Limestone - Its Use in Effective Reclamation

by

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I. Introduction

Acid mine drainage is a high iron and sulfate drainage with a low pH and a high acidity that forms under natural conditions when certain coal seams and overlying rocks are exposed to the atmosphere. The oxidation of iron sulfides, occurring primarily as pyrite or marcasite which are contained in the coal and overlying strata, can produce acid drainages either by the release of iron, sulfate and acidity directly into water or by the initial formation of hydrous iron sulfate salts. Natural waters flowing over these rock surfaces dissolve the highly soluble salts so that the resultant waters are acidic and contain large amounts of iron and sulfate.

The rocks in the overburden may also contain calcareous material. The dissolution of these minerals, which may include $CaCO_3$, $MgCO_3$, and $Ca, Mg(CO_3)_2$, by natural waters produce alkaline drainages with attendant amounts of calcium and magnesium. The rate of dissolution of calcareous material is dependent on the pH, the partial pressure of carbon dioxide (P_{CO2}), surface area of reactivity, temperature, rate of water flow and the frequency of contact between the water and the minerals (Rauch and White, 1977; Geidel, 1979). Equilibrium conditions are attained quickly, but the solubility of these minerals at atmospheric conditions is much less than the solubility of sulfide minerals. This often results in situations where the alkalinity is overwhelmed by the acidity.

It has been found that in areas where the environment of coal deposition also produced abundant alkaline strata, the formation of acidic drainages following the disturbance of these rocks is minimal (Caruccio, et.al., 1977; Williams et.al., 1982). These findings suggest that the presence, and perhaps introduction, of alkaline material in an area may decrease the acid potential or at least be an effective neutralizing material.

II. Geochemical Considerations in Treatment Strategies

Caruccio, 1968, showed that the quality of mine drainages was a function of (1) the calcium carbonate content of the strata, (2) the pH of the ground water, (3) the mode of occurrence of the iron disulfide, and (4) the neutralizing and buffering capacity of the ground water. Studies based in eastern Kentucky produced similar results (Caruccio, et.al., 1977).

The presence of calcium carbonate determines to some extent the amount of alkalinity that infiltrating waters contain before coming in contact with the acidic material. If the alkaline material is antecedent of the acid producing material in the ground water flow path then the infiltrating waters have the opportunity to develop alkalinity which may effectively or partially neutralize the acidity produced by the toxic materials (Caruccio, Geidel and Williams, 1984).

The dissolution of calcareous material releases cations (predominantly Ca^{++} and/or Mg^{++}) and alkalinity, in the form of bicarbonate (HCO_3) or carbonate ($CO{=}_3$). The calcareous material, in the form of either limestone, dolomite or calcareous cement in shale or sandstone is naturally occurring in strata associated with the coal fields and is the primary, although not sole, contributor to alkalinity in the drainages and streams. Limestone, as CaC03, when placed in an aqueous environment at atmospheric conditions achieves solubility equilibrium conditions rapidly (Plummer and MacKenzie, 1974; Neuhaus, 1986).

The solubility of the calcium carbonate ($CaCO_3$) and dolomite ($CaMg\ (CO_3)_2$) is dependent on the partial pressure of carbon dioxide (P_{CO2}), pH, temperature, surface area of reactivity, and the length of time that water is in contact with the material, In an aqueous system at a P_{CO2} of $10^{-3.5}$, atmospheric conditions, the concentration of bicarbonate alkalinity from $CaCO_3$ at equilibrium conditions is approximately 120 mg /l as $CaCO_3$ with a pH of 8.4.

For dolomite, using a calculated equilibrium constant (K) of 10-19.71 the alkalinity, which can be generated from the following equation:

$$CaMg(CO_3)_2 + 2\ CO_2 + 2\ H_2O \rightleftharpoons Ca^{++} + Mg^{++} + 4\ HCO_3^-$$

is 40 mg/l as $CaCO_3$ (or about one third that of $CaCO_3$) and has an equilibrium pH of 7.9. However, limestones are generally impure with respect to calcite and may contain dolomite, quartz, and clays. Therefore, the alkalinity available from limestones is between that of calcite and dolomite (Neuhaus, 1986).

