

HYDROMETER UNDER THE MICROSCOPE

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Abstract

For many years the hydrometer was the only technique that was internationally standardised for geotechnical particle size distribution analysis of fine soils. Nettleship et al. (1997). Recently Britain has added the pipette method as a preferred alternative. The pipette method is considered to give slightly more accurate and consistent results. Both procedures stand on the theoretical foundation of Stokes' law. Stokes' law assumes constant density of the particles being tested, that larger particles settle faster than small particles when in a liquid suspension, and that there is no interference between particles and other particles or obstructions in the suspension. It also assumes all particles to be spherical. This paper studies the hydrometer theory by analyzing settled sediments and compares the effectiveness of two dispersion procedures, namely that specified by TMH 1, with that specified by SANS 3001 (replaced TMH1).

Keywords: *Clay fraction, Dispersing agents, Hydrometer analysis, Methylene Blue*

1 Introduction

South African's soils most problematic aspect is the behavior of expansive clay, which is continuing to contribute to structures and roads infrastructure failure. Hydrometer analysis is used to determine clay fractions. Clay fraction and plasticity index are used to determine soil heave potential using Van der Merwe's diagram (1964). Most important characteristics of soil behavior depend on clay mineral content rather than hydrometer test's particle size. Hydrometer analysis assumes clay particles ranges from 2 microns (μm) downwards, silt to be particles larger than $2\mu\text{m}$ and smaller than $65\mu\text{m}$. However clay particles like kaolinite, illite and halloysite can be considered larger than $2\mu\text{m}$, also silt particles size may range down to $1\mu\text{m}$.

Hydrometer analysis monitors change in density of settling suspension. It relies on Stokes' law which assumes all particles to be spherical; it assumes complete dispersion of clay particles at the time of testing and assumes fine particles are not carried down by coarse particles. Savage (2007) suspected that clay particles are flaky, which suggest that

hydrometer analysis might under-estimate clay fraction. Savage also suspected that clay particles might be carried down by larger particles, and that dispersion may not be complete when the test is performed.

The aim of the investigation is to assess some of the questionable aspects of the hydrometer by isolating and testing sand and silt fractions after settlement. A Microscope is used to examine the samples and compare with what is expected by the hydrometer theory. Methylene blue (MB) is added since it is known to be effective in labelling clay minerals by swapping places with exchangeable cations.

2 Research Background

Most methods for predicting heave severity rely on clay fraction estimation. Van der Merwe's diagram is used to predict heave potential by using clay fraction and plasticity index. This method can be flawed if clay fraction determination is unreliable. Other critical aspects of soil behavior depend on clay mineral content. The following possible shortcomings were noted by Savage (2007) which might be a problem: (i) Stokes' law assumes all particles to be spherical and clays are flaky, (ii) De-flocculation of many clays is seldom fully completed at the time of testing, (iii) Clay particles are partially carried down by the larger particles and (iv) A relative density of 2.65 is assumed for all particles. Soil mineralogy should be taken into account and probably different treatments should be considered for different clay types. (Rodrigues et al. 2011)

The Hydrometer monitors the change in density of settling suspension. Rolfe et al, (1960) pointed out that the analysis averages the specific gravity over the submerged part of the instrument and therefore depends on the shape and depth of hydrometer submergence which might bring errors to the test results.

Nettleship et al. (1997) compared automated centrifuge sedimentation method and hydrometer analysis using particles of kaolinite clay. The results obtained from their investigation were that the hydrometer underestimated fraction of submicron clay particles, which suggested that the difference in result might be due to the time dependent aggregation of fine clay particles.

3 Research Methodology

Soil samples from Heidedal, suburb of Bloemfontein, Free State were tested and treated as per SANS 3001 GR3 and TMH1(1986), with sodium hexametaphosphate and sodium carbonate (referred to as Calgon) and sodium silicate and sodium oxalate solution before examination by microscope. While examining the samples with the microscope methylene blue was added. Results are then compared with the hydrometer theory.

3.1 Methylene Blue Adsorption Test

Methylene blue adsorption (MBA) is a measure of the clay particle surface area, which is a function of clay type and an indicator of water adsorption by clay percentage potential. (Çokça, 2001). MBA is a simple and reliable method to obtain clay mineral properties in soils. There are two methods named "spot" and "turbidimetric" method used in practice. The procedure used in the spot method was used for this investigation it is the most commonly used method in the engineering field. In determining the methylene blue value (MBV) the amount of MB solution adsorbed is used. The Association Française de Normalization (AFNOR) and American Society for Testing and materials (ASTM) standards have adopted the methylene blue stain test. Complete methylene blue index (MBI) method is accessible as

ASTM designation C837-09. MB is used to label clay minerals in this investigation, but not to determine the MBV.

