

Effect of Hydrogen Peroxide Pretreatment on Particle-Size Analysis

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Introduction

It has been an accepted procedure to pre-treat soil samples with H_2O_2 to destroy organic matter prior to particle-size analysis. Robinson (1922) reported as much as a fourfold increase in percent clay measured after destruction of organic matter with H_2O_2 . His explanation was that the organic matter acted as a cementing agent between the particles. Bayer (1930) also found an increase in clay measured after H_2O_2 pre-treatment, but these increases were not as great as those reported by Robinson.

Beale (1939) found that for lateritic soils, the H_2O_2 pre-treatment could be eliminated without affecting the result of the analysis. Winters and Harland (1930) also found that the H_2O_2 pre-treatment could be eliminated if the samples were washed with HCl.

Kilmer and Alexander (1949) concluded that because of the variety of soils received by the various U.S. Department of Agriculture Soil Survey Laboratories, that the H_2O_2 pre-treatment would be performed on all soils. This has been continued in the National Soil Survey Laboratory (Soil Survey Staff, 1967).

The H_2O_2 pre-treatment can affect the mineralogy of the soil material because of the reaction with MnO_2 (Jackson, 1973). This is an important consideration but does not have a great effect on particle-size results.

The H_2O_2 pre-treatment is a time consuming operation, and its elimination could significantly increase the number of samples completed for a given period of time. A set of analyses were made on selected soils with and without hydrogen peroxide pre-treatment to compare results and determine whether or not the H_2O_2 pre-treatment is needed and under what conditions it might be eliminated.

Materials and Method

The Purdue Soil Characterization Laboratory analyzes samples collected and described by soil scientists of the Cooperative Soil Survey. For each horizon, particle-size distribution is determined without H_2O_2 pre-treatment. In addition, horizons with more than 0.5% organic C, usually one to four horizons per profile, are pre-treated with H_2O_2 before analysis. For this study, 262 soil samples, previously analyzed, both with and without H_2O_2 pre-treatment, were randomly selected for comparison. These samples were from 140 soil profiles which represent a good cross section of Indiana soils. Clay and organic C contents were determined by the pipette and chromic acid oxidation methods, respectively (Franzmeier et al., 1977).

Results and Discussion

Linear regressions were calculated for clay measured with and without H_2O_2 pre-treatment for the nine possible combinations of three soil drainage conditions and three organic C content classes. The drainage classes used were well and moderately well and moderately well drained, somewhat poorly drained and poorly

TABLE 1. Linear Regression Equations and Correlation Coefficients ($r \pm$) Comparing Clay Content Determined with (x) and without (y) H_2O_2 Pre-treatment.

| Natural Soil Drainage | Soil Organic Carbon | Regression Equation | r^2 | Number of Samples |
|--------------------------|---------------------|---------------------|-------|-------------------|
| Well and Moderately Well | 0-1 | $y = 0.98x - 0.33$ | 0.98 | 67 |
| Well and Moderately Well | 1-2 | $y = 0.85x + 0.99$ | 0.94 | 67 |
| Well and Moderately Well | >2 | $y = 0.78x - 1.96$ | 0.93 | 13 |
| Somewhat Poorly | 0-1 | $y = 0.98x - 0.45$ | 0.99 | 23 |
| Somewhat Poorly | 1-2 | $y = 0.94x - 0.44$ | 0.98 | 21 |
| Somewhat Poorly | >2 | $y = 0.96x - 3.26$ | 0.92 | 9 |
| Poorly | 0-1 | $y = 1.00x - 0.17$ | 0.99 | 22 |
| Poorly | 1-2 | $y = 0.98x + 0.17$ | 0.99 | 20 |
| Poorly | >2 | $y = 0.93x + 0.04$ | 0.98 | 20 |
| | | | | 262 |

drained. The organic C classes were: 0-1%, 1-2%, and >2%. Table 1 contains the results of these calculations. Each of these sets of data has an r^2 value greater than 0.92. This does not mean, however, that the clay content determined with and without H_2O_2 pre-treatment are in agreement.