Changes in the P_{CO2} will also affect the equilibrium alkalinity values. Carbon dioxide levels in a surface mine may be elevated by the respiration activity of microorganisms in the soil and by the decay of organic matter. Rainwater or surface water contacting these zones of high CO_2 will entrain the gas and, in subsequent contact with calcareous material, increase the equilibrium alkalinity.

The pH of the water affects the solubility; the lower the pH, the greater the solubility of calcium carbonate. However, in surface mining environments, a low pH usually results from the oxidation of pyrite which releases iron, as well as acidity. When the low pH, high iron solution contacts the carbonate, the carbonate dissolution is enhanced, the pH rises and the iron precipitates on the carbonate. The precipitation of the iron on the carbonate surface prevents the coated limestone from further dissolution reactions.

From a standpoint of kinetics of formation, most of the alkalinity produced is generated during the initial chemical reactions and the rate of reactivity declines with time. Once the infiltrating waters achieve carbonate - bicarbonate equilibria, additional contact with calcium carbonate does not significantly increase the alkalinity of the system. Thus, regardless of the amount of calcium carbonate present in the strata, the solubility of the carbonate and, hence, the alkalinity of the system, is limited by geochemical constraints imposed by the equilibrium conditions.

All of the factors discussed above are dependent upon the availability of water. In addition, it is not only the quality or quantity of the ground or surface water coming in contact with the material but equally important the duration and frequency of contact. Limestone samples and a low sulfur gray shale were selected as alkaline producers. The results of the weathering tests showed that a definite relationship exists between the frequency of flushing and the cumulative alkalinity. The greater the frequency of flushing, the greater the cumulative alkalinity. However, each time a sample is flushed, regardless of the time interval in days, the concentrations of alkalinity in the leachate remains the same.

In contrast, when acid samples were subjected to similar leaching conditions increasing the interval between flushings did not produce a decrease in cumulative acidity, rather, regardless of the interval, the cumulative acidity remained relatively constant. This indicates that the oxidation of pyrite is a continuous process under these conditions and during the weathering tests the oxidation products accumulated in the chambers. When the samples are subsequently flushed, the soluble iron sulfate salts are placed into solution and the concentrations of acidity, which is dependent on the concentration of these salts, is proportional to he length of time between flushings. This proportionality is reflected in the daily acidity values which show that the greater the interval between flushings, the greater the acidity released.

III. Natural Alkaline Material and Study

A. Materials

The common minerals capable of contributing alkalinity in the coalfields occur as silicates (feldspars, micas, and clays) and carbonates (calcite, dolomite, and siderite). The mechanisms of bicarbonate alkalinity (mg/l $HCO\text{-}_3$)production is the main focus of this paper because of the large use in the mining industry of limestone rock material (consisting chiefly of calcite and dolomite) in treatment procedures for mitigating AMD.

The three carbonate minerals calcite ($CaCO_3$), dolomite ($CaMag(CO_3)_2$), and sidertie ($FECO_3$) constitute the bulk of carbonate material in the eastern U.S. coalfields. Previous field and laboratory studies indicate that P_{CO2} temperature, grain size and shape, petrology, solid solution, and aqueous chemistries may affect rates of alkalinity production and levels of equilibrium alkalinity.

Ca-Mg carbonate-containing material (limestone) in the absence of readily oxidizable iron-sulfides will produce alkaline waters with a pH 6.5 or greater. Weathering iron-sulfides in a carbonate poor environment will result in AMD. Environments containing both carbonates (or other alkaline materials) and sulfides may be either alkaline , acidic, neutral or variable in character.

The nature of the mine spoil effluent will be a, function of the dominant chemical reactions.

