MEASUREMENT OF LIME NEEDS OF MINED LAND AND SUBSEQUENT SOIL AND PLANT RESPONSE

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INTRODUCTION

A wide range in chemical properties may be found for mine spoils. Some of these may present difficult problems in reclamation, especially if the spoils contain high levels of acid-forming minerals such as pyrite. Even topsoil materials from naturally-occurring soils may be acid, and lime and other fertilizers may be required before permanent vegetation can be established. Therefore, it is very important to know something about the chemical properties of these materials prior to attempting to vegetate. In fact, in some cases alternative soil or spoil materials may be selected that would provide a more economical solution to reclamation rather than attempting to reclaim highly acidic spoil or soil materials.

Characterization data is essential in order to make accurate lime recommendations. These data must be based on representative samples from the site in question and supplemented with experience gained in previous reclamation activities of similar materials.

Once the properties of the potential overburden material and/or soil have been determined, the mine operator may have more than one alternative in reclaiming the site. Selection of the best material, from a physical and chemical viewpoint, to be placed on the surface to be revegetated should be considered as a first option. This choice may result in a higher mining cost, and therefore selection of the next best material, provided appropriate lime and/or fertilizers are added, may be more economical. However, one should not sacrifice short-term mining costs with longer-ranged reclamation costs, especially when lack of ground cover or other deficiencies in performance standards requires the operator to reseed and/or fertilize the site. This latter activity could restart the 5-year clock, thereby delaying Phase III bond release.

Over the past several years, at the University of Kentucky, we have had field experiments to evaluate lime recommendations for reclaiming both mine spoils as well as topsoiled areas. These research projects were all conducted in the western part of the state for at least two reasons. First of all, prior to passage of Public Law 95-87t most of the potential acid spoils occurred in western Kentucky, and secondly, I had other projects in this part of the state, hence, it was more of a matter of convenience in harvesting. We have applied this information to the eastern coalfields of Kentucky when acid spoils or soils are encountered.

The method used to determine the lime requirement is not as important as how it relates to field response to the applied lime. This response includes not only changes in pH and/or stability of pH with time, but also plant yield response and survival. Unfortunately, the accuracy of any method to predict the lime rate needed to reclaim a site may be controlled by the initial sampling methods rather than laboratory procedures. Poor techniques used in collecting mine spoil or soil samples, and subsequent sample preparation methods prior to the actual lab test, could result in erroneous data that very likely could give a poor correlation between lab data and relative success in the field. Suggestions for collecting samples are summarized elsewhere (Barnhisel, 1976a).

BACKGROUND

It is not the purpose of this paper to present a lengthy (or brief) literature review of available methods. Some of the work to be given here has been presented in more detail in earlier publications (Barnhisel et al., 1982; Barnhisel et al., 1984; and Powell et al., 1985). The approach taken was to try to establish a data base from which lime recommendations could be estimated from laboratory test results. At the time the first of these studies was established, the H_2O_2 method introduced by Smith et al. (1974) was the most popular. The acid-base accounting approach had not been fully developed. Furthermore, the H_2O_2 method we used was modified from that given by Smith and associates to make it more amenable to our laboratory conditions. A brief, generalized description of the H_2O_2 method as it is used in Kentucky will be given later.

The philosophy in making lime recommendations (Barnhisel, 1976b) evolved over a few years. However, this has changed somewhat upon passage of the Surface Mining Control and Reclamation Act (SMCRA) of 1977. In western Kentucky, under regulations of SMCRA, all highly toxic spoils are buried or at least covered with topsoil. The approach taken was to subject a sample to three tests: pH measured in water, pH measured in SMP buffer, and the H_2O_2 test. Whichever method produced the highest lime recommendation was used. Although samples exist in which any of these three tests could give the higher result, more frequently it was from the H_2O_2 method.

METHODS

Laboratory

The SMP buffer method used to estimate lime requirement was that of Shoemaker et al. (1961). The pH was measured as a 1:1 volume-weight ratio of either water or 1N KCl and the crushed soil or spoil sample.