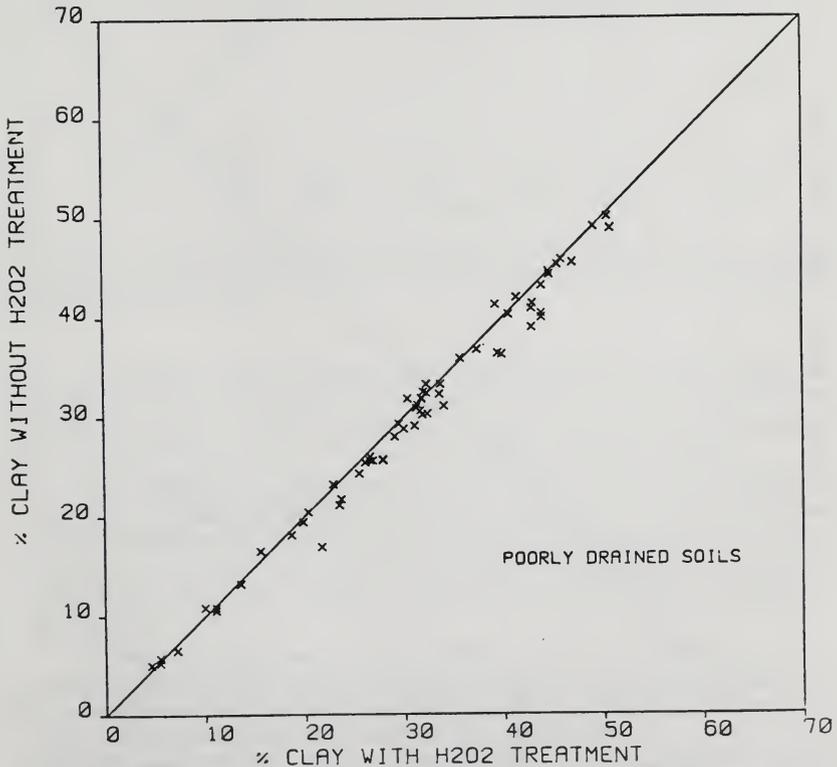


FIGURE 1. Comparison of Percent Clay Determined With and Without H_2O_2 Pretreatment for Poorly Drained Soils.

TABLE 2. *Expected Clay Content Calculated from Linear Regression Equations for Soils Containing 20 and 50% Clay Assuming No H₂O₂ Pretreatment.*

| Natural Soil Drainage | % Organic C | | |
|---|----------------------|----------------------|----------------------|
| | 0 - 1 | 1 - 2 | > 2 |
| Soil contains 20% Clay after H ₂ O ₂ Pretreatment | | | |
| | Calculated % Clay | Calculated % Clay | Calculated % Clay |
| Poorly | 19.9 | 19.7 | 18.6 |
| Somewhat Poorly | 19.5 | 18.5 | 15.9 |
| Well and Moderately Well | 19.2 | 18.0 | 13.8 |
| Soil contains 50% Clay after H ₂ O ₂ Pretreatment | | | |
| Poorly | 50.2 | 49.1 | 46.4 |
| Somewhat Poorly | 48.8 | 46.8 | 44.7 |
| Well and Moderately Well | 48.6 | 43.5 | 37.3 |

