

Project: Evaluation of gypsum and mined calcium sulfate anhydrite as pre-leaching soil amendments for sodium management

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## Summary

This study was designed to evaluate the ability of two calcium sulfate based products – gypsum (calcium sulfate dihydrate) and mined anhydrite (calcium sulfate anhydrite) to reduce soil sodium levels when applied to *Poa annua* greens prior to leaching. Key conclusions were:

- Leaching of both treated and non-treated greens resulted in significant reductions in soil salinity, sulfur, chloride, magnesium, potassium, sodium, sodium percentage and nitrate.
- One application of either mined anhydrite or gypsum at 10 lbs product/1000 sq ft did not result in further reductions in soil salts or sodium, or in an increase in soil calcium or sulfur. It was simply leaching, whether on the treated or non-treated greens, that provided the only significant reductions in soil salts.
- The lack of observable effects caused by the application of either the mined anhydrite or gypsum has several possible causes. These include the possibility that:
  - the products are not effective under these conditions due to low solubility or other issues
  - the 10 lb product /1000 sq ft rate tested, which is the rate recommended on product labels, is too low to produce statistically significant changes when used in a single application
  - leaching overwhelmed the effect of the product by moving all salts below the sampling area
- To follow up on the possibility that solubility was involved in the observed results, we obtained samples of commercial products based on gypsum (Allied Gypsum and Ultra Fine AG Gypsum Soil Conditioner), mined anhydrite (Cal-CM Plus Mini-Prilled) and on an uncharacterized source of calcium sulfate (Cal-Sul) for analysis. Results of solubility tests clearly demonstrated

that Cal-CM and Cal-Sul were much less soluble than the two gypsum products tested. While the low solubility of Cal-CM and Cal-Sul raises many questions about their potential as soil or water remediation tools, it is not the sole cause of the observed lack of efficacy in the field, since the highly soluble gypsum products also had no effect on soil salts or sodium.

- To follow up on the possibility that use rates were too low to produce detectable results, follow-up field tests will be conducted.
- Until results from planned field tests confirm the optimal rate and use patterns for calcium sulfate-based soil amendments, the benefit of these applications for sodium management cannot be determined. For the time being, the best tool for reducing sodium and other damaging soil salts on turfgrass is leaching.

### **Bottom line:**

Management of sodium on turfgrass is critically important for maintenance of turf health, disease management and good soil structure. One standard practice for reducing soil sodium levels involves the application of gypsum (calcium sulfate dihydrate), followed soon afterwards by a leaching event (Carrow and Duncan, 1998; Loveday, 1984). In this study, the value of leaching for sodium management was confirmed, with reductions in soil sodium ranging from 35% to 60% of the pre-treatment levels. The value of application of calcium sulfate products, on the other hand, was not confirmed in this study, with no additional reduction in soil sodium, beyond that seen due to leaching alone.

### Solubility evaluation:

One possible explanation for the observed lack of effectiveness in the field could be the product solubility, since dissolution of calcium sulfate into calcium and sulfate ions is necessary for the required chemical and physical reactions to occur. Rapid dissolution, within 5 minutes, is important, not only when calcium amendments are injected into irrigation water system (Burt, 1998), but also when used, as it was in this experiment, as a pre-leaching tool for sodium management. In this latter use, rapid release of calcium is necessary so that it can interact and displace sodium from the soil.

Our initial results showed that while the gypsum product rapidly (within 4 minutes) went into solution, the Cal-CM Plus and Cal-Sul went into solution slowly, with only 29 – 35% dissolved when compared to gypsum, even 24 hours after the start of the experiment. Follow-up solubility tests conducted at Brookside Labs confirmed this pattern.

It is likely that the Cal-CM Plus and the Cal-Sul may slowly dissolve in the soil, generating a slow-release of calcium, but further experiments would be required to confirm this. However, for use as a pre-leaching soil amendment, where the required time frame for activity is a few hours, the lower solubility of these products makes them a poor choice. We have requested from academics and anhydrite producers any data to support the use of non-gypsum sources of calcium sulfate for use in soil or water sodium management, but have not been able, either through our own searches of the scientific literature, or through these requests, to obtain any supporting data to date.

To help consumers discriminate among the somewhat confusing array of calcium sulfate based soil amendments, fertilizer labeling regulations in states such as California restrict the use of the word “gypsum” to products composed primarily of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  (calcium sulfate dihydrate). Products such as Cal-CM Plus or Cal-Sul, which are based on forms of calcium sulfate other than gypsum, must indicate this on their labels through use of the term “gypsum equivalent” rather than “gypsum”.

### Follow up investigations

As for the lack of field results with the more soluble gypsum product, it is clear that more work needs to be done to identify the optimal use patterns for gypsum, when used as a pre-leaching soil amendment. There is strong support in the scientific literature for the use of gypsum as a sodium management tool, but the majority of the literature identifies higher rates, and/or more frequent applications than were made in this study (Carrow and Duncan, 1998; Loveday, 1984; Richards, 1954; Wallace, 1995).