Since the modified H 2 0 2 method remains unpublished (Barnhisel and Harrison, 1976), the key features are given as follows:

- 1. Grind about 25 grams of the sample fine enough to pass a 120 mesh sieve. Actual passage is not done, but grinding equipment and conditions were chosen for this to be the case.
- 2. Weigh out 5.000 g, place in a 1000 ml tall form beaker, and add 5-10 ml of deionized water.

- 3. Add 10 ml of 30% H_2O_2 and place in water bath at 900C. Allow oxidation reaction to take place, noting if the reaction was rapid enough to boil water. After about 1-2 hours, add another 10 ml portion of H_2O_2 , repeating observations on boiling, which usually takes place within 5 minutes of adding the H_2O_2 1 Continue to repeat H_2O_2 additions until the rapid boiling reaction does not take place. If more than 70 mls of H_2O_2 are required, discard the sample and begin again using a 1.000 g sample.
- 4. After an addition of H₂O₂ does not produce boiling, remove the beaker from the water bath and place on a hot plate operated at a "medium" temperature. Boiling should start very soon after removal from the 90°C water bath. Boil for 1 hr.; D- H₂O may be needed to prevent samples from boiling below a 25 ml volume.
- 5. Cool to room temperature, and using standard base, titrate the entire sample and the acid generated to pH 7.
- 6. Express Ag lime needed in terms of tons of lime per 1000 tons of spoil or soil, i.e., tons per acre.
- 7. The lime recommendation is made by multiplying the above value by 1.25 for all values of 20 or less. Lime recommendations greater than 25 are not suggested for single applications except for special cases.
- 8. The 1.25 factor adjusts for poor mixing conditions with the acid spoil or soil, poor lime quality, etc., and is used for cases where rapid pH adjustments are desired.

The Bray-1 phosphorus method was used to estimate available P (Bray and Kurtz, 1945). Extractable Kr Ca. and Mg were determined by the neutral ammonium acetate method given by Thomas (1982). After appropriate dilutions, these elements were determined with a Technicon' atomic absorption spectrophotometer.

Field Methods

River Queen, Site I. - The overall objective of this study was to evaluate lime incorporation methods for two lime rates on changes of pH. The spoil material was predominantly sandstone, with both the SMP and H 2 0 2 lime requirement equal to 67 Mg/ha (30 T/A). Since these spoil materials had been exposed following grading for several months, all sulfide minerals had already oxidized. The second lime rate used was 134 Mg/ha (60 T/A).

Three methods of lime incorporation were used. The lime for one set of plots was incorporated with a heavy-duty disk; the lime for a second set of plots was incorporated with a chisel plow; and for the third set of plots, the lime was not incorporated. Although both the disk and chisel plow were operated at two depths? data for only the shallow (10 cm or 4 inches) set of plots will be given here. These data, as well as a more detailed discussion, are published elsewhere (Barnhisel, 1977).

River Queen, Site 2. - Several objectives were involved as a part of this experimental research project, however, for this paper only one will be addressed, that being to determine the relationship between methods to predict lime recommendations and pH response with time. The spoil material at Site 2 was similar to that of Site 1, except the sandstone spoils were freshly mined and graded.

Four lime rates were used, 11.2, 22.4, 44.8, and 89.7 Mg/ha (5, 10, 20, and 40 T/A). The 11.2 Mg/ha rate corresponded to the lime rate based on the SMP buffer test, whereas the 44.8 Mg/ha rate was equal to that predicted by the H 2 0 2 potential acidity method. The other two rates were used to assist in calibration of the lime requirement methods. There were four replications of each lime treatment, and the lime was incorporated by disking to a depth of about 18 cm (7 inches).

Spoil samples were collected from the upper 15 cm (6 inches) every 2 months for over 2 years. A more detailed explanation of the experimental treatments and results are published elsewhere (Osborne, 1982, and Barnhisel et al., 1984).