Such dominance is established as a result of rapid rates of reaction. The iron-sulfide dissolution reaction results in the production of iron and sulfate. Because $Fe_2(SO_4)_3$ is highly soluble, tending to precipitate only upon evaporation of AMD, the rate of iron-sulfide dissolution remains virtually constant provided an aqueous environment of low pH is present. The dissolution of $Fe_2(SO_4)_3$ is very rapid and complete upon contact with water. The rate of limestone dissolution, while initially more rapid than iron-sulfide dissolution, will decline quite markedly within a period of 24 hours as Ca^{+2} , HCO_3 saturation levels are approached. In theory, if the system were continually flushed with water, limestone dissolution, which outpaces the slower sulfide dissolution, would predominate resulting in alkaline effluents. If, as is more often the case, intermittent flushing of the mine spoil occurs, then formation of $Fe_2(SO_4)_3$ would result. Upon each new flushing the dissolution of $Fe_2(SO_4)_3$, a process much more rapid than limestone dissolution, would render initial through-flowing water acid. If water flow continued, the latter through-flowing water would become alkaline upon exhaustion of available $Fe_2(SO_4)_3$. If water flow ceased, however, the sulfide dissolution reaction would outpace limestone dissolution as Ca_{+2} $HCO\text{-}_3$ saturation levels are approached. Then, with evaporation, the formation of $Fe_2(SO_4)_3$ would again place the spoil system in the position to deliver yet another slug of acidity upon the next flushing of the spoil (Geidel, 1979).

By placing limestone above the spoil, Geidel (1982) was able to witness over several years a perceptible decrease in the rate of acid formation relative to the untreated spoil. Neutralization of AMD was also noted, though the effluent remained below federal standards. Recent work with alkaline (limestone plus strong base) trenches placed so as to intercept influent groundwaters has exhibited more promising results (Caruccio and Geidel, 1985). Observations of these natural acid-base reactions have produced a two pronged attack on the problem of AMD, that of AMD prevention and treatment of existing AMD.

Limestone is the least expensive of all bases considered and has the advantage of producing a low-volume high-density sludge requiring less frequent dredging of settling ponds. Limestone use is also least likely to adversely affect the environment. The disadvantages of using limestone for neutralizing AMD are the low solubility of limestone and the inability to raise effluents much above pH 6.5.

B . Study

Because of the coal industry's use of limestone as a neutralizing material to ameliorate AMD, this study was undertaken to (1) identify any differences in relative limestone reactivity and (2) to identify the primary causative factors responsible for the differences. To accomplish these objectives simulated weathering experiments were performed under controlled conditions. The relevant water chemistry characteristics of each experiment were monitored, and the resultant data compared to results from whole rock analyses.

The simulated weathering or leaching experiment was designed to observe variations in alkalinity generated by distinct alkalinity-producing rock types. The samples, primarily limestone, were leached in deionized water. The experiments were performed at ambient conditions ($P_{CO2} = 10^{-3.5}$ 25^0 C) as well as other temperatures (5^0C, 10^0C, 15^0C) and P_{CO2}'s (N = 1 atm). Aliquots taken from the leach water were analyzed for alkalinity, specific conductivity, pH and Ca^{++} and Mg^{++} contents. These quantitative observations, made at specific intervals over the duration of an individual experiment, were used to determine if the alkaline production capabilities of the different rock types were significantly different.

The whole rock analyses were performed to delineate the chemical, mineralogical, and petrological character of each sample type. To this end, analyses by X-ray diffraction, X-ray fluorescence, and atomic absorption spectrophotometry were performed. Petrographic analyses were made on both stained and unstained thin-section by polarizing microscope. Acid-base accounting, an analytical procedure heavily relied upon by the mining industry, was carried out on each sample. Samples consisting primarily of clay were analyzed for cation exchange capacity as were two limestone samples and a sideritic sample. Correlative comparisons were then made between the rock chemistry or petrology and resultant alkalinities generated by the leaching experiments.

All field samples used in this study were identified in the field for carbonate material by effervescence upon application of 1:3 HCL. The outer weathered portion (3CM) of each sample was removed with a rock hammer. Samples $FeCO_3$, NY, and all BM (Bellington Mine) samples were collected from cores in storage at USC repository and prepared in a fashion identical to the field samples. Bellington Mine samples were chosen on the basis of known alkaline production potentials and a non-acidic character as determined from previous leaching tests.