### Recommendation

Until results from planned field tests confirm the optimal rate and use patterns for calcium sulfate-based soil amendments, the benefit of these applications for sodium management cannot be determined. For the time being, the best tool for reducing sodium and other damaging soil salts on turfgrass is leaching.

## **References**

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## **Materials and Methods**

### Solubility study I (conducted at the PACE Turf laboratory)

A sample of Mini-Prilled Cal-CM Plus (Mini Prilled Calcium Sulfate, SGN 125, 23% calcium) was obtained from a bag of the material stored at a golf course. The Cal-CM Plus product was ground using a mortar and pestle into a fine powder. Allied Custom Gypsum (food and pharmaceutical grade gypsum) was used for comparison. One gram of either gypsum or Cal-CM Plus was added to 500 CC of distilled water on a stirring plate. Mixing was constant for 240 seconds. The solutions were left, without stirring, until 12 hours, when final readings was taken (solutions were stirred just prior to taking of readings to re-suspend any solids that had settled during the 12 hour period). Electrical conductivity of the solution was monitored using a Spectrum Technologies Field Scout EC meter, and visual observations were recorded.

### Solubility study II (conducted at Brookside Laboratories, New Knoxville, OH)

Samples of the following products were sent to Brookside Laboratories for solubility analysis:

- Cal-CM Plus Mini Prill
- Cal-Sul
- Ultra Fiine AG Gypsum soil conditioner

0.2 grams of each product was mixed in 200 ml of deionized water and shaken at 160 rpm. Aliquots were taken for analysis at time = 0 and then at 60, 120, 180, 240, 300, 360, 420, 480, 560 and 1440 minute time points. Each sample was then analyzed for soluble calcium and soluble sulfur using ICP (inductively coupled plasma) analysis.

### Field study I: Cal-CM Plus and leaching (conducted at Mission Viejo Country Club, Mission Viejo, CA)

Just prior to leaching, Mini-Prilled Cal-CM Plus (Mini Prilled Calcium Sulfate, SGN 125, 23% calcium) was applied to test areas at a rate of 10 lbs product/1000 sq ft. Leaching was carried out using customized cycle-soak program with a 5 minute cycle and a 10 minute soak to deliver approximately 6 inches of water. Rainbird 750 heads on a 60 ft spacing and 70 psi deliver 1.13 in/hour precipitation rate. Irrigation began 7:00 pm Sunday night, June 7, 2009 and finished 4:00 am Tuesday morning. Most heads finished Monday evening.

Treated greens were 3, 10 and 11. Non-treated greens were 1, 8 and 13.

Soil samples were collected before and after leaching using a  $\frac{3}{4}$  inch diameter soil probe to a depth of two inches. Individual cores were collected throughout each green to a volume of about 500 cc. The thatch layer was left on the core samples. The soils were then analyzed using Mehlich III extraction for most nutrients with KCl extraction used for nitrogen and 1:2 soil:water electrical conductivity evaluation converted to a saturated paste extract equivalent. Additional soil tests using ammonium acetate extraction (exchangeable cations) and saturated paste extraction (soluble salts) analyses were included. Soil analysis was conducted by Brookside Laboratories, New Knoxville, OH.

Details on each of the analytical procedures conducted appears in the Appendix under “Brookside Analytical Methods” and “Brookside Laboratories Soil Methodologies”, as well as in Missouri Agricultural Experiment Station’s NCR Research Publication 221 (1998).

Samples are labeled as “01” to indicate before leaching results and “02” to indicate results after leaching was conducted. The letter “c” indicates the samples are from the non-treated check greens and the letter “t” indicates that the samples were collected from the gypsum treated greens. For example, “01c before” refers to the non-treated check greens before leaching and “02t after” represents samples from treated greens after leaching.

#### Field study II: Gypsum and leaching (conducted at Mission Viejo Country Club, Mission Viejo, CA)

Just prior to leaching, Ultra Fine AG Gypsum Soil Conditioner was applied to test areas at a rate of 10 lbs product/1000 sq ft. Leaching was carried out using customized cycle-soak program with a 5 minute cycle and a 10 minute soak to deliver approximately 6 inches of water. Rainbird 750 heads on a 60 ft spacing and 70 psi deliver 1.13 in/hour precipitation rate. Irrigation began 7/13/09 and finished roughly 30 hours later on 7/14/09.

Treated greens were 3, 10 and 11. Non-treated greens were 1, 8 and 13

Soil samples were collected, labeled and analyzed as described above.