Alston. - In 1978, a field study was begun at Alston Surface Mine in Ohio County, Kentucky. Details of plot construction are given elsewhere (Barnhisel et al., 1979). The objectives of this study were: (1) Determine what practices in relation to soil replacement depth are (or are not) needed to return reclaimed prime farmland to a state of equal or higher productivity, (2) determine if the graded overburden is suitable for rooting medium and for which specific crops, (3) determine the extent and severity of soil compaction, and (4) determine if a response to liming of the typically acidic subsoil materials would occur. An area of spoil was graded to a 2% slope and a 3 x 2 x 2 factorial experimental design was used to evaluate the following factors. The three main blocks were subsoil (B2t horizon) thickness variables. In one case this was 80 cm (32 inches) thick, or equivalent to the thickness for prime land. For the second treatment the subsoil was not replaced, and in the third case it has a thickness of 40 cm (16 inches). Each of these main treatments was divided into two areas prior to replacement of the topsoil (Ap horizon); to one-half, 45 Mg/ha or 20 T/A of Ag limestone was incorporated into the subsoil (or spoil) before the 20 cm (8 inches) of Ap horizon was replaced. After the soil was replaced, the area was divided into plots of 7.3 x 11 meters (24 x 36 feet) for the three test crops. There were 4 replications for each crop of corn, soybeans, and alfalfa. Each of these 72 plots was divided into half, and one-half was subsoiled to a depth of 50 cm (20 inches); the other half was not subsoiled. The soil reconstruction process was completed in 1978 and the area was seeded to a wheat cover crop.

Soil samples were collected periodically for analysis, and based upon these results appropriate fertilizers were applied for each crop using high management recommendation. Additional samples were collected for bulk density measurements.

Alfalfa <u>(Medicago sativa L.)</u> was established in April 1979 by broadcasting seed to a freshlydisked soil surface. Corn <u>(Zea mays L.)</u> was planted in May from 1979 to 1984. Soybeans <u>(Glycogen max L.)</u> was planted in 1979 through 1983. Wheat <u>(Triticum aestivum L.)</u> was planted in 1981 and was followed by soybeans, using a double-cropping system. In addition, 9meter-wide (30-foot) equipment turn-strips were seeded to tall fescue <u>(Festuca arundinacea</u> Schreb.), however, these areas were not harvested for yield measurements. A crop-rotation system was used for the corn-soybeans-wheat portion of the experiment. In 1984 (and 1985) the entire area was plowed and planted to corn.

Yields of corn, wheat, and soybeans were collected by a combine modified to harvest small plots. Alfalfa yields were collected using a rotary mower.

RESULTS AND DISCUSSION

River Queen. - After about one month, the first set of samples was collected from all plots. Due to the free iron in these spoils, there was a sharp color change separating the limed versus non-limed portion. For example, the upper 10 cm of the limed portion had a red color (2.5YR 4/6)r whereas the acid zone had a yellow color (10YR 7/8). The pH values for the limed zones were only slightly higher than those given in Table 1, data collected after one year.

Few significant differences occurred as a result of lime rate within a given method of incorporation for any given sampling depth. However, the pH's were significantly higher for the 0-5 and 5-10 cm sampling depths where lime was incorporated, as compared to being left on the surface. In all cases, the pH's for samples from the 0-5 cm sampling depth were significantly higher than those from the 5-10 cm depth. Similarly, the pH's for the 5-10 cm depth were higher than for the 10-15 cm sampling depth for those treatments where the lime had been incorporated.

Lime	Method of			Sample	Dep	th	in cm.		-	-
Rate	Incorporation	0	- 5		5 -	10		10 -	- :	15
Mg/ha		-			- pH	-				
67	Disked*	6.5	ab	A**	4.8	а	в	3.3	b	с
134	Disked	6.8	а	A	5.1	a	в	3.4	b	С
67	Chiseled*	6.2	b	A	5.1	a	в	3.8	a	с
134	Chiseled	6.8	a	A	4.9	a	в	3.4	b	с
67	Not Incorp.	5.4	с	A	3.8	b	в	3.0	b	в
134	Not Incorp.	5.1	с	A	3.5	b	в	3.2	b	В

Table 1 Effect of lime rate applied to acidic sandstone spoils and the method of lime incorporation on the pH at three sampling depths after one year.

* Lime incorporated into sandstaone spoils approximately 10 cm.

** Means with the same letters are not significantly different at LSD alpha = .10, use upper case letters for comparisons within a given lime treatment and lower case letters between lime treatments for a given sample depths.

