EXTENT OF INACCURACY IN THE HYDROMETER TEST

by

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DECLARATION

I, the undersigned, declare that the thesis hereby submitted by me for the degree *Master of Engineering in Civil Engineering* at the Central University of Technology, Free State, is my own independent work and has not been submitted by me to another University and/or Faculty in order to obtain a degree. I further cede copyright of this thesis in favor of the Central University of Technology, Free State.

Priscilla Ketlametswe Monye

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Date: July 2018

Bloemfontein, South Africa

ABSTRACT

Assessment of particle size distribution is a fundamental aspect of any soil characterization and is commonly used for soil classification. While it is relatively easy to classify the coarse fraction, classification of the soil fines remains problematic, particularly the fraction less than 2µm. Hydrometer analysis has been an international standard tool for fines assessment for many years but there may be serious flaws. A number of shortcomings facing the hydrometer have been alluded to by many researchers. Some of these have been addressed by laser scattering techniques. Others, including completeness of dispersion and the amount of clay carried down with coarser fractions, remain problematic.

Hydrometer analysis appears to underestimate clay content when compared to microscopic analysis and tends to overestimate clay fraction when compared to laser diffraction method. The analysis is based on Stoke's law which estimates the rate at which particles settle in a suspension. The accuracy of hydrometer analysis depends critically on all of the clay present being dispersed into the suspension. Microscopic analysis, however, showed that, with the various types of dispersant specified in the hydrometer method, some samples experienced good dispersion while some were poorly dispersed.

Most of the commonly used methods of heave prediction in South Africa rely on an estimate of the clay fraction. Hydrometer analysis is not sufficiently reliable for a critical test such as heave prediction unless a means can be found of identifying which soils do not disperse effectively in the specified deflocculant.

Hydrometer and pipette analysis measures only the particle size distribution of the fine soil fraction, but some important aspects of soil behavior such as a change in volume depend on the clay mineral content. An assessment of clay mineral contents is therefore also required.

The necessity for a better method of estimating the clay fraction is illustrated by the many buildings and roads that have failed due to heave movements. A good example of this is a government subsidy housing site at Kimberley, where hydrometer analysis showed only 6% clay fraction. Van der Merwe's method therefore predicted zero heave and houses were built on that assumption. Heave did in fact occur and one house became structurally unsound while still under construction, whilst many others became unsound shortly after construction.

This investigation assessed some aspects of the reliability of the hydrometer by separating the sand, silt and clay fraction of selected soil samples by sedimentation and the conducting tests on these fractions. Microscopic examination using a methylene blue staining technique was used to compare the composition of sediment layers with that expected from hydrometer theory.

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7. Mme MaNthatisi, for your support and love.
8. My family and friends. May the Lord Jehovah bless you all.

Dedication

To my son Lincoln, my precious gift from God. My precious niece Kaboentle. My siblings, Keatlaretswe, Kaelo and Kitso. Love you always, your support always amazes me.

"Proverbs 3"

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LIST OF ABBREVIATIONS

AFNOR	Association Française de Normalization
ASTM	American Society of Testing and Materials
B	Bentonite
$Ca(HCO_3)_2$	Calcium bicarbonate
$CaSO_4$	Calcium sulphate
CEC	Cation Exchange Capacity
g	Grams
hr	Hour
K	Kaolinite
LD	Laser Diffraction
LL	Liquid Limit in %
LS	Linear Shrinkage in %
ℓ	Litres
MB	Methylene Blue
MBA	Methylene Blue Adsorption
MBI	Methylene Blue Index
µm	Micrometre
MS	Microscope
Min	Minute
ml	Millilitres
Na^+	Sodium
NS	Not Specified
TSPP	Sodium Pyrophosphate
PI	Plasticity index %
PL	Plastic Limit in %
SANS	South African National Standard
SEM	Scanning Electron Microscope
SSA	Specific Surface Area
TMH	Technical Methods for Highways

TSPP	Tetrasodium Diphosphate Decahydrate/ Sodium Pyrophosphate
w%	Natural Moisture Content