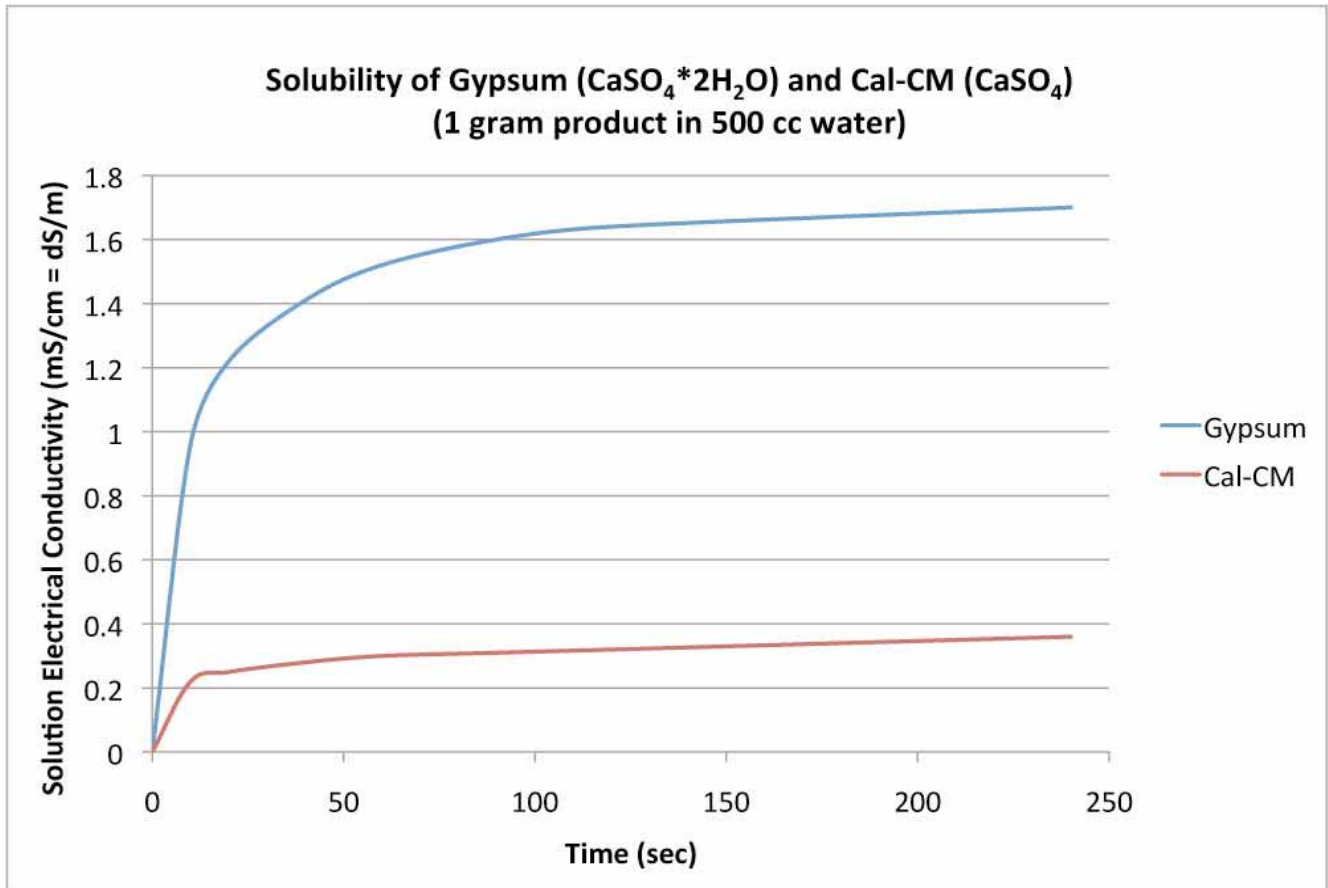
Details of the analytical procedures conducted appears in the Missouri Agricultural Experiment Station’s NCR Research Publication 221 (1998).

## Results: Solubility Study I

Increases in electrical conductivity (EC) over time are an indication of the degree of dissolution of the test products. The higher the EC, the greater the amount of product that has gone into solution. The speed of solubility of gypsum is most critical when the product is used to amend low salinity irrigation waters that have moderate or high SAR values. Finely ground materials are used to ensure that the product has dissolved into solution within 4 min. “With a good grind, manufacturers expect about 90% of the slurry to be dissolved within 1 minute and 100% dissolution within five minutes” (Burt, C.M. 1998). As seen in Figures 1 and 2, the EC of the gypsum solution increased rapidly in the first 100 seconds, and plateaued off at 1.71 dS/m (mean value) at the 240 second (4 minute) mark. In contrast, Cal-CM Plus ECs were at least four times lower at the 4 minute mark, with a mean value of 0.35 dS/m. After 12 hours, the Cal-CM Plus solubility had increased to 0.57 dS/m, but was still three-fold lower than the gypsum EC. These results were confirmed through visual observations of the two different solutions (Figure 3). The gypsum solution went from cloudy at zero time, to clear, within three minutes of product addition, while the Cal-CM solution remained cloudy for the 12 hour duration of the experiment.

