Removal of organic colloids from soil with minimum damage to clay structures

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ABSTRACT: The presence of organic material in soils may pose problems for some engineering soils properties tests. It was formerly common practice to apply a standard treatment of Hydrogen Peroxide for organic material removal. It has, however, been found that this treatment causes major damage to clay minerals, and Hydrogen Peroxide treatment is now recommended only for highly organic soils such as peat. Investigations have suggested that some potential problems for engineering soils tests may be caused by the presence of organic colloids rather than bulk organic content. This paper examines the possibility of removing organic colloids without damaging the fundamental make-up of the soil being tested. Three oxidising agents were tested for their ability to remove organic colloids without destroying clay colloids. It appears to be possible to achieve this without great difficulty.

1 INTRODUCTION

Treatment of samples with hydrogen peroxide (H_2O_2) was, for many years, common practice in preparation for some soil testing procedures including hydrometer analysis. It was, however, discovered that H_2O_2 causes significant damage to clay structure, and its use is no longer recommended, except in the case of highly organic soils. BS 1377 Part 2, for example, recommends that it be used only when organic content is greater than 0.5% (BS 1377 Part 2 9.4.6.1).

Mikutto et al (2005) noted in a comprehensive review of organic matter removal from soils that H_2O_2 may not be the best reagent for organic material removal since, among other things, it can cause dissolution of poorly crystalline minerals, disintegration of expandable clay minerals, and transformation of vermiculite into mica-like products. Their findings suggest that sodium hypochlorite (NaOCl) and disodium peroxodisulphate ($Na_2S_2O_8$) may be less damaging to clay structure. They also found that both de-flocculation by chemical dispersion and alkaline conditions are crucial for effective removal of organic material.

Nettleship et al. (1997) conducted particle size distribution (PSD) on pure kaolinite of 0.8μm median-size. They performed PSD by hydrometer and centrifuge and concluded that comparison of results suggested agglomeration might be taking place in the hydrometer. Little attention appears to have been given to this suggestion, possibly because pure kaolin of sub-micron size is not typical of the soils which usually interest engineers. Experiments by Stott & Theron (2016) and Monye et al. (2017) indicate that aggregation is in fact a cause of error in the hydrometer for many engineering soils. It appears to be the action of electrostatically charged colloids which draw together silt and clay particles to form agglomerations. This occurs in spite of treatment with chemical dispersant. Substantial agglomeration appears to take place within a few minutes of the end of the mechanical dispersion procedure (Stott & Theron 2017). The charge per unit mass carried by organic colloids is about twice as much as that carried by the most active smectites, and about 20 times as much as that carried by kaolinite. This suggests that organic colloids could make a considerable contribution towards the problem of agglomeration if they are present in the hydrometer test. Both organic and inorganic colloids have particles with size of the order 1μm - 0.01μm and both are commonly found in soil to considerable depth.

2 INVESTIGATION OF POSSIBLE ORGANIC COLLOID REMOVAL

This investigation comprised three stages. The first stage involved examining the effect of various concentrations of the three reagents noted above (H_2O_2, NaOCl and $Na_2S_2O_8$). The aim of this stage was to find the lowest concentration of each reagent which would remove organic colloids.

The second stage involved examination of the effects of these same reagents on clay colloids. The aim of this stage was to find the highest concentration of each reagent which would not destroy clay colloids. Comparison of the results of stage one and two suggested that one of the reagents, bisodium peroxodisulphate ($Na_2S_2O_8$), should be able to remove organic colloids without damaging clay colloids.

The third stage involved performing hydrometer tests on a soil of engineering interest which has a significant visual organic content. Hydrometer analyses were done by a well-known accredited soils testing laboratory using British Standard procedures and ASTM procedures. Hydrometer analyses were done at the Central University of Technology (CUT) geotechnical research laboratory using the SANS 3001 GR3 procedure. Tests were also performed using the SANS procedure modified by the addition of the indicated reagent at concentrations corresponding to just above the minimum, the mid-range, just below the maximum and also beyond the range deduced to be effective for removal of organic colloids without damaging clay. A test was also performed at a concentration subjectively estimated to be approximately equivalent in its vigour of reaction on the soil to treatment according to BS 1377 Part 2 9.4.6.1 for soils with high organic content.

3 MATERIALS AND METHODS

Tests in stage 1 were performed on commercial compost sold with the declaration that it contains only organic materials. The supplier maintained that no soil was used in its production, and no soil was apparent to visual inspection. Tests in stage 2 were performed on a heavy stiff dark brown clay from approximately 1.5m below the surface at a roads project near Thaba Nchu in central South Africa. No organic material was visible and organic material was not observed in microscopic analysis. The tests in stage 3 were performed on a very expansive brown clay from a depth of 1-1.5 m at a proposed piled foundation test site near Steelpoort in North-Central South Africa. Organic material was evident to the naked eye and under the microscope.