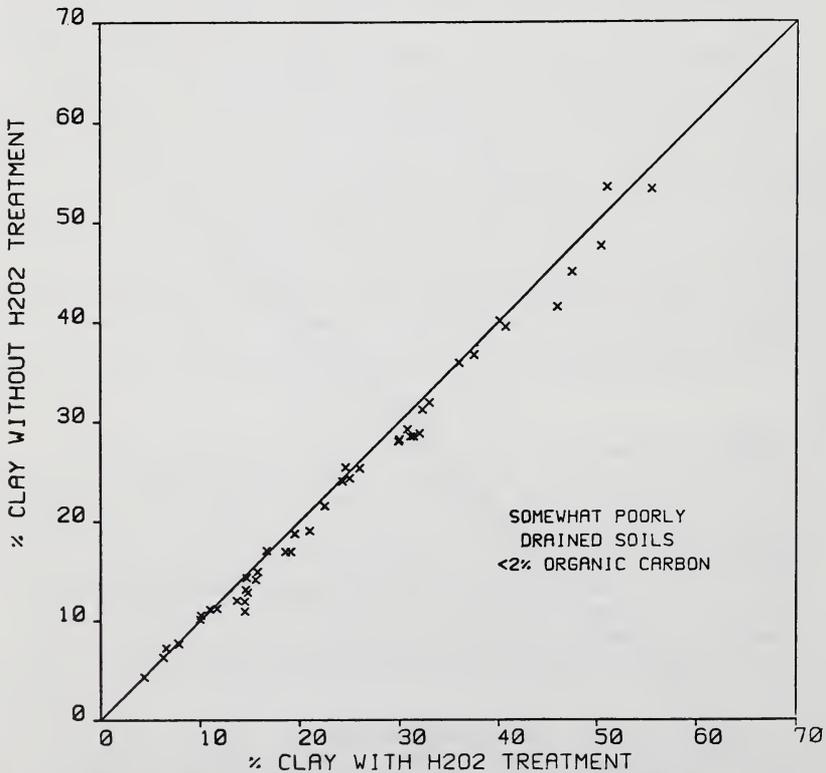


FIGURE 2. *Comparison of Percent Clay Determined With and Without H₂O₂ Pretreatment for Somewhat Poorly Drained Soils With Less Than 2 Percent Organic Carbon.*

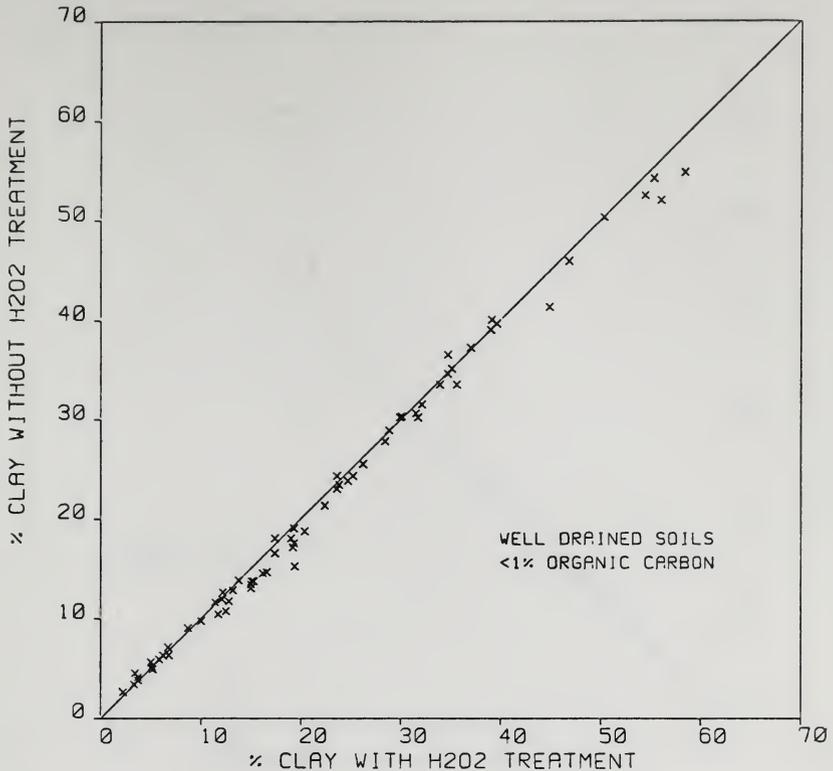


FIGURE 3. Comparison of Percent Clay Determined With and Without H_2O_2 Pretreatment for Well and Moderately Well Drained Soils With Less Than 1 Percent Organic Carbon.

