

## Experimental Evaluation of Lime Requirement Estimators for Global Sites

*Interim Work Plan, Africa Study 3*

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### Introduction

Aquaculture ponds with acid-bottom muds and soft waters are commonly treated with lime to raise soil pH and base saturation levels and to increase the alkalinity of the pond water to an acceptable level. Pond mud pH readings of less than about 6.0 or pond water alkalinities of 20 mg CaCO<sub>3</sub>/l or less are indications that a given pond needs to be limed (Boyd, 1979). Aquaculturists have used a number of methods (both agricultural and aquacultural) to estimate the amount of lime that should be added to ponds. Agricultural methods generally estimate the lime requirement (LR) for raising soil pH to a particular level; however, aquacultural methods go a step further and estimate the LR for raising pond water alkalinity to a desired level.

Study B of Work Plan Seven (revised) was designed to determine whether different LR estimation procedures produced similar results and to evaluate the suitability of different estimators for different types of soils by testing them in laboratory microcosms. Those experiments, reported in the Thirteenth Annual Technical Report (Bowman and Seim, 1995a; 1995b) demonstrated that different methods for the estimation of LRs produced varied results and that the use of some

methods did not always achieve the desired results in terms of alkalinity in the water column. This study, an extension of Study B of Workplan Seven, was designed to 1) investigate the use of artificial enclosures ("isolation columns") as in-pond test units for liming studies, and 2) compare the results obtained in such enclosures with results obtained in laboratory microcosms.

### Materials and Methods

A pond with acid soil and low-alkalinity water at Soap Creek (Department of Fisheries and Wildlife, Oregon State University, Corvallis, Oregon, USA) was selected for the installation of isolation columns (ICs). Soil samples were collected from the top 10 cm of soil in the 1-m deep area of the pond using a 5-cm PVC core sampler. The samples were air-dried and crushed to pass a 2-mm sieve, and subsamples were submitted to the Soil Physics Laboratory and the Central Analytical Laboratory (Department of Crop and Soil Science) at OSU for characterization. Analyses included determination of the sand, silt, and clay contents of the mineral fraction, pH, acidity, exchangeable

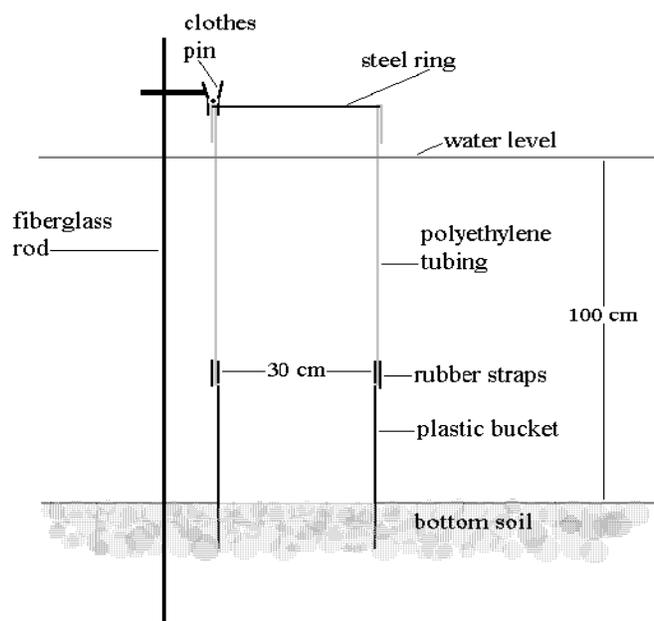


Figure 1. Design (not drawn to scale) of the artificial enclosures (isolation columns) used in the study. All materials used were locally available with the exception of the “layflat”, polyethylene tubing ordered from a plastics distributor.

bases (Ca, Mg, Na, K), SMP lime requirement, percent organic carbon, and total nitrogen content. Percent base saturation, percent organic matter (OM), and estimated cation exchange capacity (CEC) of the clay fraction were calculated using appropriate data from these analyses. Another subsample was used to determine pond lime requirement by the method of Pillai and Boyd (1985).

Isolation columns were constructed using 5-gallon plastic buckets (with the bottoms cut out), 29-cm diameter “layflat” polyethylene tubing, rigid 30-cm steel rings, plastic clothespins, and 6-ft fiberglass plant stakes (Figure 1). Six of these columns were pressed firmly into the pond bottom (minimum depth of 10 cm into the soil) in an already-filled pond at Soap Creek. The columns were placed along a pond-bottom contour where the water depth was approximately 1 m. Two treatments were applied to the columns in triplicate: three columns were limed according to the Pillai and Boyd (1985) estimate, and three columns were left unlimed.