Stages 1 and 2 used hydrogen peroxide (H_2O_2), sodium hypochlorite (NaOCl), and bisodium peroxodisuphate ($Na_2S_2O_8$). Stage 3 used bisodium peroxodisulphate ($Na_2S_2O_8$). All three stages used sodium hexametaphosphate ($Na(PO_3))_6$ and sodium carbonate (Na_2CO_3) as prescribed in SANS 3001 part GR3, and reagent grade Methylene Blue ($C_{16}H_{18}N_3SCl$), which was used to label clay and organic colloids and make them visible under the microscope. A normal biological microscope was used to study the soil suspensions produced at the various stages of the investigation. The microscope was equipped with objectives of magnification x10, x40, x60 and x100 and a 9-megapixel digital camera. Most samples were photographed using the x40 and x60 objectives, since these often give the best combination of clarity and depth of focus.

The hydrometer tests in stage 3 of the investigation were performed with standard hydrometer equipment following the standard procedures detailed in SANS 3001 GR3.

4 TESTS OF OXIDISING AGENTS ON ORGANIC COLLOIDS AND SOIL

Organic compost was treated with standard dispersant and mechanically agitated in a manner similar to the SANS 3001 GR3 preparation of soil samples; small quantities of methylene blue were added until colloids were visibly stained and easily identifiable under the microscope. Small quantities of oxidizing agent solution were added using a medical syringe. After each addition the suspension was left until all visible chemical activity had ceased and then a sample was taken for microscopic examination. The procedure was continued until all visible colloids were destroyed.

Soil samples were prepared using dispersant and mechanical agitation as specified in SANS 3001 GR3 for preparation of samples for hydrometer analysis. Methylene blue was progressively added until colloids were easily identifiable. Oxidising agent was added using the same procedure as noted above for the compost. The procedure was continued until colloid content was noticeably diminished.

5 HYDROMETER INVESTIGATION

Samples of the high-organic-content soil noted in materials and methods above were prepared following the procedures of SANS 3001 GR3. Two samples were tested following the normal GR3 procedure. The remaining samples were treated with $Na_2S_2O_8$ following the mechanical dispersion process. The oxidant was added to the 400ml suspension container, stirred and left overnight. The suspension was mechanically agitated again at 1450 rpm for 15 minutes and the normal hydrometer procedure continued from that point. Prior to the first reading of the hydrometer 50ml of suspension was withdrawn for microscopic examination.

6 RESULTS

6.1 *Oxidising agents*

The oxidising agents hydrogen peroxide (H_2O_2) and sodium hypochlorite (NaOCl) both removed clay colloids at lower concentrations than they removed organic colloids. Hydrogen peroxide destroyed clay colloids at a concerntation of about 5 g.dm^{-3} while a concertation of about 8 g.dm^{-3} was needed to remove organic colloids. Sodium hypochlorite destroyed clay colloids at a concentration of about 3 g.dm^{-3} while a concertation of about 6g.dm^{-3} was needed to remove organic colloids. There appears therefore to be no possibility of removing organic content without permanently damaging the soils using either of these reagents. Bisodium peroxodisulphate ($Na_2S_2O_8$), however, removed organic colloids at a lower concentration than that which removed clay colloids. The range of concentrations which appears to be suitable for removing organic colloids without damaging clay colloids runs from about 1.5 g.dm^{-3} to 5 g.dm^{-3}.

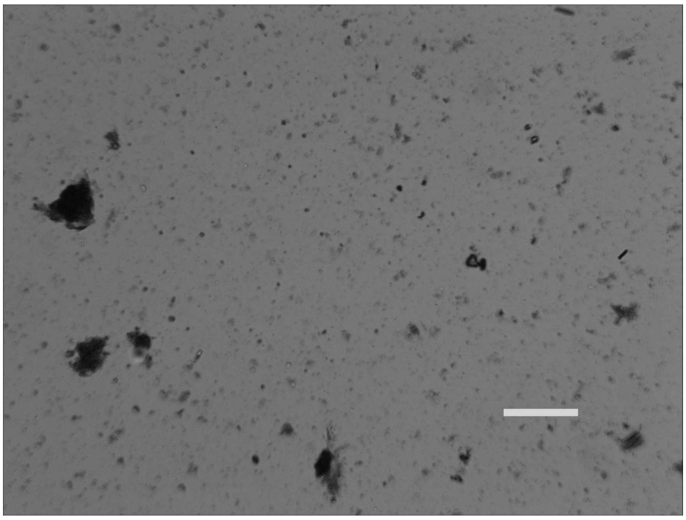

Figure 1. Low magnification view (x10) of compost suspension scale bar 100μm x 10 μm