## Figure 1. Increase in solution EC

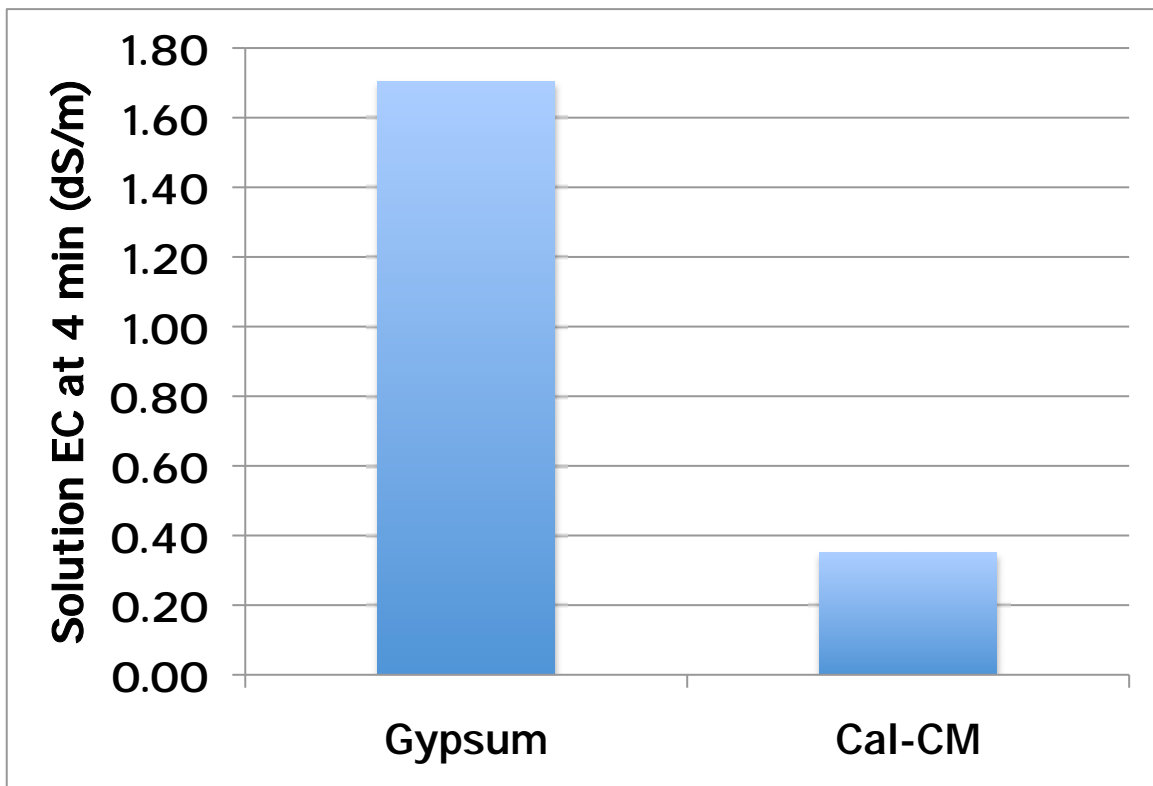
1 g gypsum or 1 g Cal-CM Plus was introduced into 500 cc water, and placed on a stirring plate. Cal-CM Plus exhibited low solubility in water. When monitored 12 hrs later, the solution salinity reached 1.79 dS/m for gypsum and 0.57 for Cal-CM Plus.