CHAPTER 1 : INTRODUCTION

1.1 Background

Particle size distribution is an important factor in the classification of soils. Hydrometer analysis has been adopted in international standards for determining the fines content of soils. This fines content is split into silt and clay fractions based on the size of the particles. Silt comprises particles between 2μm and 75μm whereas clay comprises particles smaller than 2μm. Estimation of the clay fraction is required for numerous soil evaluations including potential heave for building foundations (Stott and Theron, 2015). Van der Merwe's method (Van der Merwe, 1964) is commonly used to predict heave using Plasticity Index (PI), the percentage passing 425μm and clay fraction. Skempton's clay activity (Skempton, 1953) is determined by PI/clay fraction. These methods may be compromised if clay fraction cannot be accurately determined by hydrometer or pipette analysis.

The hydrometer test monitors the decrease in the density of a suspension of soil in water as the particles settle. The rate at which particles settle depends on their size with the larger particles settling faster than finer particles. As the density decreases, the hydrometer sinks lower in the suspension. If the temperature is kept constant at calibration temperature, density will be read directly from the stem of the hydrometer. Should temperature differ, a correction is made to the readings. The hydrometer analyses the rate at which particles settle by measuring the specific gravity of the suspension. However, due to the size of the bulb of the hydrometer, it does not measure the specific gravity of the suspension at a point. It averages the specific gravity over the height of the bulb the readings obtained therefore depend on the shape and depth of hydrometer submergence. This may bring errors in the analysis (Rolfe *et al.* 1960).

1.2 Problem Statement

Hydrometer analysis appears to underestimate clay content when compared to microscopic analysis and tends to overestimate clay fraction when compared to laser diffraction method. It is based on Stoke's law which estimates the rate at which particles settle in a suspension. The accuracy of the hydrometer analysis depends critically on all of the clay particles present being dispersed into the suspension. If clay particles remain flocculated (joined together), they may settle faster than the individual particles. Microscopic analysis (Stott and Theron, 2016) shows that, for the types of dispersant typically specified in the hydrometer method, some samples experience good dispersion while others are poorly dispersed. With poorly dispersed soils, the hydrometer method tends to underestimate the fines content of soils and makes the soil appear to be coarser than it is.

Keller and Gee (2006) mentioned that hydraulic soil property estimates work best for coarse-textured, structure-less soils of low clay content and results should be treated as empirical estimates.

Many heave prediction methods are based on an estimate of clay fraction of the soil. Hydrometer analysis can therefore not be considered reliable for a test as critical as heave prediction unless a means can be found of identifying which soils do not disperse effectively in the specified deflocculant. Hydrometer and pipette methods measure only the particle size distribution of the fine soil fraction. However, some important aspects of soil behavior, like change in volume, depend not only on the clay content but also on the mineralogy of the clay. An assessment of clay mineralogy is therefore also required.

Many technical papers (Brink and Hörtkorn, 2016; Ristow and Likos, 2000 and others) investigate different dispersing agents, the use of different hydrometer types, sample preparation before testing and even sending one sample to many geotechnical laboratories using different testing standards (e.g. SANS 3001, 2012; ASTM D422, 2007; TMH1, 1986). Brink and Hörtkorn (2016) determined that sample preparation, drying and mixing technique had an impact on the results. Their investigation reported clay contents of 62% and 12% for a single sample tested using two different testing standards. Jacobsz and Day (2008) suggested insufficient care was taken by commercial testing laboratories. Eshel *et al.* (2004) found that the percentage of clay-size particles inferred from laser diffraction (LD) techniques was lower than mass percentage of the clay fraction derived by pipette method. In other attempts to investigate whether clay particles are actually spherical (as assumed by Stokes' law), researchers have used the scanning electron microscope (SEM), which shows some types of clay particles to be flaky, spherical, needle-like or platy (Figure 1.1).