The pH prior to applying lime was about 3.3. There was no evidence that lime had moved below the zone in which it had been incorporated. Lime applied to the surface was ineffective in changing the pH below 5 cm (2 inches). In this experiment, the surface-applied lime had become coated with a thin layer of iron oxide. It is believed that this precipitated

iron had sealed in the $CaCO_3$ preventing, or at least limiting, further neutralization of the acid.

The pH data collected from the second experimental site on the River Queen Mine are given in Table 2. These pH values represent the upper 15 cm (6 inches) and vary as a function of both lime rate and time. The initial pH for all plots prior to liming was 3.3. The pH had increased significantly for all lime rates after one month.

There were significant differences between the first three lime rates for all sampling periods, and *significant* differences also occurred between the highest two lime rates after 1 year had elapsed. The pH values had essentially reached their maximum values within one month. The pH values for the 11.2 and 22.4 Mg/ha rates declined significantly between 1 and 5 months and then remained more or less constant for 1.5 years. However, between the last two sampling dates, the pH's increased, and in the case of the 22.4 Mg/ha lime rate, this increase was significant.

Lime Rate	-)*			-		Time	in 5	Mo	nth	s -	-		1	5		2	7	-
Mg/ha					-				pH	-		-						-	-
11.2	3.3	a	с	5.1	с	A	4.	6 c	в		4.4	с	в	4.3	đ	В	4.7	đ	в
22.4	3.3	a	с	6.1	b	A	5.	3 b	в		5.4	b	В	5.1	с	В	6.3	c	A
44.8	3.3	a	в	6.6	ā	A	6.	6 a	A		6.7	a	A	6.6	b	A	6.9	b	A
89.7	3.3	a	в	6.9	a	A	6.	9 a	A		7.0	a	A	7.2	a	A	7.2	a	A

Table 2	Effect of	lime	rate	applied	to	acidic	sandstone	spoils	on	рН
	over time									

* Sampled prior to applying lime and incorporation by disking.

** Means with the same letters are not significantly different at LSD alpha = .10, use lower case letters for comparisons within a given sampling time and upper case letters between lime treatments for a given lime treatment.

The increase in pH between months 15 and 27 is the result of salt removal. When lime (CaCO₃) reacts with acid (H_2 SO₄), salts are formed such as gypsum (CaSO₄ 2H₂O). Salts may have an effect of lowering the pH, as was the case here. When these salts were removed by leaching, the pH increased. This salt effect may be more pronounced in some pH ranges than in others, as was the case between pH's 5 and 6, where the relative solubility of Al rapidly changes.

The most significant aspect of these two studies is with respect to the relationship between lime recommendations based on the SMP and H_2O_2 methods and actual field results. For the first site, lime recommendations for both methods were equal, and this was verified in the field. This is used as indirect evidence that all sulfide minerals had already oxidized.

There were significant differences between the recommended liming rates for the second site. The SMP method indicated 11.2 Mg/ha was needed, whereas the H_2O_2 method indicated 44.8 Mg/ha. It is obvious that the SMP method underestimated the lime needs, as the pH never reached the desired level; in this case we were applying rates needed to reach a value of pH 6.4.

The lime rate (44.8 Mg/ha) calculated from the H_2O_2 method raised the pH slightly above the predicted level, and this pH was maintained for at least 27 months.

Alston. - As indicated earlier, there were several objectives associated with this study, but the one of primary interest with respect to this paper was to evaluate the liming of the subsoil on crop yield. The effects of this treatment on changes in, pH are shown in Table 3. The 45 Mg/ha of lime that was incorporated into the upper 15 cm (6 inches) significantly increased the pH of this acidic subsoil by about 1 pH unit for both the prime farmland treatment (100 cm) and the treatment in which only 60 cm of soil was replaced. Although the pH's were significantly lower between the 15-30 and 30-45 cm sampling depths for both main treatments, it appears that there was some downward lime movement following the 8 years after the soils were reconstructed.