As examples of how close the clay contents determined without H_2O_2 pre-treatment were to the standard values with pre-treatment expected results were calculated from the regression equations. To make this calculation, we assumed clay contents following H_2O_2 pre-treatment of 20 and 50% as standards.

The results of these calculations are shown in Table 2. If we expect the normal variation in clay content by pipette to be within 1.5% for soils that contain 20% clay and within 3.5% for soils that contain 50% clay, then in some cases, the value determined without pre-treatment is adequate. All poorly drained soils, somewhat poorly drained with <2% organic C, and well drained with <1% organic C meet this standard.

FIGURES 1 through 5 show the comparison of clay content as determined with and without H_2O_2 pre-treatment. Soil samples are grouped by drainage class and by amount of organic C discussed above. In some cases, a pre-treatment with H_2O_2 makes little differences in the measured clay content. For poorly drained soils (FIGURE 1), somewhat poorly drained with <2% organic C (FIGURE 2), and well and moderately-well drained soils with <1% organic matter (FIGURE 3) most clay contents determined without H_2O_2 pre-treatment are within 2% of the clay content

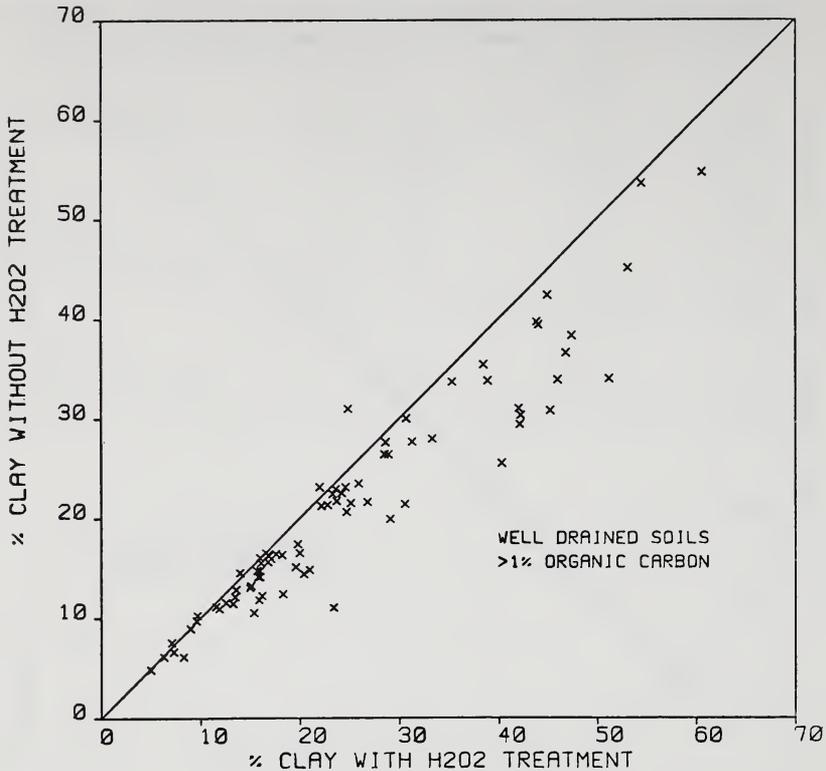


FIGURE 4. Comparison of Percent Clay Determined With and Without H_2O_2 Pretreatment for Well and Moderately Well Drained Soils With More Than 1 Percent Organic Carbon.

found after H_2O_2 treatment. Much of this variation might be expected in the pipette method itself. For these samples, the slope of the regression equation is 0.97 or 0.98. The intercept is $\pm 0.5\%$ clay and the r^2 is 0.98. The points fall close to the 1:1 line shown in the figures which would be perfect agreement.