Laboratory microcosms (MCs) were set up in a constant temperature room at the Oak Creek

Laboratory of Biology, Oregon State University, Corvallis, Oregon. Glass beakers with a capacity of 800 ml were filled with 750 ml of soft dilution water (alkalinity of approximately 18 mg  $\text{CaCO}_3/\text{l}$ , to approximate the alkalinity of the pond at Soap Creek). The appropriate amount of agricultural limestone was thoroughly mixed with 25 g of the soil from the Soap Creek pond and then added to the dilution water. The soil-lime-water mixture was stirred vigorously with a glass rod for ten seconds to begin the experiment. An unlimed treatment and a control (no soil or limestone) were also prepared. Each treatment was applied in triplicate. Water temperatures in the MCs were maintained between 23 and 26°C for the duration of the experiment. Samples of approximately 12.5 ml were removed after 1, 3, 7, 14, 21, and 28 days (on the same schedule as the samples from the isolation columns) for determination of total alkalinity. Alkalinity was determined according to the methods described in *Standard Methods* (APHA, 1989).

Water column samples were collected from the ICs and MCs after 1, 3, 7, 14, 21, and 28 days for total alkalinity determination. Samples were taken from the ICs at approximately 1100 hours and from the MCs at approximately 1400 hours on each sampling day. The experiment was initiated on July 12, 1995, and completed after 28 days, on August 9, 1995. The initiation, completion, and sampling dates for the isolation column component and the laboratory component of the experiment were the same.

## Results and Discussion

The physical and chemical characteristics of the Soap Creek pond bottom soil (also used in the MCs) are shown in Table 1. The textural class of the soil was clay (48.7%), and it had a CEC of 51.45 cmol/kg. The estimated CEC of the clay fraction was approximately 106 cmol/kg, which suggests that it is comprised mainly of 2:1-type clay minerals. The soil survey for the Benton County area (USDA, 1975) also places the soils at Soap Creek in the fine (35-59% clay), montmorillonitic (a 2:1-type clay mineral) class. These characteristics put the soil in the 2:1 Clayey class used in the POND<sup>®</sup> Version 2 (Bolte et al., 1994) soil classification system. The soil had a pH of 6.82, and its percent base saturation was approximately 90. The LR estimated for this soil by the SMP method (Shoemaker et al., 1961) was 1519 kg/ha, whereas the LR estimated by the Pillai and Boyd (1985) method was considerably higher at 5086 kg/ha.

Total alkalinity trends (mean treatment values) for samples taken from the Soap Creek pond (ICs and open pond) and the laboratory microcosms (MCs) are shown in Figure 2. The alkalinity trend for limed ICs shows an increase from the initial value of 18.31 to almost 30 mg/l (as CaCO<sub>3</sub>) during the first day and a subsequent gradual increase over the 28 days of the experiment, with a final alkalinity of 52.25 mg/l. Data from one replicate of the limed IC treatment was excluded from analysis because it deviated considerably from the mean for the treatment, suggesting some large source of error. Liming at the rate determined by the Pillai and Boyd (1985) method was successful in raising and maintaining total alkalinity to the desired level in the ICs. Visual inspection suggests that the alkalinity trend for unlimed ICs was similar to that of the open pond. In contrast with the trend for limed ICs, these alkalinities rose only slightly from the initial value of 18.31 mg/l, and neither ever exceeded 26 mg/l, indicating that the effect of the isolation column itself on alkalinity was minimal over the period of this experiment. This suggests that the use of isolation columns of this size and design may be reasonable for this type of testing; however, a Newman-Keuls multiple range test showed them to be significantly different at the 95% confidence level (Table 2).