The testing of alkaline production under constant leaching conditions was accomplished by leachings carried out in a sealed plastic-plexiglas glove box. The internal volume of the chamber was 862.5 liters (100 cm x 125 cm x 69 cm) excluding internal apparatus and sample materials. A 1/4 inch plexiglas sheet (100 cm x 125 cm) was used on one wall to serve as both a window and an anchor for four latex accordioned gloves. The remaining walls consisted of three layers of heavy translucent plastic drop-cloth.

The temperature control system consisted of a 125 x 100 cm metal refrigerator coil (placed along the back wall of the chamber) through which water was pumped from a submersible pump located in a constant temperature bath outside the glove box. The temperature bath had an automated feedback system capable of maintaining $+/- 0.5^0$ C, in the bath itself, over a period of one week. The chamber temperature, as recorded by a thermometer immersed in 500 ml of water, was slightly more variable ($+/- 3^0$ C over a period of one week).

The gas control system consisted of compressed N and CO_2, gas cylinders controlled and monitored manually through the manipulation of regulator valves. A fan was placed in the glove box to homogenize gas composition and temperature. The feedback system consisted of periodically extracting 2 cc of gas from the chamber with a syringe inserted through a septum in the wall of the glove box. This sample of gas was then analyzed on a CARLE (AGC-111) gas chromatograph using a PORAPAK Q column and a helium gas train. Gas standards of 100 ppm and 1000 ppm CO_2, in N were used for calibration.

Analytical instrumentation consisted of a YST Scientific (model 32) conductivity meter and probe and a Fisher-Accumet (model 830) pH meter and probe. The meters, located outside the glove box, were attached to probes sealed inside the chamber. Standardized buffer solutions of pH 3 and 7 and a 0.001 N KC] solution were also placed within the chamber. A drain, consisting of a funnel, ringstand, and Tygon tubing, bent and filled with water so as to exclude outside air, was used in conjunction with squirt bottles, containing deionized water, to clean the pH and conductivity probes between measurements. A self-feeding 50 ml buret containing 0.02 N H_2SO_4 was attached to a ringstand in the glovebox and was supplied with additional titrant by a reservoir (with an ascarite air filter) resting on a shelf above the glove box. Measurements of pH, specific conductance, and acidimetric titrations were all performed in accordance with Standard Methods (1980) and individual operation manual instructions.

Gas-tight, air driven magnetic stirrers were used to provide agitation during leaching. Gas outlet tubes, delivering exhaust outside the chamber, were sealed at the chamber walls with clay and duct tape as were all wire and gas leads entering the glove box.

Equipment introduced at the beginning of each new experiment consisted of 600 ml Pyrex "leaching" beakers (each filled with 500 ml of deionized water and containing a 1 inch teflon magnetic stir bar), acid washed I mm mesh fiberglas sample nets (pretested for alkalinity, Ca^{+2}, and Mg^{+2} contributions). Each net, which held 100 mg of 2-4 mm sample, was placed in the glove box in an open plastic bag. At the start of a leaching run each sample net was suspended with the sample fully immersed in water, and attached to the leaching beaker with rubber bands. Pyrex funnels, ring supports, and cellulose mesh filters were used to remove suspended solids. 50 ml graduated cylinders were used to measure the volume of an analyzed aliquot and 50 ml Pyrex beakers were used to hold sample for analysis of pH and alkalinity. A 1/2 inch teflon stir bar was used to agitate the sample during analysis. Whirl-paks were used to store samples for future analyses and each sample was acidified with 1.0 N HCL kept in the glove box.

Ca^{+2} and Mg^{+2} determinations were performed on a Perkin-Elmer 306 atomic-absorption spectrophotometer. Calibration curves were constructed using standards prepared from Fisher certified standards. Necessary dilutions were performed utilizing an adjustable Finnpipette pipet and Eppendorf microliter pipets.

IV. Results

A. Whole Rock Analysis

The rock types involved in this study fell primarily into two mineralogic sub-groups, a calcite-dolomite group and a siltstone group consisting of fine-grained quartz and clay minerals. Siderite nodules and two sandstones were studied as well. The results of X-ray diffraction, performed on powdered samples (<43 microns) of each rock type, are shown in Table l . These results are qualitative, indicating the presence of detectable mineral phases (making up 3-5% of the sample by volume). the relative abundances of minerals within the untreated samples are ranked from left to right, in order of decreasing abundance, in Table A under the heading "Composition". All clays other than kaolinite were reported under the group classification "clays". X-ray diffraction analysis indicated that only low-Mg calcites (2-3 mole % magnesium) are present in the study samples.