On the other hand, for well drained soils with $>1\%$ organic C (FIGURE 4) and somewhat poorly drained soils with $>2\%$ organic C (FIGURE 5), there is a greater difference between the clay content determined with and without H_2O_2 pretreatment. For these samples, the points tend to be farther from the 45° line. It is clear that in practically all cases the clay content measured without H_2O_2 pretreatment is considerably lower than that for clay content measured after H_2O_2 pre-treatment.

The degree of aggregation due to organic matter is assumed to be related to the increase in measured clay after H_2O_2 pre-treatment. As organic C content increases, aggregation increases as shown by lower clay content calculated for Table 2. Steinhardt and Norton (1978) demonstrated that an increase in organic matter content increases the strength of soil aggregates in a comparison of samples from permanent pasture and continuously cropped area of the same soil type. From

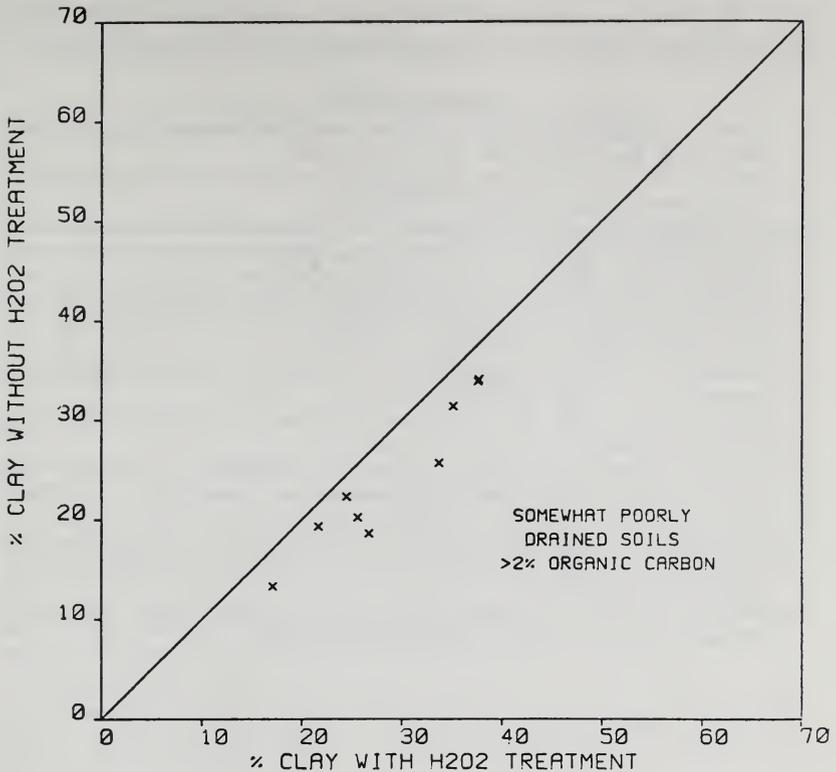


FIGURE 5. Comparison of Percent Clay Determined With and Without H_2O_2 Pretreatment for Somewhat Poorly Drained Soils With More Than 2 Percent Organic Carbon.

Table 2, we see a similar trend that for a given natural soil drainage, aggregation increases as organic C content increases.

Also, from Table 2, for a given organic C content, the degree of aggregation, expressed as a difference in clay content determined with and without pretreatment, increases with better drainage. The cause of this is not clear, but it may be due to the organic matter in the better drained soils being increasingly resistant to decomposition. This could result in stronger aggregates which would hold together without H_2O_2 pre-treatment, but which breaks down when organic matter is destroyed.

Summary

The increase in clay content due to oxidation of organic matter with H_2O_2 prior to particle-size distribution analysis increases with increasing organic C content and better natural drainage of the soil. For well and moderately well drained soils with >1% organic C and somewhat poorly drained soils with >2% organic C, H_2O_2 pre-treatment is needed.

For well drained soils with <1% organic C, somewhat poorly drained soils

with <2% organic C, and all poorly drained soils, the differences in results with or without H₂O₂ pre-treatment are not enough to justify the time required.

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