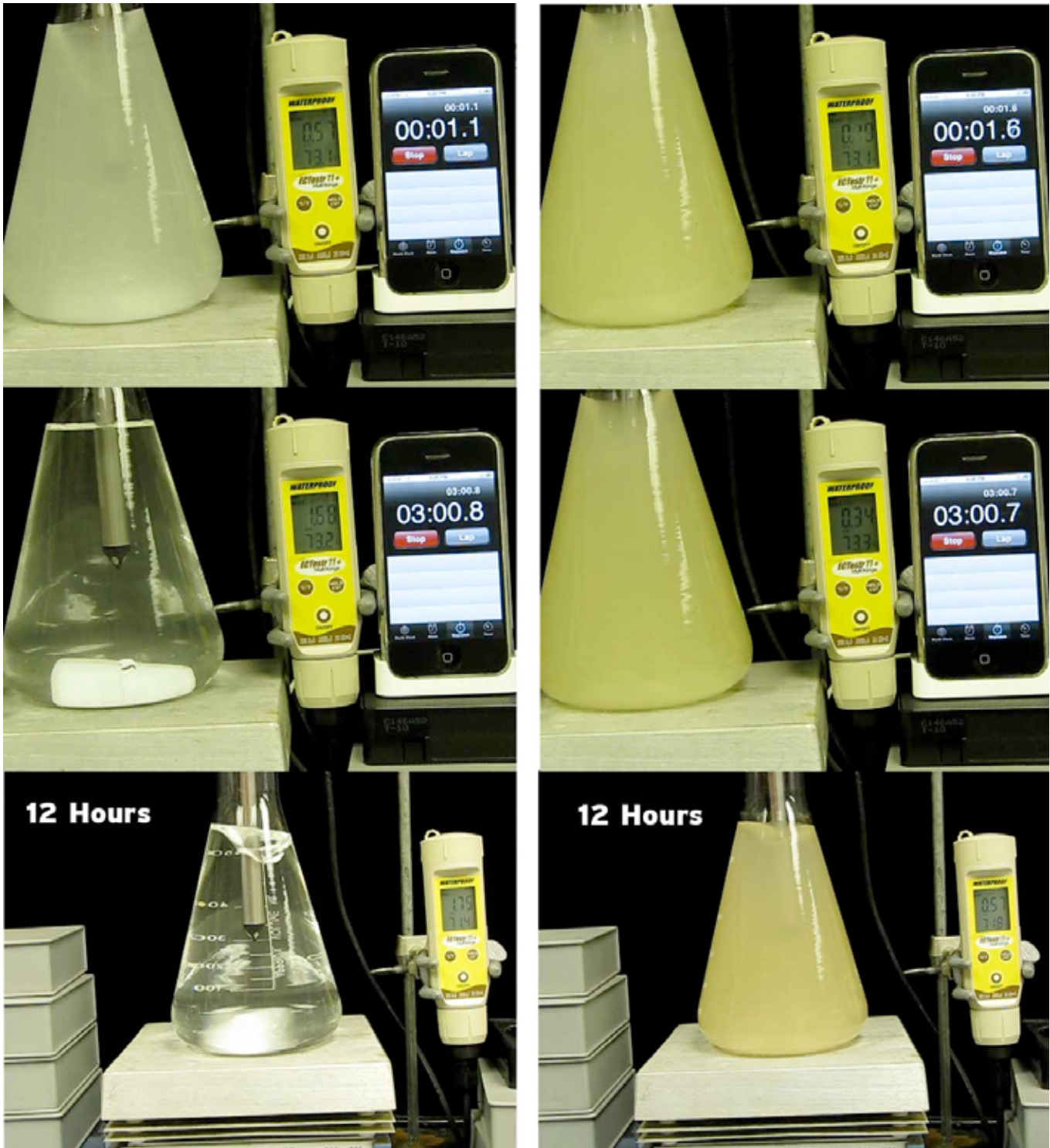
## Figure 2. Comparison of Solubility of Gypsum vs. Cal-CM Plus

Readings taken 4 minutes after 1.0 gram of each product was added to 500 cc of distilled water. Higher EC values indicate greater solubility. Results shown are an average of 4 replicates. Standard error of the means for gypsum and Cal-CM Plus were 0.003 and for 0.007, respectively. The means are significantly different from one another, with the probability due to chance <0.001 (Fisher's protected LSD).



### Figure 3. Visual and electrical conductivity comparison

Gypsum (flask on left) and Cal-CM Plus (flask on right) solubility. One gram of each product was introduced into the flask at time zero (top photos). Solubility and EC readings at 3 minutes and 12 hours after product introduction (middle and bottom photos) show that the majority of the gypsum solubilized, but that a significant portion of the Cal-CM Plus did not.