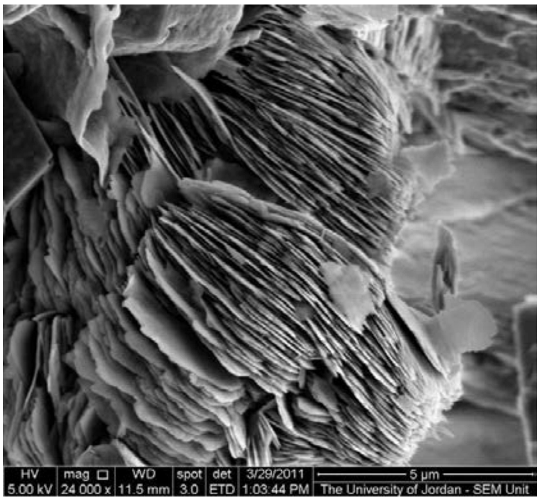

Figure 1.1: SEM kaolinite image. Digital image. *Thermo Fisher Scientific.* Accessed 30 November 2017

https://www.fei.com/image-gallery/kaolinite-clay-sheets/

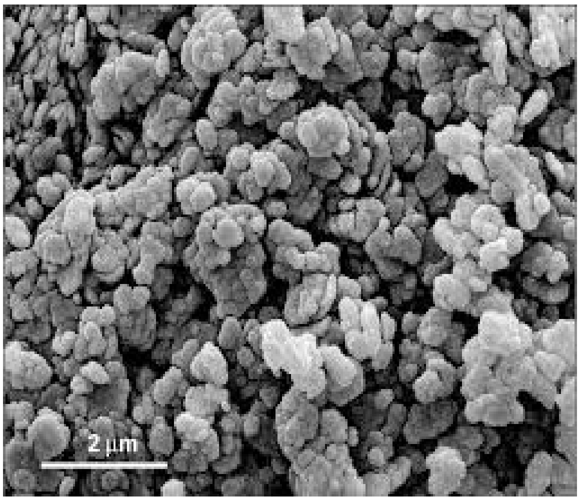

Figure 1.2: SEM image of Felipe II kaolin (from IR Wilson *et al.*, 2006)

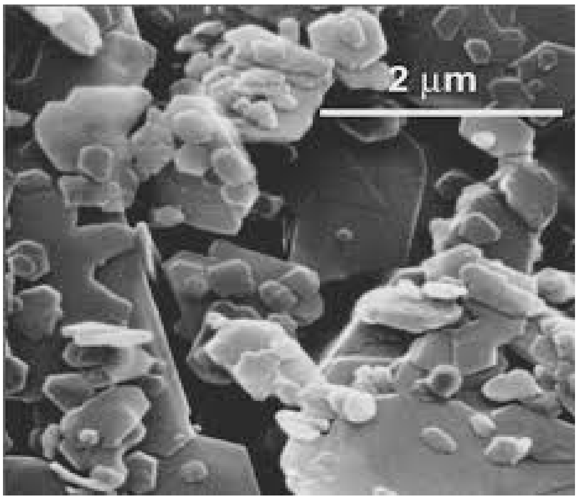

Figure 1.3: SEM image of kaolin from Xpixuna deposit (from IR Wilson *et al.*, 2006)

The SEM is widely considered to be suitable to study clays because it provides magnified three-dimensional views of clay surface with great depth of focus (Bohor and Hughes, 1971). It is, however, important to point out that before SEM investigation, samples are dried and gold or carbon coated. They thus differ from the samples tested by the hydrometer method.

1.3 Project Objective

The main objective of the project was to simulate and investigate the sediments deposited during hydrometer tests, with the possibility of finding shortcomings and exploring the effect of different dispersing agents including those mentioned in SANS 3001-GR3: 2014 and TMH 1 (1986). The following specific objectives were set:

i. Assessment of two of Savage's (2007) suggested shortcomings:

    a) Incomplete de-flocculation at the time of testing.

    b) Clay particles being partially carried down by larger particles.

ii. Confirmation of Nettleship *et. al.'s* (1997) contention that agglomeration may occur during settlement in the hydrometer test.

iii. Assessment of Stott and Theron's (2015) speculation that the agglomerations which they observed will not settle at the rate of their individual particles.

1.4 Scope of Study

This study investigates the hydrometer analysis theory by examining the sedimented material microscopically. It is important to find a reliable method to assess clay fraction as it has a critical bearing on engineering problems encountered. There appears to be no recognised alternative to which the hydrometer analysis can be compared. Other methods such as laser diffraction have been tried but have not given convincing results for soil.