Results from the LECO sulfur analysis showed that all samples contained less than 0.4% total sulfur.

Petrographic work was performed on all limestone samples. Two hundred point counts were performed on both stained (alizeran red) and unstained thin section. The limestone samples showed much petrographic variation. Mineral compositions ranged from 52.2% calcite-47.8% dolomite to 97.8% calcite-2.2% dolomite. Grain occurrence ranged from 0% to 62.3%. Cement (matrix) textures ranged from 73.5% micrite-26.5% sparite to 100% sparite.

Table 1 - Mineralogic Composition - X-Ray Diffraction Data

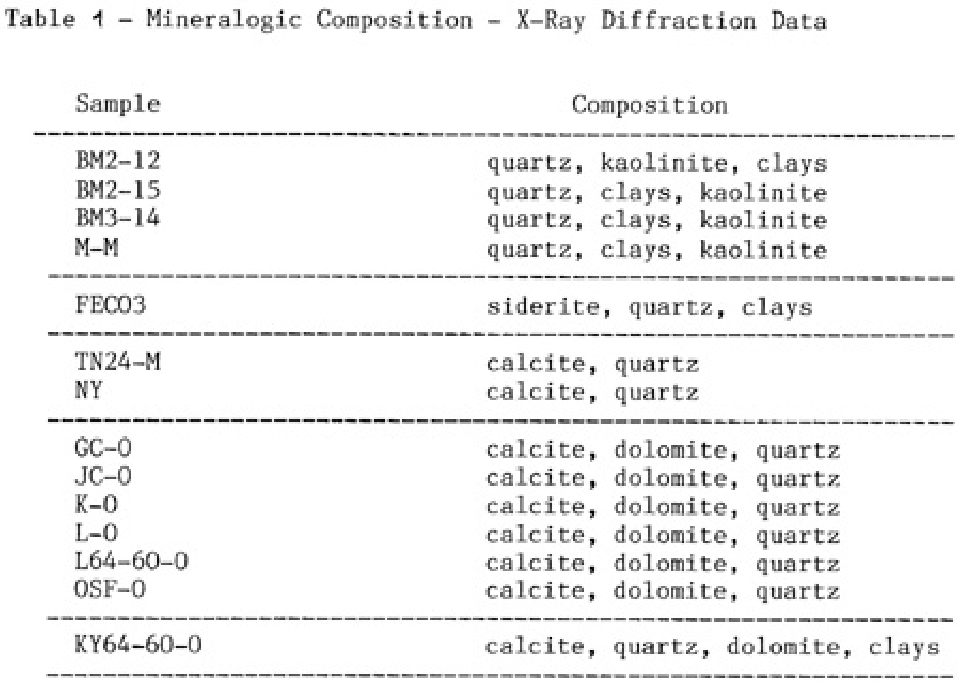

Table 1 - Mineralogic Composition - X-Ray Diffraction Data

Sample	Composition
BM2-12	quartz, kaolinite, clays
BM2-15	quartz, clays, kaolinite
BM3-14	quartz, clays, kaolinite
M-M	quartz, clays, kaolinite
FECO3	siderite, quartz, clays
TN24-M	calcite, quartz
NY	calcite, quartz
GC-0	calcite, dolomite, quartz
JC-0	calcite, dolomite, quartz
K-0	calcite, dolomite, quartz
L-0	calcite, dolomite, quartz
L64-60-0	calcite, dolomite, quartz
OSF-0	calcite, dolomite, quartz
KY64-60-0	calcite, quartz, dolomite, clays

Discernable differences in preferential dissolution behavior were evidenced between the limestone samples. The percentage of dissolved sample ranged from 8.2% to 91.3%, dissolved calcium concentrations range from 496 to 738 ppm while Mg concentrations went from 0.5 to 38.0 ppm.