In the laboratory, the alkalinity of limed MCs rose rapidly from an initial level of 18.09 to 29.95 mg/l during the first day, and it continued to rise until it peaked at 62.13 mg/l on day 14. Alkalinity then gradually declined to 52.02 mg/l on day 28. Alkalinity in unlimed MCs rose from an initial level

Table 1. Soil characterization data for Soap Creek Pond 7.

| Variable                                | Value      |
|---|------------|
| % Sand                                  | 11.5       |
| % Silt                                  | 39.8       |
| % Clay                                  | 48.7       |
| Textural Class                          | clay       |
| pH                                      | 6.82       |
| Acidity (meq/100 g)                     | 10.45      |
| Ca (meq/100 g)                          | 29.45      |
| Mg (meq/100 g)                          | 16.40      |
| Na (meq/100 g)                          | 0.20       |
| K (ppm)                                 | 90.00      |
| K (meq/100 g)*                          | 0.23       |
| Sum of Bases (meq/100 g)                | 46.28      |
| CEC (meq/100 g) (by analysis)           | 51.45      |
| CEC (meq/100 g) (by sum of cations)     | 56.73      |
| Estimated CEC of Clay (meq/100 g)       | 105.65     |
| Probable Mineralogy Class               | 2:1 clayey |
| Base Saturation (%) (by analysis)       | 90.0       |
| Base Saturation (%) (by sum of cations) | 81.6       |
| SMP Lime Requirement (kg/ha)            | 1519       |
| Pillay & Boyd Lime Requirement (kg/ha)  | 5086       |
| % Carbon                                | 0.55       |
| % Organic Matter**                      | 0.93       |
| Total Nitrogen                          | 0.04       |

\* Calculated as ppm K/391.

\*\* Calculated as 1.7 x %C.

Table 2. Newman-Keuls multiple range test results for alkalinities on day 28 of the isolation column experiment.

| Treatment                | Count | Mean Alkalinity (mg/l) | Homogeneous Groups |
|--------------------------|-------|------------------------|--------------------|
| Unlimed Microcosm        | 3     | 11.88                  | *                  |
| Laboratory Control       | 3     | 19.32                  | *                  |
| Unlimed Isolation Column | 3     | 23.45                  | *                  |
| Open Pond                | 3     | 25.70                  | *                  |
| Limed Microcosm          | 3     | 52.02                  | *                  |
| Limed Isolation Column   | 2     | 52.25                  | *                  |

\* Denotes a statistically significant difference.