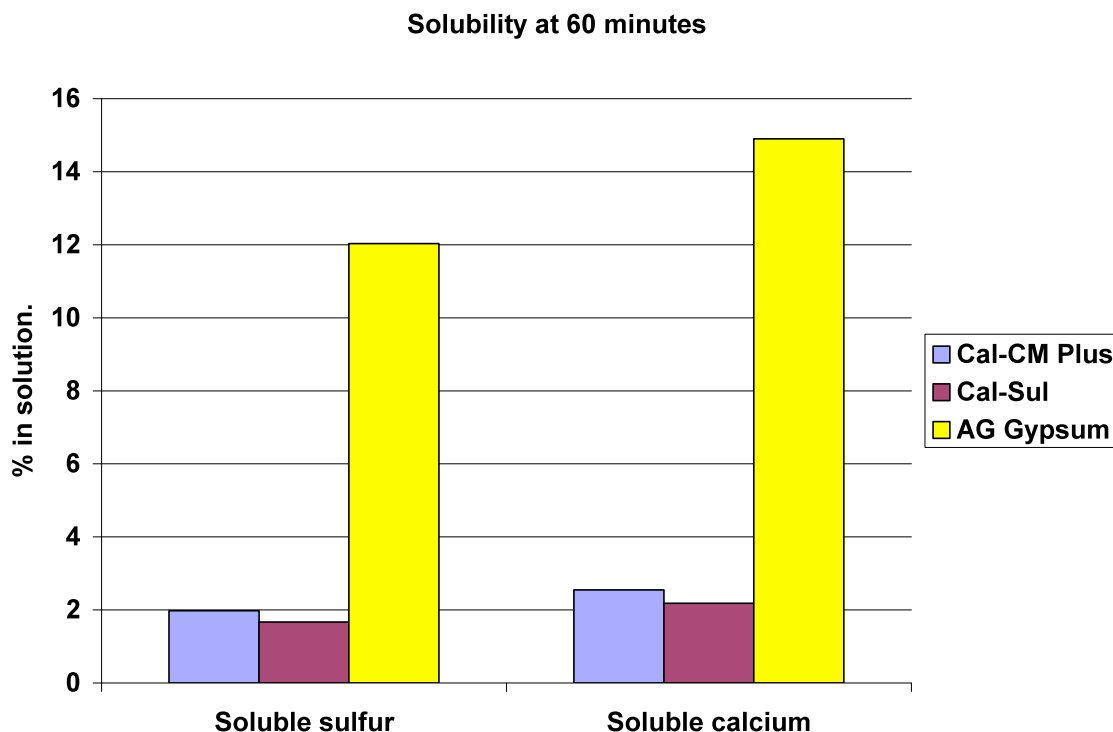


## Results: Solubility Study II

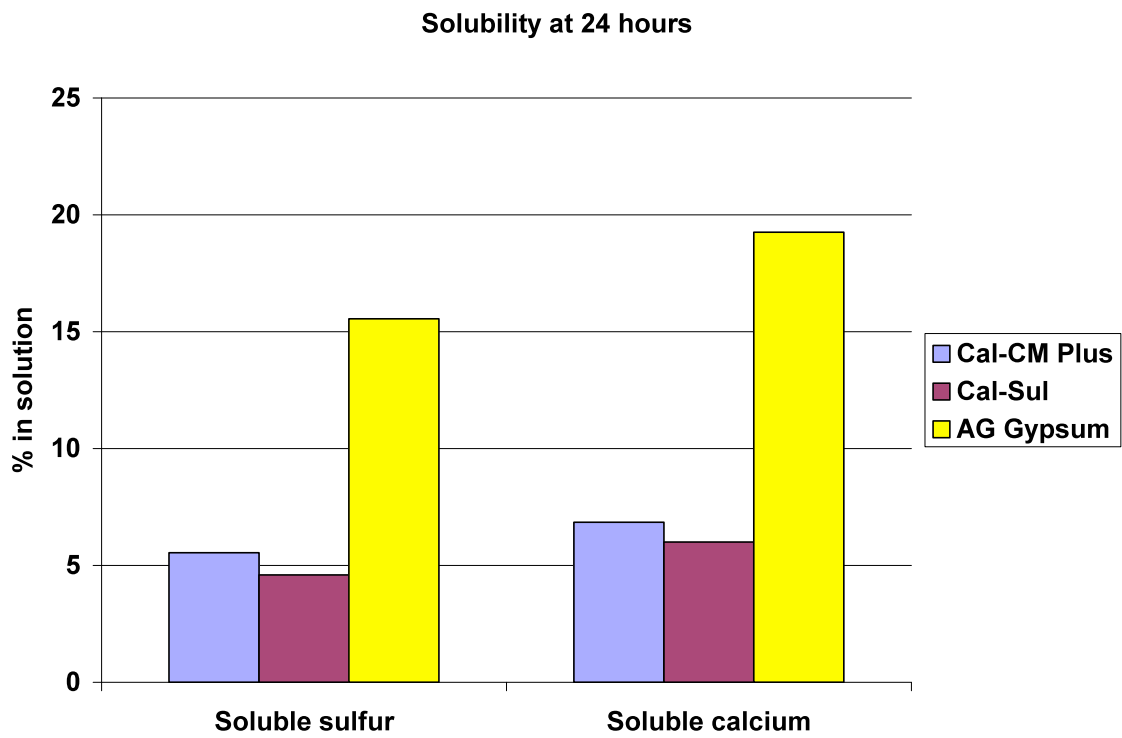
Results mirrored those generated in Study I. However, this study was conducted at Brookside Laboratories, where more time points and more extensive analytical procedures were conducted (see Materials and Methods).

The AG Gypsum product went into solution rapidly, with 14.9% soluble calcium and 12.03% soluble sulfur detected after 60 minutes (Figure 4). In contrast, the Cal-CM and Cal-Sul products went into solution slowly, with (respectively) 2.55% and 2.185% soluble calcium and 1.98% and 1.67% soluble sulfur detected after 60 minutes. At the 24 hour sampling time point (Figure 5), the AG Gypsum product was fairly well solubilized, with 19.26% soluble calcium and 15.55% soluble sulfur. In contrast, the Cal-CM and Cal-Sul products were only partially solubilized, with 6.845% and 6.005% soluble calcium detected respectively, and with 5.54% and 4.59% soluble sulfur detected, respectively.