B. Alkaline Production Potentials

The alkalinity producing capabilities of nine limestones, five clayey-siltstones, and one siderite nodule were measured in tests that generally ranged in duration from 100 to 300 hours. Most of these tests were performed at ambient laboratory conditions, though other tests were performed at 5^0 C, 10^0 C, and 15^0 C. The leachate parameters measured included alkalinity, specific conductance, and calcium and magnesium concentrations.

The differences in the amount of alkalinity produced by the various limestones leached at 25^0 C and $P_{CO2} = 10^{-3.5}$ atm were shown to be statistically significant for leaching times of 6, 12, and 144 hours. Differences in the production of alkalinity between the limestone samples became yet more perceptible as the leaching periods were extended to 300 hours (Figure 1). Two of the siltstone samples and the siderite sample surpassed the limestone samples in alkalinity produced at about 150 hours elapsed leaching time (Figure 2). At 300 hours one of the siltstone samples and the siderite sample had produced nearly twice as much alkalinity as had any of the limestone samples. Two of the siltstone samples produced much less alkalinity than the other samples throughout the leaching period.

Although the difference in the amount of alkalinity produced was statistically significant, the absolute values of alkalinity ranged from 55 mg/l to 72 mg/l. For all intents and purposes, when one considers that the acid levels, the treatment for which the use of limestone is indicated, are on the order of several hundreds, the variation in alkalinity production becomes impractically different. During the course of this study, and based on the samples used, we discerned no major differences in alkalinity production or neutralization potential and, accordingly, see not need to consider the "quality" of limestone as an important consideration in reclamation measures.

V. Reclamation Strategies

In our study recently completed, the amount of alkalinity which would be produced from various limestones varied during the first few hours, but produced similar amounts of alkalinity at equilibrium conditions. After approximately 200 hours, all of the limestones, each containing differing amounts of dolomite, quartz and clay, produced solutions containing between 55-72 mg/l alkalinity. This suggests that the quality of limestone used in reclamation is not an important factor in the success of the reclamation strategy.

A second consideration for the effective use of limestone in reclamation is the placement of the material. The limestone can be applied in any of three locations within the backfill.

Limestone may be placed either on the mine floor, mixed or blended with the overburden, or spread as a surface or near surface application. Limestone placed on the mine floor has shown little success in preventing or reducing acid drainages (Geidel and Caruccio, 1985). Water accumulates on the mine floor and if it is acidic, either from infiltration of water through toxic material in the backfill or from an acid producing pavement, the limestone is rapidly armored with iron and carbonate dissolution is diminished. The placement of the material and the effect on the limestone is similar to that which is observed if limestone is placed in an acid pool or stream.

The second location for placement of limestone, i.e., mixed with the backfill material, may be successful if the mixing occurs prior to pyrite oxidation. Several studies have indicated that the presence of significant amounts of carbonate can inhibit the oxidation of pyrite. Temple and Koehler (1954) showed that calcium and magnesium (presumably as a calcium-magnesium carbonate) were present in stable pyrite nodules but absent in the reactive ones. However, after the nodules were immersed in a mild hydrochloric acid solution, which dissolved the carbonate, the stable nodules began to oxidize. This suggests that once acidic waters are formed, the pyrite is effectively stripped of its protective carbonate and pyrite oxidation is enhanced. Additionally, Caruccio, et.al., (1981) showed that rock samples containing both significant amounts of carbonate and pyrite did not produce acidity during simulated weathering tests until the carbonate had been leached out of the sample. This further indicates that the carbonate may have an inhibitory effect on the oxidation of pyrite.

Limestone application on the surface or near surface horizon may be effective in a mine when the acid production potential is very low or when the limestone is applied immediately after construction of the backfill. The advantages to application in this position are that the limestone will not become coated with iron because it is positioned antecedent of the acid material and the limestone is wetted or flushed with each rainfall event so that maximum leaching and minimum flushing intervals occur thereby maximizing the available alkalinity (Geidel and Caruccio, 1983).