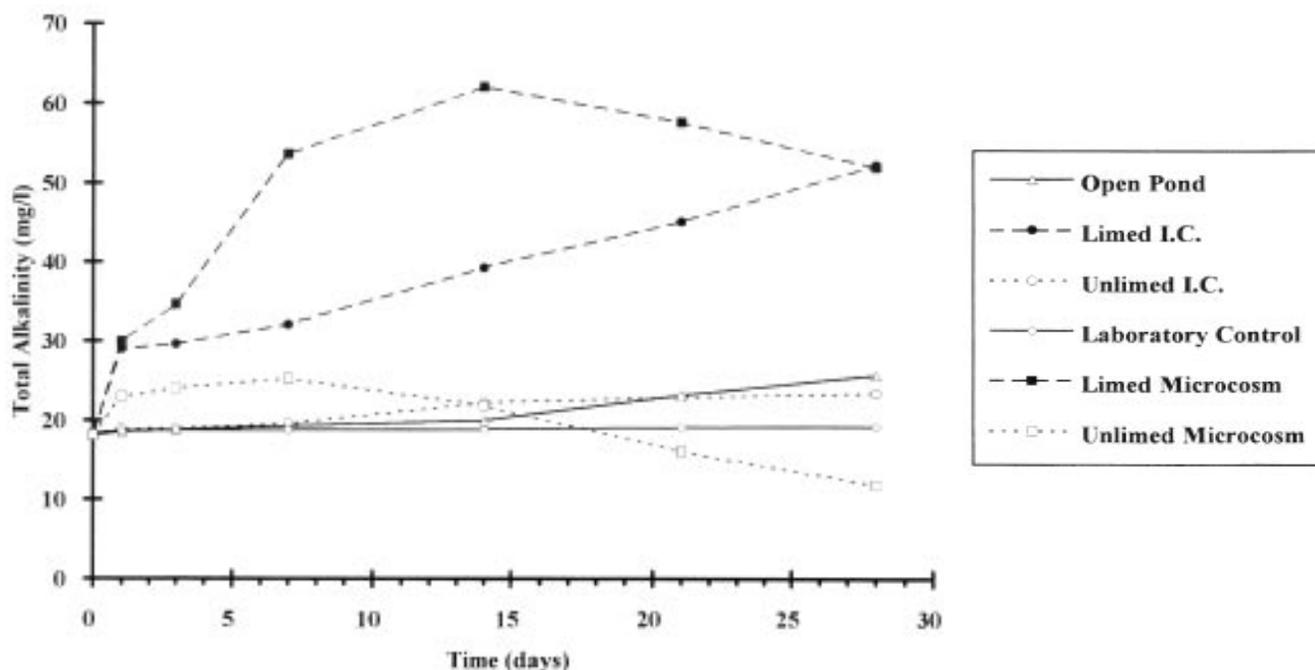


Figure 2. Trends in total alkalinity for limed and unlimed in-pond enclosures (isolation columns), open pond water, and limed and unlimed laboratory microcosms containing soil from the same pond. Limed and unlimed isolation columns and microcosms treated identically responded differently. The alkalinity trends in unlimed isolation columns were similar to the alkalinity trend of the open pond.

of 18.09 to 25.25 mg/l on day 7 but then dropped steadily over the remainder of the experiment to a level of 11.88 mg/l by the end of the experiment. This trend illustrates the depletion of alkalinity by acid soils discussed by Boyd (1979). Alkalinity in laboratory controls remained virtually unchanged throughout the experiment.

A comparison of results from the laboratory MCs with those of the ICs and the open pond reveals different responses to identical treatments in the two systems. The alkalinity increase in limed MCs during the first day matched almost exactly that of the limed ICs at Soap Creek (18.09 to 29.95 mg/l and 18.31 to 28.83 mg/l, respectively), but the two systems behaved very differently over most of the remainder of the experiment, diverging widely by day 14 before beginning to converge near the end of the experiment. By day 28, however, mean alkalinities in these two systems were not greatly different (Figure 2), with alkalinities in the ICs averaging 52.25 mg/l and alkalinities in the MCs averaging 52.02 mg/l. Unlimed MCs and ICs also

behaved differently; IC alkalinities remained nearly constant through the course of the experiment while MC alkalinities rose slightly before decreasing to below 12 mg/l by day 28. Multiple range analysis of day-28 alkalinities (Newman-Keuls, 95% confidence level) showed significant differences among all treatments except the limed ICs and the limed MCs, which formed a homogeneous group (Table 2). The noticeable differences among alkalinity trends in laboratory MCs and pond ICs suggests that one or both of these systems may not adequately simulate the effects of liming in real ponds although the similarity of the alkalinity trends in unlimed ICs and the open pond suggests that ICs of this design and size may have some potential as test units in aquaculture ponds.

Some mention should be made of the practical aspects of installing and using the ICs. Installation in already-filled ponds was found to be somewhat more difficult than originally anticipated because the normal algal bloom in the pond, together with

mud turbidity caused by working in the area, made it impossible to see the pond bottom or even the top of the bucket portion of the ICs during installation. Another problem was that both the polyethylene tubing and the top of the plastic bucket itself were very slippery, making it difficult to apply firm, steady pressure to the top of the bucket to press it down into the pond bottom. Finally, care had to be taken while inserting the fiberglass support rods into the pond bottom, as a slight misjudgment during this operation could result in the polyethylene tubing being punctured.

Some problems were also encountered after installation. The first was that it was necessary to adjust the level of the top of the IC to compensate for decreases or increases in water level in the pond, and this was difficult to do with the design tested. A second problem was related to an as-yet not understood phenomenon that occurred in the limed ICs. Towards the middle of the experiment, from about day 11 on, losses of water volume (evidenced by a partial inwards collapsing of the polyethylene column) were observed in these ICs but not in the unlimed ICs. One hypothesis was that a soil-limestone interaction occurred in the bottom soil, thereby sealing it and preventing evaporation replacement water from entering the IC from the bottom. Another hypothesis was that  $\text{CaCO}_3$  interacts with the polyethylene itself, sealing it against water movement. It might be useful to carry out further tests of isolation columns to determine if simple designs such as the one tested in this study can be improved for easier installation and maintenance, and to verify if in-column reactions are truly representative of the reactions that occur when the same treatments are applied to open ponds.

## Anticipated Benefits

The in-pond enclosures of the type tested appear to be suitable for conducting lime application experiments, although design improvements and further testing would be beneficial. If enclosures that are consistently reliable can be developed, they might be used for testing a number of different kinds of pond treatments within single ponds. This may result in reduced variability among experimental units and in considerable reductions in the amounts of pond space, time, and other costs required to conduct pond-based research.

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