3.2 Microscopic examination

Soil samples were prepared and treated as per SANS 3001 Gr3, 2012 and TMH 1, 1986. After mechanical stirring they were transferred to settle in the settlement containers designed for this study. After a week of settlement, water was drained and the sediments were dried at 45°. Dry sediments were separated into sand and silt particle layers before microscope examination. An optical microscope was used with a 9 megapixel camera attached to it. A small sample of each separated layer was suspended in small amount of distilled water and a drop was extracted to be placed on the microscope slide. Photographs were taken before and after MB addition. Soil samples were examined which had been treated as per SANS3001 Gr3,

4. Findings and Discussion

4.1 Heidedal Sample TP1LR1

4.1.1 Coarse Sand Layer

Sample from Heidedal Test pit 1 layer 1 in the Free State was used for this investigation. Methylene blue was added to the sample, dispersion for this soil was not complete but reasonable. Figure 1 shows a grain from the sand layer after dispersion with Calgon. The grain appears to have a thin, deeply blue stained coating of very small particles, almost certainly a smectite, and probably montmorillonite. The lower right side of the large grain appears to consist of clay particles surrounding a silt core and appears to form a bridge attaching to another sand grain. Figure 2 shows a sample of the same soil dispersed with sodium silicate and sodium oxalate solution. The sand grain has deeply blue stained patches, it is appears that almost the entire sand grain is covered by a thick layer of very small particles, probably smectite. The dispersion of samples treated by calgon appears to be more successful than that treated with sodium silicate and sodium oxalate solution.



Figure 1. Coarse Sand layer from Heidedal soil, Test Pit 1 Layer 1 (TP1, LR1). Dispersant Sodium Hexametaphosphate and Sodium Carbonate.

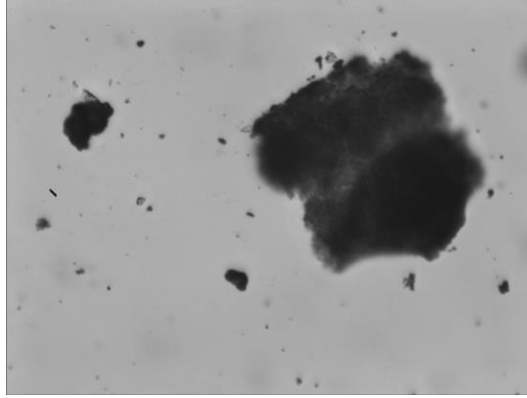


Figure 2. Coarse Sand layer from Heidedal soil, Test Pit 1 Layer 1 (TP1, LR1).
Dispersant Sodium silicate and sodium oxalate solution.

4.1.2 Fine Sand Layer

Figure 3 shows a sand particle, half of which has clay clinging to it and half has only a thin coating of small deeply blue stained particles what seems to be the sand grain is completely covered with small deeply blue-stained particles. Clay and silt sized particles are visible in Figure 4 which is from the corresponding layer in the sample dispersed with Sodium Oxalate and Sodium Silicate. Some of the sand grains and silt-sized particles show deep stain (probably montmorillonite) while others show little methylene blue staining (probably kaolinite). Again the calgon appears to be more successful in dispersion, but still far from satisfactory.

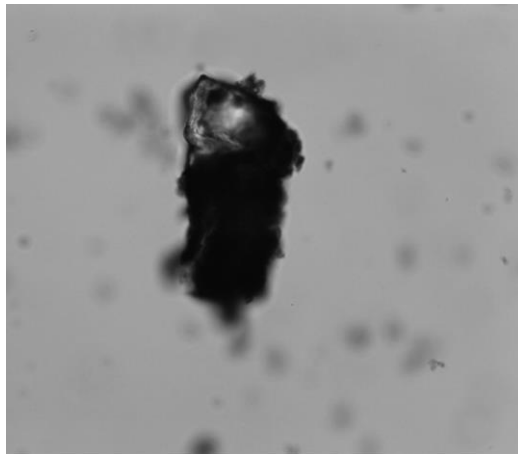


Figure 3. Fine Sand layer from Heidedal soil, Test Pit 1 Layer 1 (TP1, LR1).
Dispersant Calgon solution.

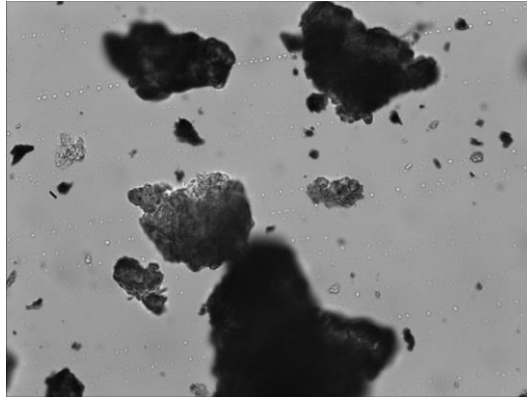


Figure 4. Find Sand layer from Heidedal soil, Test Pit 1 Layer 1 (TP1, LR1).
Dispersant Sodium silicate and sodium oxalate solution.

