demonstrate that liming with $Ca(OH)_2$ in a pasture made up of primarily white clover could reduce the tissue magnesium levels compared to magnesium-containing liming materials. The legume used in the present study, *Lotus corniculatus*, may be particularly sensitive to the Ca:Mg ratio in the soil. In a study on acidic, infertile Appalachian soils, Baligar *et al.* (1985) examined the differences in growth and nutrient uptake in six legume species. The soils were limed with $Ca(OH)_2$, and the only species that did not respond positively to the lime was the birdsfoot trefoil. However, the liming reduced the magnesium levels in all legume species to low to deficient rates, although, unlike birdsfoot trefoil, the other legumes, including white clover, responded with increased yield. Clearly, response of legumes to liming is species-specific, but the nature of the species-specific response factor or factors is not known.

It may be helpful to attempt to interpret the results of these experiments in the light of the two probable functions of the limestone - the amelioration of rhizotoxicity with its concomitant root growth inhibition, and the reduction of metal uptake (particularly of nickel) and concomitant effects on shoot growth and vigour. The amelioration of root toxicity would involve such things as the hydroxylation of aluminum ions and competitive exclusion of metals by base ions, whereas shoot effects would involve mechanisms that reduce nickel uptake such as magnesium:nickel interactions, plasma membrane integrity effects and (once again) competitive exclusion. On this basis one would expect response to dolomitic limestone to be expressed in the leaves more than in the roots, and this is indeed the case in three of the four experiments. The reduced shoot growth of *Lotus corniculatus* in the Coniston area soil at the higher calcium-magnesium ratios could be the result of a lime induced magnesium deficiency. Because the Mg^{2+} ion is also involved in the amelioration of nickel toxicity (Robertson 1985), its effectiveness is reduced when it is in lower concentrations, relative to calcium.

On the other hand, the presence of an increased response to the lower Ca:Mg ratios in the roots of the *Lotus corniculatus* growing in the Falconbridge soils, but not in the shoots, would indicate a reduction in copper and aluminum toxicity afforded by the higher magnesium concentrations.

Christenson *et al.* (1973) examined oat plants growing in acid soils amended with different calcium and magnesium sources. They showed that a high Ca:Mg ratio suppressed magnesium uptake, although low pH had a greater effect on magnesium uptake than did calcium source. This could explain, in part, the fact that birdsfoot trefoil only responded to dolomitic limestone at a low liming rate. Sumner *et al.* (1978) showed that, in highly weathered soils which have been limed with finely ground limestone, the amount of exchangeable Mg increases to roughly pH 6, then decreases, as the pH approaches neutrality. Thus, as the pH of an acid soil is adjusted towards pH 6, a greater proportion of the magnesium will be held on exchange sites, rather than in the soil solution, making it more available to the plants (Sumner *et al.* 1991). While on the exchange sites, the competitive disadvantage of the magnesium is lost, and the lime induced deficiency no longer exists. This was observed in the last Sudbury experiment, in that there were no significant responses to Ca:Mg ratios at the higher lime rate. Tan *et al.* (1991) also found that, on aluminum toxic soils, there was a significant response of aluminum sensitive sorghum genotypes to the addition of magnesium at low rates of liming, but as the lime rate increased the effectiveness of the magnesium was reduced. In the light of Sumner *et al.*'s (1991) observations, it is possible that, if the Sudbury soils were limed to above pH 6, there would be a decrease in available magnesium, resulting in a reduced yield.

The importance of soil toxicity level was again apparent in the Ca:Mg ratio experiment. There was a strong response in the highly toxic Coniston soil, whereas there was little response in the less toxic Falconbridge soil. Shoot growth responded significantly to Ca:Mg ratio in the Coniston soil, but not in the Falconbridge soil. Root growth in the toxic Coniston soil was highest at the 2:1 ratio, while in Falconbridge soil it peaked at 8:1. Plants on the uncontaminated Cartier soil did not respond to either liming rate or Ca:Mg ratio.

Since other authors have demonstrated lime induced magnesium deficiency in various soil types and plant species, the fact that it is not evident in <u>all</u> Sudbury area soils indicates that there are other factors operating. From the experiments so far conducted, it is clear that the effects of the calcium-magnesium ratio in limestone are complex and interactive, and it is difficult to identify which factors in the soil are reacting with which elements of the limestone. There is evidence, however, that there is a relationship between the phytotoxicity of limed acid metal-contaminated soils and the calcium-magnesium ratio of the limestone employed. Further studies are needed to further define this relationship.

It is recommended that the practice of using dolomitic limestone to detoxify barren Sudbury soils be continued, since there is a risk of induced magnesium deficiency at certain sites when calcitic limestone is used.

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