**Figure 4. Solubility at 60 minutes**



**Figure 5. Solubility at 24 hours (1440 minutes)**



## Results: Field Study I

Leaching produced dramatic reductions in soil salts, SAR (sodium absorption ratio) and nitrogen -- whether or not the areas had been pre-treated with Cal-CM (Tables 1 – 3). Treatment with Cal-CM did not produce any additional reductions in soil salts, other than those produced by leaching, The same trends were observed regardless of the soil analytical method used (Mehlich 3, vs. saturated paste vs. ammonium acetate extraction).

**Table 1. Key soil quality factors – Standard extraction methods**

Values represent the average for three greens that were treated with Cal-CM (greens 3, 10 and 11, labeled “treated”) or non-treated (greens 1, 8 and 13, labeled “check”) before leaching (Before) and after leaching (After). Values are significantly different when there is less than a 5% ( $P < 0.05$ ) likelihood that the difference was due to chance. Means followed by the same letter are not significantly different using Fisher’s LSD.

Parameter	Check Before	Check After	Treated Before	Treated After
pH	7.3 a	7.3 a	7.3 a	7.4 a
Organic Matter (OM%)	2.5 a	2.9 a	2.6 a	2.9 a
Sulfur (SO <sub>4</sub> -S ppm)	167 a	82 b	167 a	77 b
Phosphorous (P ppm)	125 a	137 a	113 a	112 a
Calcium (Ca ppm)	1412 ab	1307 b	1477 a	1149 b
Magnesium (Mg ppm)	175 a	161 b	186 a	156 b
Potassium (K ppm)	286 a	226 b	290 a	214 b
Sodium (Na ppm)	249 a	160 b	260 a	150 b
Calcium percentage	65 a	68 a	66 a	67 a
Magnesium percentage	14 a	14 a	14a	15 a
Potassium percentage	6.9 a	6.1 a	6.6 a	6.4 a
Sodium percentage	10 a	7.4 b	10 a	7.6 b
Electrical Conductivity (EC dS/m)	2.5 a	1.5 b	2.4 a	1.5 b
Chloride (Cl ppm)	238 a	92 b	253 a	90 b
Boron (B ppm)	1.1 a	1.1 a	1.0 a	1.0 a
Iron (Fe ppm)	141 b	136b	152 a	144 ab
Manganese (Mn ppm)	109 a	111 a	124 a	120 a
Copper (Cu ppm)	2.0 a	2.1 a	2.2 a	2.0 a
Zinc (Zn ppm)	19 b	19 b	22 a	20 b
Ammonium nitrogen (NH <sub>4</sub> ppm)	7.1 a	9.7 a	8.2 a	7.7 a
Nitrate nitrogen (NO <sub>3</sub> ppm)	29 a	11 b	26 a	8.9 b
Total nitrogen (TOTN ppm)	36 a	21 b	35 a	17 b

<sup>1</sup>P = Fisher’s Protected LSD probability that the values are the same

**Table 2. Key soil quality factors – Ammonium acetate extraction**

Values represent the average for three greens that were treated with Cal-CM (treated) or non-treated (check) before leaching (Before) and after leaching (After). Values are significantly different when there is less than a 5% ( $P < 0.05$ ) likelihood that the difference was due to chance. Means followed by the same letter are not significantly different using Fisher's LSD.

Parameter	Check Before	Check After	Treated Before	Treated After
Calcium (Ca ppm)	1460 a	1477 a	1639 a	1444 a
Magnesium (Mg ppm)	192 b	183 b	216 a	183 b
Potassium (K ppm)	355 a	290 b	385 a	284 b
Sodium (Na ppm)	280 a	179 b	306 a	175 b
Sulfur (S ppm)	342 a	236 b	358 a	222 b

<sup>1</sup>P = Fisher's Protected LSD probability that the values are the same

**Table 3. Key soil quality factors – Saturated paste extraction**

Values represent the average for three greens that were treated with Cal-CM (treated) or non-treated (check) before leaching (Before) and after leaching (After). Values are significantly different when there is less than a 5% ( $P < 0.05$ ) likelihood that the difference was due to chance. Means followed by the same letter are not significantly different using Fisher's LSD.