This technique, however, is not well suited to the reclamation of older mines where acid drainages have been established. Application rates of 40-65 tons/acre were applied to an unreclaimed surface mine which produced acidic drainages. The acidity of the drainages, when compared to controlled sites which received no limestone treatment, decreased and the decrease would be attributed to both neutralization and a decrease in the oxidation rate of pyrite. However, insufficient alkalinity was released to produce either neutral or alkaline discharges from the site (Geidel and Caruccio, 1983). In another older mine, sodium carbonate in conjunction with limestone was required to significantly decrease the acidity in seeps emanating from the toe of spoil (Caruccio, Geidel, and Williams, 1985). This treatment method provided positive improvements in drainage quality.

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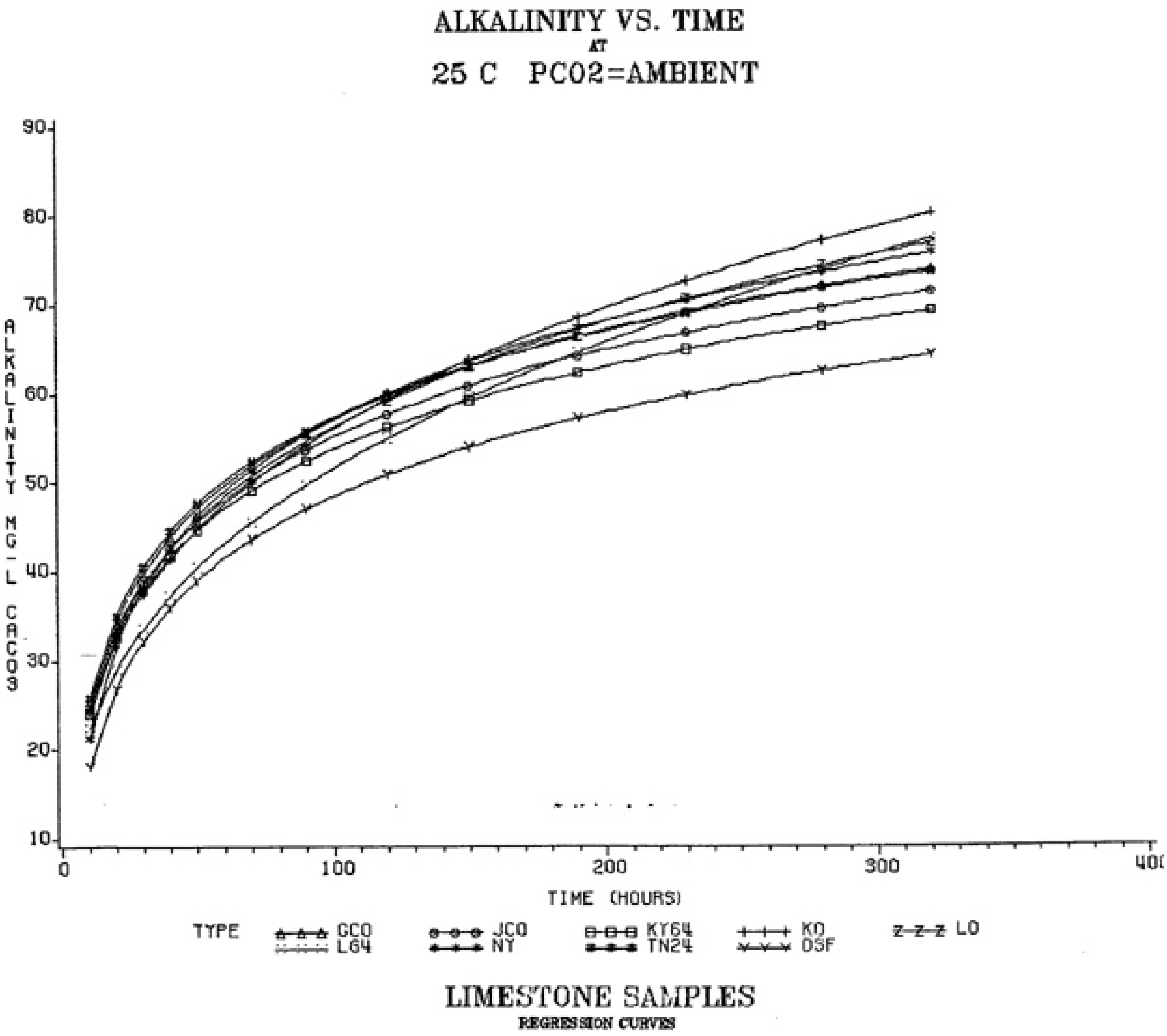