4.1.3 Coarse Silt Layer

The coarse silt particles in figure 5 appear to have blue stained edges. Most of the smaller silt and clay particles are completely blue stained. Diffused nebular structures are visible and appears to be clouds of extremely small clay particles. Figure 6 is the corresponding layer from the sample dispersed with Sodium Oxalate and Sodium Silicate. The large particles are all completely covered by deeply stained clay particles. Many of the clay particles are extremely small, probably montmorillonite, packed more tightly together than in the sample shown in Figure 5.

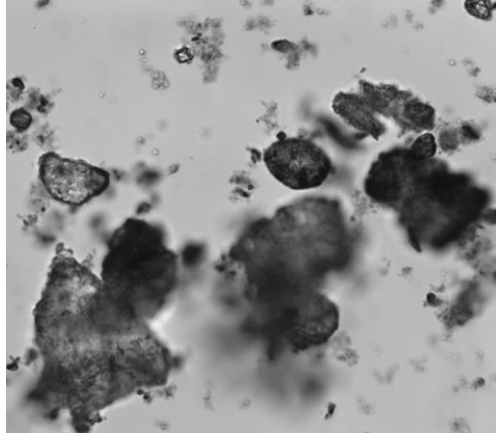


Figure 5. Coarse Silt layer from Heidedal soil, Test Pit 1 Layer 1 (TP1, LR1).
Dispersant Calgon solution.

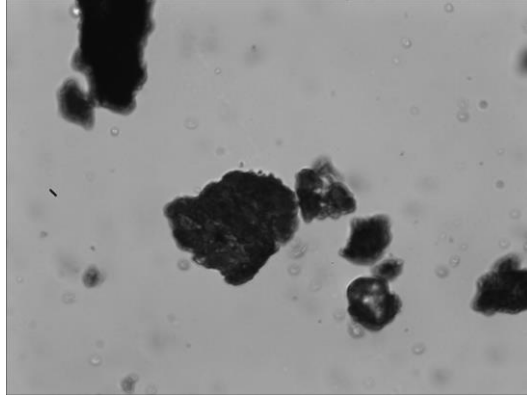


Figure 6. Coarse Silt layer from Heidedal soil, Test Pit 1 Layer 1 (TP1, LR1).
Dispersant Sodium silicate and sodium oxalate solution.

4.1.4 Fine Silt Layer

Figure 7 shows many clouds of extremely small clay particles forming agglomerations with silt and large clay particles. There's a significant difference between the sample that was treated with calgon and that of sodium silicate solution. Figure 8 shows the corresponding layer from the sample treated with sodium silicate/sodium oxalate. The fine clay particles appear to stick far more tightly to the large particles and do not form extended dispersed clouds.

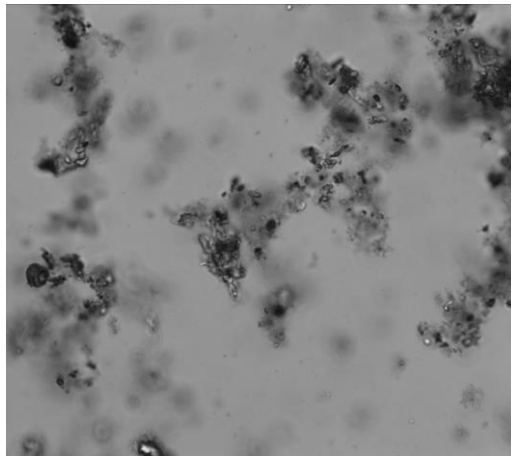


Figure 7. Fine Silt layer from Heidedal soil, Test Pit 1 Layer 1 (TP1, LR1).
Dispersant Calgon solution.

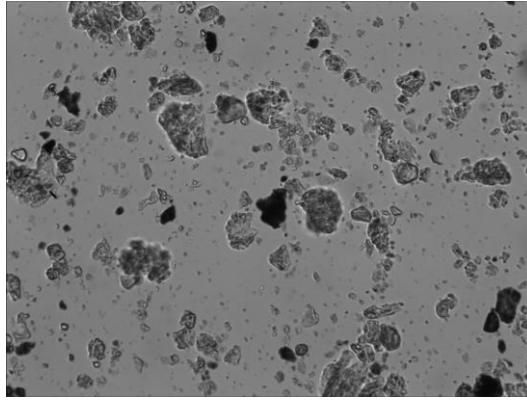


Figure 8. Fine Silt layer from Hededal soil, Test Pit 1 Layer 1 (TP1, LR1).
Dispersant Sodium silicate and sodium oxalate solution.