CHAPTER 2 : LITERATURE STUDY

2.1 Background

Some critical aspects of soil behaviour depend on mineral composition of the clay rather than particle size distribution as given by the hydrometer test. There is a range of sizes (between approximately 1 and 2 microns) where clay sizes and silt sizes overlap. Savage (2007) suggested the following as problematic aspects of hydrometer analysis:

i. Stokes' law assumes all particles to be spherical but clay particles are often flaky,

ii. De-flocculation (dispersion) 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, which may not be true.

Savage proposed an alternative indirect method of estimation based on Skempton's activity formula, but this has not been widely accepted. Brink and Hörtkorn (2016) advised against engineering conclusions based on results like Atterberg limits and hydrometer readings only, they should be used only for comparative purpose due to the observed range of hydrometer values for fines.

The physical and chemical properties of particular clay minerals are dependent on their structure and composition (Murray, 2007). The structure and composition of most clays consists of basic building blocks comprising octahedral and tetrahedral sheets. This accounts for differences in the physical and chemical properties of various clay minerals. Diagrammatic sketches of octahedral and tetrahedral sheets from Murray (2007) are shown below with the explanation.

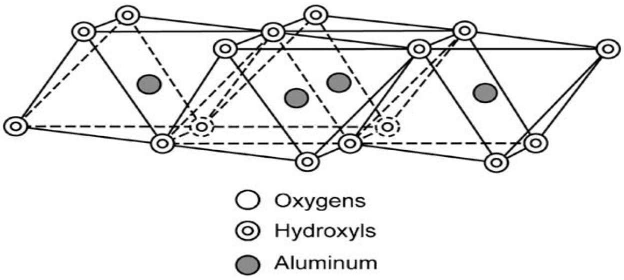

Figure 2.1: Octahedral sheets diagram. (Murray, 2007)

Octahedral sheets consist of packed oxygen atoms and hydroxyls in which aluminium, iron and magnesium atoms are arranged in octahedral coordination as illustrated by figure 2.1. In the tetrahedral sheet the silicon atom is equidistant from four oxygen atoms, or sometimes hydroxyls, which are arranged in the form of a tetrahedron (Fig. 2.2).

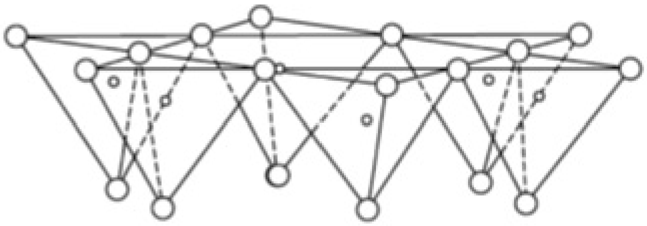

Figure 2.2: Tetrahedral sheet diagram. (Murray, 2007)

Clay minerals have cation exchange capacity (CEC) which depends on their atomic structure and specific surface area (SSA). CEC is one of the most important fundamental properties of clays and provides a relatively simple chemical means of assessing clays (Pan and Shen, 2003). SSA is a physical property which influences the engineering properties such as plasticity and activity in addition to general physical and chemical processes in the soil (Maček *et al*. 2013).

Methylene Blue (MB) has been found to be effective in labeling clay by replacement of exchangeable cations (Türköz and Tosun, 2011). The methylene blue test was established for determining the suitability of granular material in manufacturing concrete by detecting the clay content of granular material. MB behaves like cationic dye when mixed with water. When MB solution is mixed with soil solution, chloride ions in the MB solution change place with cations in clay minerals and are adsorbed on the surface of clay minerals (Türköz and Tosun, 2011). The MB Adsorption (MBA) test was found to be reliable and a simple method to obtain information on the properties and presence of clay minerals in the soil. Different testing techniques have been developed including the turbidimetric method which uses MB electrolytes and the stain test which is widely used in engineering practice (Chiappone *et al,* 2004). Stain (spot) tests quantify ionic adsorption capacity of soil by measuring the quantity of MB dye needed to cover the total surface of soil particles (Maček *et al.* 2013).