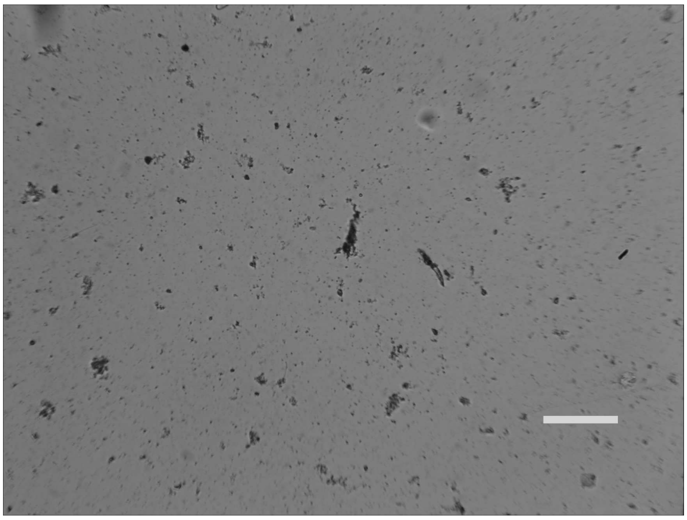

Figure 2. Low power view (x10) of compost suspension after the addition of 3.7 g.dm^{-3} $Na_2S_2O_8$ scale bar 100 μm x 10 μm

Figure 1 shows a low magnification view of the compost suspension without oxidant. Colloidal agglomerations are evident as fuzzy nebulae. Figure 2 shows a similar low magnification view with the addition of 3.7 g.dm^{-3} $Na_2S_2O_8$. No colloidal agglomerations are evident.

6.2 *Hydrometer tests*

The tests for stage 3 of the investigation were conducted on a high-plasticity clay with high visual organic content. Copious fine root hairs were visible under the microscope.

This soil was also tested at an accredited commercial laboratory where hydrometer analysis was performed on two samples using both ASTM and British Standard procedures. The ASTM standard considers the fraction finer than 5 μm to be clay whereas the British Standard considers the fraction finer than 2 μm to be clay. Since the South African standard also considers the fraction finer than 2 μm to be clay, it is appropriate to compare the results obtained at the CUT laboratory with those found by the British Standard procedure. Results are shown in Table 1.

Table 1. Hydrometer results: no oxidising agent

Sample tested by	Test procedure	Indicated % clay
Commercial Lab: 1	BS 1377	64
Commercial Lab: 2	BS 1377	66
CUT Lab: 1	SANS 3001	62
CUT Lab: 2	SANS 3001	62

This soil has been extensively tested and the coefficient of variation of its suction potential has been found to be 10. This may give a fair indication of general variability of soil properties, in which case the range of laboratory values is within expectations. To minimise the effect of variability on results, all of the CUT specimens were taken from one sample which was thoroughly mixed, disaggregated and re-mixed. Individual specimens of this one mixture were used for all hydrometer tests. Results of tests to examine the possibility of removing organic colloids are shown in Table 2.

Table 2. Hydrometer results SANS 3001 procedure modified by addition of oxidising agent

Oxidant	Mass added in preparation	Indicated % clay
$Na_2S_2O_8$	1.5g	56
$Na_2S_2O_8$	3.26g	56
$Na_2S_2O_8$	5.0g	56
$Na_2S_2O_8$	7.5g	52
$Na_2S_2O_8$	15g	35

The results for concentrations close to the upper and lower limits and close to the middle of the range indicated by the initial investigation are significantly lower than the results for untreated samples and are constant. For concentrations above the indicated range hydrometer values of clay % fall substantially, as can be seen graphically in Figure 3.

These results are consistent with the possibility that organic colloids were removed in the range indicated by stages 1 and 2 of this investigation, and that higher concentrations progressively destroy the clay fraction.

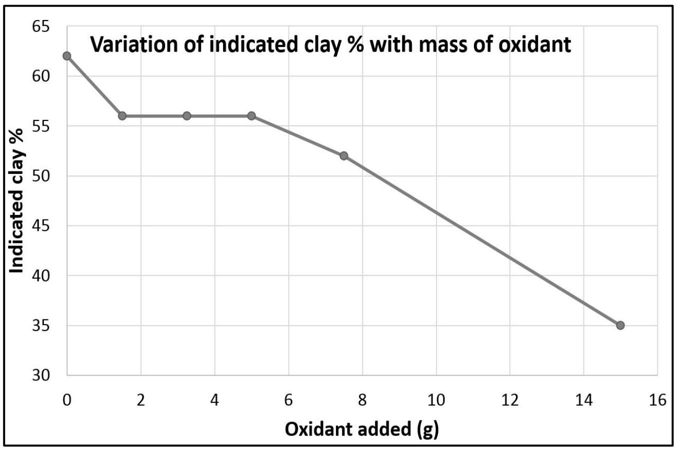

Figure 3. Indicated clay fraction for various quantities of $Na_2S_2O_8$ added during preparation

Figure 4 shows a medium-high magnification view of the suspension extracted from the hydrometer without oxidising agent treatment. There are agglomerations shrouded in blue stained colloids and two pieces of organic material, probably root hairs, completely covered with blue stained colloids. Figure 5 shows a similar view of suspension extracted from the hydrometer following treatment with 3.25g of oxidant.