Figure 1

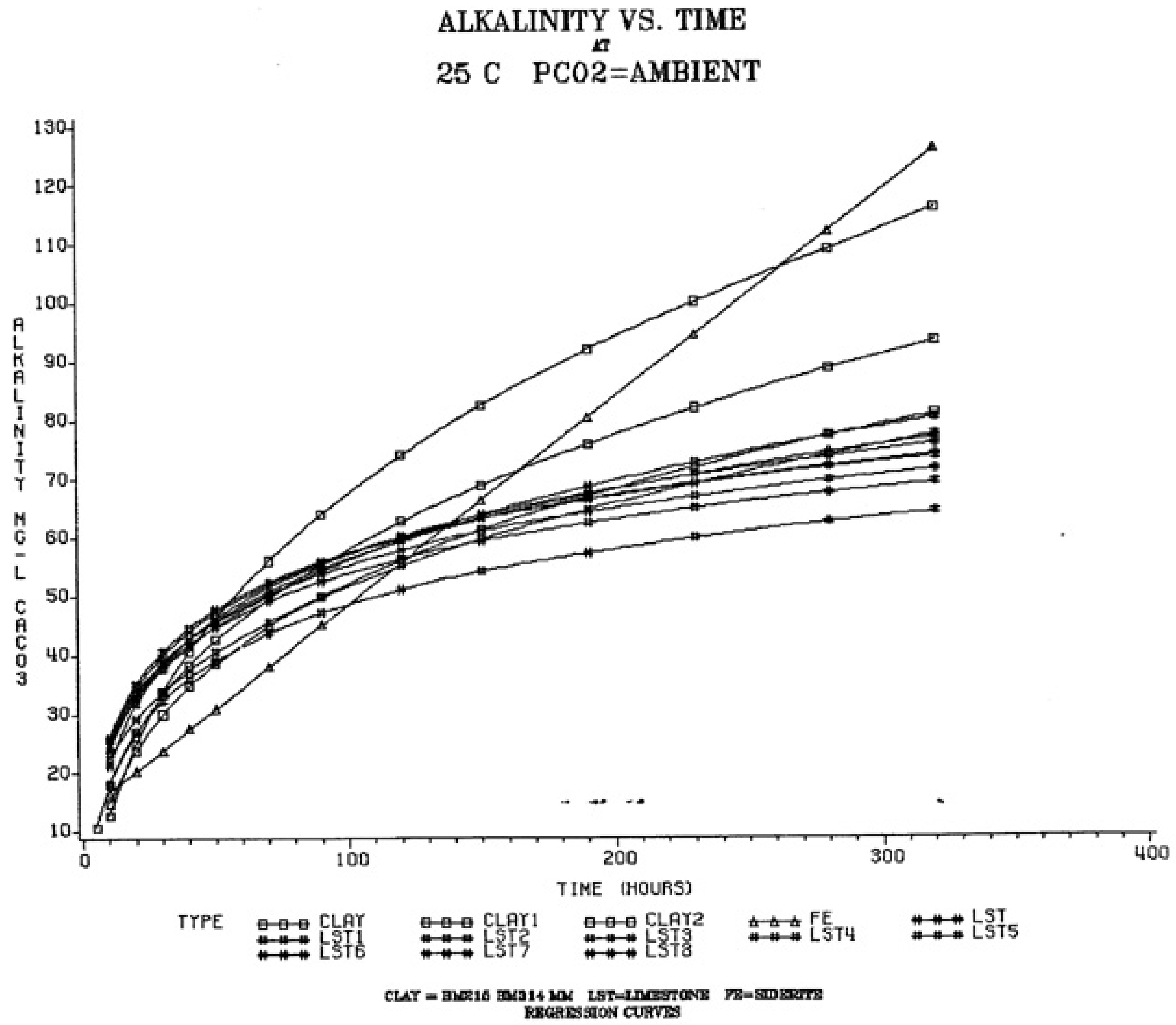

Figure 2

Figure 2