MBA value can indicate a low or high value which corresponds to low clay activity or high clay activity respectively. MB staining procedures are specified by the *Association Française de Normalization* (AFNOR) and by the American Society of Testing Materials (ASTM). Both standards describe procedures for obtaining a semi-quantitative evaluation of the activity of a soil and indications of the probable type of clay minerals contained in the soil. AFNOR (1993) derives the *"valeur de bleu"* (VB) and ASTM (1984) derives a comparable "methylene blue index" (MBI). Chiappone *et al.* (2004) came to the conclusion that both procedures can give good estimates of clay content in certain circumstances. MB molecules have a negatively charged $(Cl)^-$ anion and a large positively charged cation. MB replaces natural cations of clay irreversibly (Topal, 1996). When MB solution is added to a mixture of clay and filtered water, the MB cations replace cations on the external surfaces of the clay.

Results of several particles size analyses and sedimentation methods have given fair agreement when compared to each other for particles larger than 1µm. Disagreements are observed for submicron particles (Nettleship *et al.* 1997). They concluded that the cause of underestimation of clay fraction by hydrometer may be due to hydrolysis of polyphosphate dispersants and suggested evaluation of dispersants to check that they do not suffer from time-dependent effects. Rodriguez *et al.* (2011) suggested that soil class mineralogy should be taken into account and different treatments are required for different clay types.

A study was conducted by Rolfe *et al.* (1960) in which montmorillonite, kaolinite and illite clay minerals were dispersed in water of different hardness using various deflocculents including sodium tripolyphosphate, sodium hexametaphosphate, sodium hexametaphosphate plus sodium carbonate and tetrasodium pyrophosphate. The investigation concluded that it is not practical to disperse calcium saturated clays with tripolyphosphate, hexametaphosphate and tetrasodium pyrophosphate and that sodium hexametaphosphate, sodium tripolyphosphate can control the degree to which clay particles are dispersed in different water hardness. Tripolyphosphate was found to be an effective dispersant for montmorillonite in water of low to medium hardness, kaolinite was easily dispersed by all the phosphates and illite was found to be difficult to disperse. The chemistry of dispersion is a research topic which is being conducted in an attempt to understand the subject. Uncertainties regarding the effectiveness of the dispersant in the hydrometer test may yield potentially misleading results.

Wintermyer and Kinter (1955) studied the effectiveness of 19 different chemical dispersing agents including Na^+ hexametaphosphate, $TetraNa^+$ pyrophosphate and Na^+ oxalate plus Na^+ silicate. The investigation included a selection of 16 soils including two clay minerals, bentonite and kaolinite. All except one of their samples were from the United State of America. Dispersion was by air-jet dispersion cup and high-speed motor stirrer. Na^+ polyphos and Na^+ tripolyphosphate were highly effective with most soils but were not effective with laterite, a ferruginous soil from the tropics. The laterite soil was highly dispersed by $triNa^+$ phosphate and $tetraNa^+$ pyrophosphate which were found not to effective for other soil groups. This agrees with the findings of Rodriguez *et al.* (2011) about different treatments for different clay types. Out of 19 dispersant, 13 were found to be unsatisfactory, some producing little dispersion and others were ineffective.

Clay particles have charge along their edges due to broken bonds, and negative charge on their faces. The charge along the edges depends on pH. At high pH (as in Calgon), the charge will be positive; at normal or low pH it will be negative. When the solution is added to a soil sample, the negatively charged edges attract positively charged anions from the solution resulting in electrostatic attractions and repulsions around the soil surface. The dispersion process is still not well understood and is a subject of on-going research. The usual assumption is that the dispersant which gives the highest clay yield has done a good job of dispersion. Kaur and Fanourakis (2016a) found Sodium silicate/oxalate to be the least effective as it yielded 0% clay content for the black soil which they investigated. Wintermyer and Kinter's (1955) research found this solution to be one of the less effective dispersants of the 19 that were used in their series of testing.