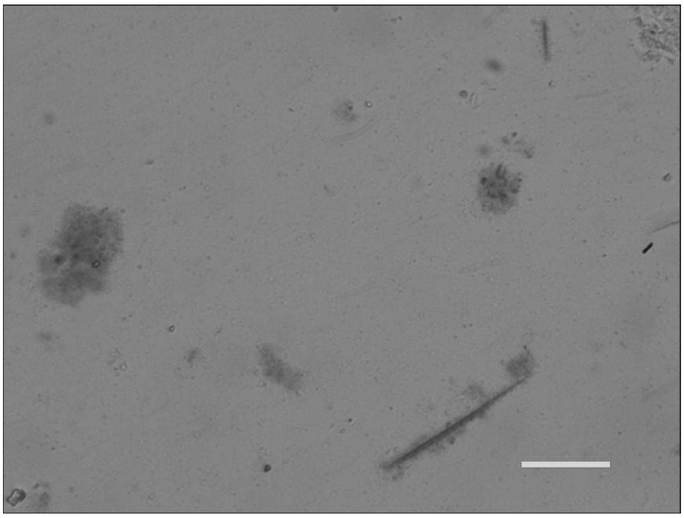

Figure 4. Medium-high magnification view (x40) of high-plasticity clay suspension without treatment. The root hairs at lower right and upper right are completely covered with colloids. Scale bar 30μm x 2μm.

Both figures 4 and 5 show rounded, nebular agglomerations of stained colloids which could be either organic or clay. The fact that organic elements in figure 5 are clean and those in figure 4 are coated lends support to the possibility that it is organic colloids which have been removed. It is clear that the concentrations indicated remove only the organic colloids and not the bulk of organic material.

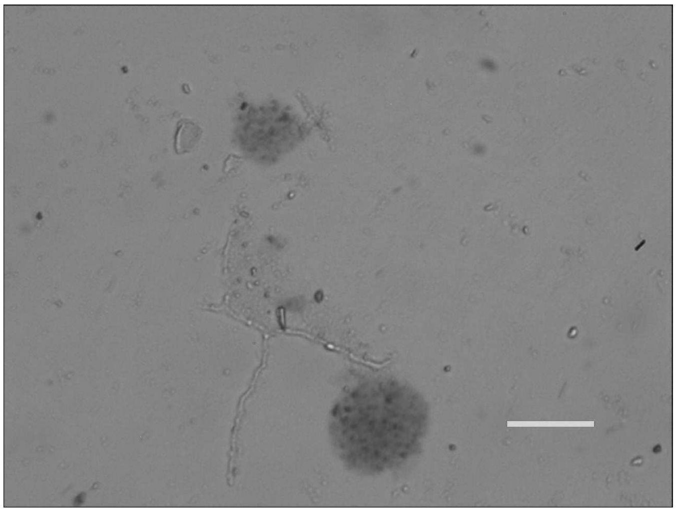

Figure 5. Medium-high magnification view (x40) of high-plasticity clay after the addition of 3.25 $g.dm^{-3}$ $Na_2S_2O_8$. The root hair at lower centre has no colloids surrounding it. Scale bar 30μm x 2μm.

7 CONCLUSIONS

Treatment with $Na_2S_2O_8$ in the range of approximately 1.5 $g.dm^{-3}$ to 5 $g.dm^{-3}$ appears to be able to remove organic colloids from soil without damaging the soil's clay fraction. Preparation of samples using this oxidising agent within this range of concentrations should therefore be far more satisfactory than the use of Hydrogen Peroxide, as used to be the standard, since, as noted in 6.1 above, it destroys clay colloids at a lower concentration than that at which it destroys organic colloids.

Organic colloids have about double the electric charge density of the most active clay particles. Their removal could therefore lead to less error in hydrometer estimates of clay fraction due to agglomeration - which is caused by electrical attraction between charged components of the soil. The tests performed on this particular soil using $Na_2S_2O_8$ treatment in the above range gave a hydrometer value of clay fraction 10 % less than those of the untreated soil.

There appears to be no standard by which hydrometer values can be compared with confidence, and several researchers have raised doubts about the hydrometer's reliability for high clay-content soils. It is therefore difficult to determine to what extent the successful removal of the organic colloids may provide a truly reliable estimate of clay fraction.

8 REFERENCES

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