It is possible that some of the apparent lime movement could have resulted in sampling at a fixed depth interval. Although differences in thickness of the replaced topsoil or Ap horizon could have occurred, as well as variations in depth of lime incorporation, we believe that the differences in pH of this data are great enough to support the statement that lime had moved. More careful and more detailed sampling would be required, and is planned in the future, to verify this conclusion.

Exchangeable or extractable cations were determined from the same set of samples. Data for Ca are given in Table 4.

Depth cm		60 cm* Limed	No Lime	100 cm* Lime	 No Lime		
0	- 15	7.02 a A**	6.54 a B	7.11 a A	6.48 a B		
15	- 30	6.84 a A	5.78 b В	6.91 a A	5.94 Ь В		
30	- 45	6.20 b A	5.43 c B	6.15 b A	5.46 c B		

Table 3 Effect of liming the subsoil of two reconstructed soils on pH

- * Total soil depth = 20 cm topsoil over 40 cm or 80 cm of subsoil.
- ** Means with the same letters are not significantly different at LSD alpha = .10, use lower case letters for comparisons within a given lime treatment and upper case letters between lime treatments for a given sample depths.

Depth cm		 Lir	60 cm* Limed			ime	e	 Lir	100 cm* Lime					
0	-	15	4688	ab	A**	3709	a	с	4581	a	AB	4109	a	вс
15	-	30	5443	a	A	3187	b	A	5635	a	A	3591	b	в
30	-	45	4038	b	в	2823	с	с	4866	a	A .	3062	с	с

Table 4 Effect of liming the subsoil of two reconstructed soils on extractable calcium.

* Total soil depth = 20 cm topsoil over 40 cm or 80 cm of subsoil.
** Means with the same letters are not significantly different at LSD alpha = .10, use lower case letters for comparisons within a given lime treatment and upper case letters between lime treatments for a given sample depths.

The values of extractable Ca were related to the imposed treatments, as the highest Ca levels were from the limed, 1530 cm sampling depth. The Ca values from the 15-30 cm depth were

not significantly greater than those from the 0-15 cm depth, but they were significantly greater than those for the 30-45 cm depth in 3 of the 4 cases. Since the no-lime treatment had significantly more Ca in the 15-30 cm sampling zone than in the 30-45 cm zone, part of the difference observed for the lime treatments may be related to the chemical properties of the original soil at this site.

Data for Mg and K are not given, although differences did occur. The levels of K were not significantly different with respect to lime treatment, but differences were found as a function of sampling depth. The K levels from the topsoil series of samples were significantly greater than the K extracted at the 15-30 cm sampling depth. The Mg data were indirectly related to lime treatment, in that the no-lime treatment had higher Mg levels. This is a function of not using dolomitic lime, and the fact that soils with acid pH's can result in weathering of minerals such as chlorite, which increases the extractable Mg levels.

The effect that liming the subsoil had on crop yields is shown in Table 5. For corn, which is the critical crop in Kentucky with respect to release of Phase III bonds for prime farmland, liming the subsoil produced little beneficial response. If anything, liming reduced corn yield. When statistical comparisons were made, in 6 cases the no-lime treatments were higher, whereas in only one case did the limed treatment outyield its no- lime pair, and in 7 cases yields were not significantly different.

Crop	Year	60	100	cm		
		Limed	No Lime	Limed	No Lime	
			bu/	'A		
Corn	1979	89 A**	78 B	82 B	83 B	
Corn	1981	72 A	71 A	70 A	71 A	
Corn	1982	67 A	65 A	58 B	59 B	
Corn	1984	86 A	85 A	69 B	85 A	
Corn*	1984	100 A	102 A	88 B	105 A	
Corn	1985	62 C	80 B	60 C	. 86 A	
Corn*	1985	71 C	78 B	73 C	85 A	
G. Sorghum	1986	74 A	65 B	67 B	62 B	
G. Sorghum*	1986	65 C	57 C	93 A	77 B	

Table 5	Effect	of	liming	the	subsoil	of	two	reconstructed	soils	on
	crop yi	ield	ds							

* Corn or grain sorghum yields were from plots that were in alfalfa from 1979 through 1983. Corn yields for 1980 and 1983 are not given as values were low due to a severe drought at this site.