Parameter	Check Before	Check After	Treated Before	Treated After
Calcium (Ca ppm)	258 a	99 b	232 a	97 b
Magnesium (Mg ppm)	68 a	27 b	61 a	27 b
Potassium (K ppm)	99 a	43 b	94 a	44 b
Sodium (Na ppm)	234 a	93 b	213 a	98 b
Sulfur (S ppm)	272 a	103 b	238 a	104 b
SAR (calculated)	3.18 a	2.14 b	3.21 a	2.22 b
Soluble salts (ppm)	2016 a	860 b	1922 a	885 b

<sup>1</sup>P = Fisher's Protected LSD probability that the values are the same

## Results: Field Study II

Leaching produced dramatic reductions in soil salts, SAR (sodium absorption ratio) and nitrogen -- whether or not the areas had been pre-treated with Cal-CM (Tables 4 – 6). Treatment with Cal-CM did not produce any additional reductions in soil salts, other than those produced by leaching, The same trends were observed regardless of the soil analytical method used (Mehlich 3, vs. saturated paste vs. ammonium acetate extraction).



**Table 4. Key soil quality factors – Standard extraction methods**

Values represent the average for three greens that were treated with gypsum (greens 3, 10 and 11, labeled “treated”) or non-treated (greens 1, 8 and 13, labeled “check”) before leaching (Before) and after leaching (After). Values are significantly different when there is less than a 5% ( $P < 0.05$ ) likelihood that the difference was due to chance. Means followed by the same letter are not significantly different using Fisher’s LSD.

Parameter	Check Before	Check After	Treated Before	Treated After
pH	7.23 ab	7.40 ab	7.17 b	7.50 a
Organic Matter (OM%)	3.40 a	3.17 a	3.17 a	3.27 a
Sulfur (SO <sub>4</sub> -S ppm)	227 a	69 b	190 a	59 b
Phosphorous (P ppm)	155 ab	160 ab	167 a	142 b
Calcium (Ca ppm)	1896 a	1421 ab	1658 ab	1176 b
Magnesium (Mg ppm)	271 a	189 b	234 ab	159 b
Potassium (K ppm)	307 a	210 b	283 a	186 b
Sodium (Na ppm)	365 a	182 b	341 a	159 b
Calcium percentage	64 a	67 a	64 a	68 a
Magnesium percentage	15 a	15 a	15 a	15 a
Potassium percentage	5.38 a	5.38 a	5.57 a	5.54 a
Sodium percentage	11 a	7.79 b	11 a	7.99 b
Electrical Conductivity (EC dS/m)	2.71 a	1.47 b	2.63 a	1.45 b
Chloride (Cl ppm)	283 a	87 b	287 a	79 b
Boron (B ppm)	1.20 a	1.00 a	1.13 a	0.97 a
Iron (Fe ppm)	188 a	140 b	171 a	138 b
Manganese (Mn ppm)	112 a	106 a	106 a	107 a
Copper (Cu ppm)	2.90 a	2.31 ab	2.61 ab	1.93 b
Zinc (Zn ppm)	25 a	20 b	23 ab	20 b
Ammonium nitrogen (NH <sub>4</sub> ppm)	7.10 a	7.53 a	7.03 a	8.77 a
Nitrate nitrogen (NO <sub>3</sub> ppm)	10 a	3.63 b	12 a	4.23 b
Total nitrogen (TOTN ppm)	18 a	11 a	19 a	13 a

<sup>1</sup>P = Fisher’s Protected LSD probability that the values are the same

**Table 5. Key soil quality factors – Ammonium acetate extraction**

Values represent the average for three greens that were treated with Gypsum (treated) or non-treated (check) before leaching (Before) and after leaching (After). Values are significantly different when there is less than a 5% ( $P < 0.05$ ) likelihood that the difference was due to chance. Means followed by the same letter are not significantly different using Fisher's LSD.

Parameter	Check Before	Check After	Treated Before	Treated After
Calcium (Ca ppm)	1546 a	1322 b	1416 ab	1299 b
Magnesium (Mg ppm)	229 a	180 b	210 ab	169 c
Potassium (K ppm)	303 a	218 b	287 a	196 b
Sodium (Na ppm)	325 a	179 b	311 a	167 b
Sulfur (S ppm)	392 a	212 b	332 a	202 b

<sup>1</sup>P = Fisher's Protected LSD probability that the values are the same

**Table 6. Key soil quality factors – Saturated paste extraction**

Values represent the average for three greens that were treated with Gypsum (treated) or non-treated (check) before leaching (Before) and after leaching (After). Values are significantly different when there is less than a 5% ( $P < 0.05$ ) likelihood that the difference was due to chance. Means followed by the same letter are not significantly different using Fisher's LSD.

Parameter	Check Before	Check After	Treated Before	Treated After
Calcium (Ca ppm)	166 a	75 b	210 a	77 b
Magnesium (Mg ppm)	50 a	22 b	62 a	22 b
Potassium (K ppm)	66a	25a	130a	26a
Sodium (Na ppm)	206 a	97 b	255 a	99 b
Sulfur (S ppm)	190 a	74 b	240 a	78 b
SAR (calculated)	3.59 a	2.52 b	3.96 a	2.56 b
Soluble salts (ppm)	1583 a	730 b	2125 a	755 b

<sup>1</sup>P = Fisher's Protected LSD probability that the values are the same