Figures 9 to 12 show samples from the fine silt layer from a different clay layer from the same test pit. The same pattern can be seen in each case. The Calgon-dispersed samples have tenuous nebulae of very fine dispersed clay particles, while the Sodium Silicate and Sodium Oxalate-dispersed samples show fine clay particles clinging more closely to silt particles.

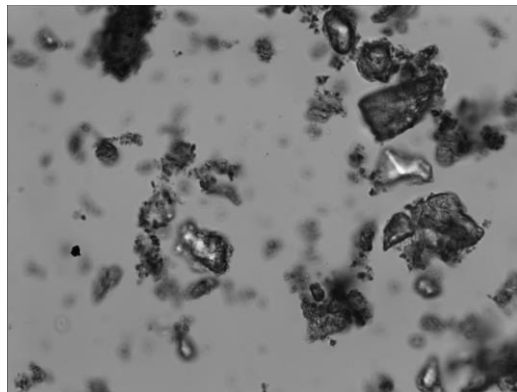


Figure 9. Fine Silt layer from Hededal soil, Test Pit 1 Layer 2 (TP1, LR2).
Dispersant Calgon.

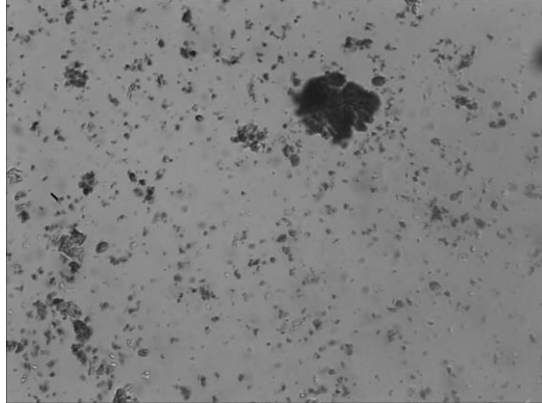


Figure 10. Fine Silt layer from Hededal soil, Test Pit 1 Layer 2 (TP1, LR2).
Dispersant Sodium silicate and sodium oxalate solution.

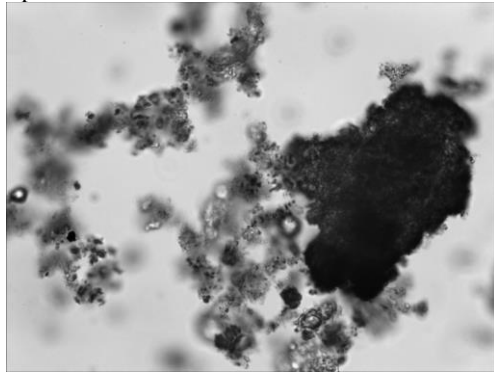


Figure 11. Fine Silt layer from Hededal soil, Test Pit 1 Layer 3 (TP1, LR3).
Dispersant Calgon.

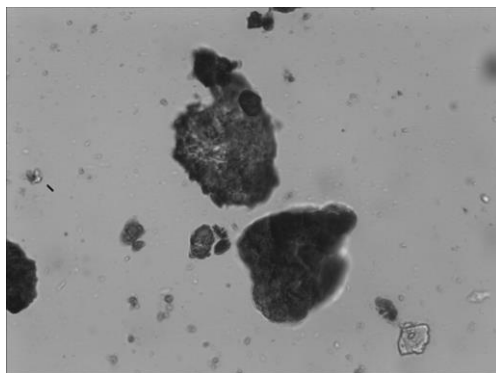


Figure 12. Fine Silt layer from Hededal soil, Test Pit 1 Layer 3 (TP1, LR3).
Dispersant Sodium silicate and sodium oxalate solution.

5. Conclusion

The hydrometer theory assumes that the bigger particles settle first. The hypothesis to be examined is that clay and silt particles may be carried down by larger particles earlier than expected in the settlement process. The dispersion of soil samples was significantly different between the mixture sodium hexametaphosphate and sodium carbonate and a mixture of sodium silicate and sodium oxalate solution. The soil samples treated with sodium hexametaphosphate showed slightly better dispersion while that of sodium silicate appeared to be less effective.

The expectation of smaller particles settling with bigger particles as suspected by Savage appears to be confirmed. Sand layers for all treatments show a far smaller tendency to draw down smaller particles compared to silt layers. More clay-sized particles content is evident in the silt layers. Calgon seems to disperse the fine clay particles into clouds which form agglomerations with silt particles, whereas the Sodium Oxalate and Sodium Silicate leaves these fine clay particles more closely attached to larger particles.

The study seems to be confirming the view that for high-clay content soil, hydrometer may be unreliable for determining the clay fraction. This could be the reason projects fail due to heaving clay while the geotechnical results predicted little chance of heaving. For soils with high-clay content a more cautious approach appears to be required.

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