** Means with the same letters are not significantly different at LSD alpha = .10. Comparisons are valid only within a given year and across lime and soil depth treatments. Grain sorghum did respond to the lime treatment applied 8 years earlier in 3 out of the 4 comparisons. Grain sorghum is more sensitive to exchangeable Al than corn, and pH values for the 30-45 cm zone of the no lime treatment were low enough for this to explain the data. However, since even the surface soil of the limed treatment was higher, we cannot say conclusively that subsoil liming was solely responsible for the differences in grain sorghum yields given in Table 5.

One other significant observation was made among corn yields in 1984 and 1985, that being the influence which the previous crop had on yield. Although letters to denote significant differences between these data sets are not given in Table 5, in 1984, in all cases, corn yields following alfalfa were significantly higher than those following a corn - wheat - soybean rotation. In 1985, only the limed treatments where alfalfa had been the previous crop were significantly higher.

For grain sorghum, alfalfa having been grown previously had a significant and positive effect on the prime farmland treatments, but a negative yield response was obtained for more shallow treatment.

CONCLUSIONS

Lime Recommendations. - **Several** potential problems exist in making lime recommendations for neutralization of acid mine spoils (Barnhisel, 1976b). This discussion is with respect to the unusual nature of mine spoils and not with regard to the test employed. It is also assumed that the sample from which the recommendation is made represents the area. Mine land in which the overburden consists of more than rock stratum may be very heterogeneous. The most critical phase of a testing program is that of sampling (Barnhisel, 1976a).

Most lime recommendations for agricultural soils are based on a plow layer depth of about 17 cm. When large rates are required for mined land (> 30 metric tons per hectare), greater quantities are needed in order to react with upward movement of acid water from below the zone of lime incorporation. Lime recommendations which do not reflect the level of potential acidity that can be released upon the oxidation of sulfide minerals, such as pyrite, are subject to failure.

The lime recommendations which are the most difficult to make are for spoils that contain carbonates. This problem is due to the uncertainty of the particle size distribution of the natural carbonate materials. In the process of sample preparation, large natural fragments of limestone rock may be finely ground, and the data collected by either SMP or P.A. tests no longer represent the field condition. Lime recommendations for spoils that have been limed prior to sample collection may also result in erroneous values if the applied lime has become coated with a thin iron oxide layer.

Incorporation of Lime. - Improper or inadequate mixing of lime may be a serious limitation in obtaining reliable revegetation of acid mine spoils. Lime can be mixed by disking, and rapid neutralization reaction rates may be obtained. However, a heavy-duty disk is needed to mix the lime, and under the best conditions depths of only 15 to 20 cm may be obtained. Such operations are more easily accomplished if the spoils have been freshly graded or disked. Adequate mixing is more frequently achieved when the spoils are dry, provided the disk penetrates to the desired depth. If the spoils are damp when the lime is applied, repeated disking treatments are needed to insure adequate incorporation, and the second disking operation should be done after the spoils have dried somewhat and prior to the next rainfall event.

Droughty Characteristics of Acid Spoil. - Regardless of the amount of lime applied, and even with effective incorporation, any spoil which requires, a large lime rate (greater than 30 metric tons/ha) will tend to be droughty, even in humid climates. This droughtiness is not a deficiency of rainfall, but rather is due to limited root interception. Most plants require a rooting depth of more than 15 to 20 cm, while highly acidic spoils may have pH's less than 4 immediately below the zone of lime incorporation (see Table 1).

Toxicities and Deficiencies of Plant Nutrients. - The rooting zone of plants in acidic spoils. may contain toxic levels of ions such as Cu, Ni, Zn, Mn, or Fe, although lower levels of some of these elements are required for plant growth. As the result of a potentially wide range in pH of mined land, deficiencies of Mn, Mo, and B, as well as other essential elements, may exist for some plants. Acid spoils may contain large amounts of sulfate which may exist with several cations, such as Fe^{2+} , Ca^{2+} , and Mg^{2+} Potassium may be present but precipitated as jarosite [KFe₃(SO₄)₂(OH)₆], and in such cases the spoils may not supply enough K for adequate plant growth.

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