It has been found that adding sodium carbonate to sodium hexametaphosphate improves dispersion for some soils compared to when used alone (Bindu and Ramabhadran, 2010). Sodium carbonate increases pH. It also reduces solution hardness by causing precipitation of $Ca(HCO_3)_2$, $CaSO_4$, etc. The increase in pH makes OH groups available to neutralise negative charges at the broken surfaces of the Silica and Alumina sheets of the clay particles

TSPP has better dispersing capabilities than sodium hydroxide and sodium silicate according to Yoon and Mohtar (2014). The study conducted by Wintermyer and Kinter (1955) found TSPP to be highly effective for laterite soil groups but less effective for podzolic soil groups where other four poly-phosphates were highly effective (Na^+ polyphos, Na^+ tripolyphosphate, Calgon and Na^+ tetraphosphate).

Kaur and Fanourakis (2016a) also investigated the effect of type, concentration and volume of dispersing agent. Five dispersants were tested on two soil types, alluvium and black clay. The alluvial soil was from Sebokeng Township in southern Gauteng and black soil from Brits in the North West Province. Hydrometer analysis was used to determine their clay content. Their results indicated that the percentage of clay-sized material from the hydrometer test can fluctuate considerably based on the type of dispersing agent. For alluvial soil, they found that dispersing in 125ml at the concentration 33g plus 7g per litre yielded maximum clay content. However, for their black soil, dispersing in 200ml at the concentration of 35g plus 7g per litre yielded maximum clay content. They further concluded that there is no correlation between the mass of the dispersing agent in solution and the clay content yielded, which seems surprising in light of the findings in the body of their report.

Kaur and Fanourakis, (2016b) also investigated the optimum concentration and volume of three different dispersants using the ASTM hydrometer 152H:E100 (H2) and the Bouyoucos hydrometer 152H (H). The same Black soil was used as for their previous test. They additionally tested a light brown soil from Linksfield in Gauteng and red soil from Springfield, also in the Gauteng Province area. It was not clear which soil activity values were obtained using which hydrometer, but it can reasonably be assumed that the H2 hydrometer was used, since this is prescribed by SANS 3001 (2012). Activities for their black soil, brown soil and red soil were reported as 1.07, 1.5 and 0.5 respectively. Their conclusion from their study was that the use of the H2 hydrometer resulted in a lower clay content for more active soil. They further found that while using two different hydrometers, optimum concentration of Calgon and sodium pyrophosphate yielded almost the same clay content though the concentrations of the dispersants were not equal.

Singh *et al.* (2002) investigated the automation of the hydrometer analysis. The aim of the project was to exclude the labor-intensive part of the hydrometer analysis. However, their investigation did not address the important aspect of clay mineralogy. The results of the Auto-Hydrometer and Hydrometer analysis did correspond with each other. Their investigation also did not address the fact that hydrometer analysis has difficulty with irregular shaped particles, it assumes that all particles are spherical. Bardet and Young (1997) concluded that sedimentation analysis is questionable for determining the grain-size distribution of soil particles smaller than 5 µm accurately and that for 1 µm particles, the results become unreliable due to Brownian motion. They proposed that Buoyancy analysis has advantages when compared to the hydrometer. Duration of the test can be reduced by shortening sedimentation distance. They maintain that the procedure is more accurate than the hydrometer and its corrections are simpler. The sphere used in their analysis is smaller than the hydrometer and this reduces sedimentation disturbance when inserting and removing the sphere. Operator error is minimized as readings can be directly forwarded to a computer. Buoyancy method and hydrometer analysis gave results which agreed well for silt but became questionable with clay.

Rolfe *et al.* (1960) and others highlighted the point that there is no suitable standard for comparing hydrometer results, though attempts have been made to devise universal methods of mechanical analysis.

An M.Sc. thesis by Farouzan (2016) dealing with the behaviour of expansive soils, investigated correlations between various methods used to recognize the swelling potential of soils. Soil index property tests, hydrometer analyses and swelling property tests were conducted on two natural expansive soils from a site in Turkey and on samples of bentonite and kaolinite in mixtures of various proportions. Results of their soil property tests are shown in table 2.1.

Table 2. 1: Soil Index Properties Results. (Forouzan, 2016)

Sample Type	**LS (%)**	**LL (%)**	**PL (%)**	**PI (%)**	**Clay Fraction (%)**	**Activity**	**Specific Gravity**	**Max. Dry Density (cm^3/gr)**	**Optimum Water Content (%)**
100%K	31,61	45,64	44,45	1,19	37	0,03	2,74	1,41	29
10%B+90%K	27,28	72,86	58,22	14,64	39	0,38	2,70	1,36	29,4
20%B+80%K	21,34	112,76	78,82	33,94	44	0,77	2,65	1,34	30,4
30%B+70%K	16,56	158,64	101,22	57,42	47	1,22	2,60	1,3	32,6
40%B+60%K	16,34	198,76	115,46	83,30	49	1,70	2,56	1,26	36,2
50%B+50%K	16,25	249,51	127,62	121,89	55	2,22	2,50	1,24	37,2
Natural Soil Type 1	9,60	64,43	56,18	8,25	57	0,14	2,68	1,38	33,8
Natural Soil Type 2	10,93	86,51	70,32	16,19	53	0,31	2,74	1,4	32,5

The thesis comments positively on the hydrometer results, noting that the higher indicated clay contents correspond with increased heave potential. The author failed, however, to comment on the fact that although all of the kaolinite and bentonite mixtures are nominally 100% clay, the hydrometer results give clay fractions ranging from only 37% for pure kaolinite to 55% for an equal mixture of kaolinite and bentonite. The hydrometer result for pure kaolinite appears to be close to what might be expected by comparison with Photograph 17 in Stott and Theron (2016), where agglomeration of individual kaolinite particles can be seen. All of the nominally 100% clay results indicate that, for high clay content soils, it would be unwise to rely on the hydrometer to give a meaningful assessment of clay fraction. The results suggest that the hydrometer is likely to under-estimate the clay fraction for high clay-content soils by a significant margin. The hydrometer appears to be an inadequate standard by which to judge other procedures for particle size determination of soils.

2.2 Hydrometer

The hydrometer method is discussed below as specified in the following standards:

i. South African Standard, SANS 3001-GR3 (2012) which replaced TMH1 method A6 (1986).

ii. British standard, BS 1377: Part 2: 1990

iii. American standard, ASTM : D 422-63 :1990

A summary of the above standards is included in table 2.1 and the standards are discussed in detail below.

All three standards are based on Stokes' law which estimates the rate at which particles settle in a suspension. Allen (1996) summarized the following assumptions made by invoking Stokes' law:

i. The particle must move as it would in a fluid of infinite extent;

ii. Particles must be spherical, smooth and rigid and there must be no slip between them and the fluid;

iii. Terminal velocity must have been reached;

iv. Settling velocity must be low so that inertia effects are negligible;

v. The fluid must be homogeneous compared with the size of the particle.

Lu et al. (2000) found that the Stokes' equation underestimated the particle dimension by two orders of magnitude for particle size ranging from 0.1 µm to 100µm and aspect ratios from 10 to 500. Their investigation compared hydrometer analysis results, sieve analysis, laser diffraction analysis and scanning electron microscopy (SEM) to confirm their argument that Stokes' equation may not be appropriate for calculating particle-size distributions for fine-graded particles.

Table 2.2: Standards summary

Standard	**Hydrometer Type**	**Dispersing Agent**	**Quantity (ml)**	**Soil Fraction (g)**	**Clay Fraction (µm)**
SANS 3001-Gr3	152H	Na^+ hexametaphosphate (33g) + Na^+ carbonate (7g)	125ml	30	< 2
TMH 1-A6	152H	Na^+ Silicate + Na^+ oxalate	5ml each	50	<5
BS 1377-Part 2	M100	Na^+ hexametaphosphate (33g) + Na^+ carbonate (7g)	100ml	30	<2
ASTM:D422-63:1990	151H or 152H	Na^+ hexametaphosphate (40g) + Na^+ carbonate (NS)	125ml	50	<5

2.2.1 SANS 3001-GR3

Part GR3 of the SANS 3001 is used to determine the clay and silt content of soils, the clay fraction being defined as material smaller than 2µm. The method replaced TMH1 Method A6 and is similar to the ASTM and the BS methods. The following apparatus is specified and the main items are described;

i. ASTM hydrometer 152H

ii. Sodium hexametaphosphate

iii. Sodium carbonate.

Raw samples are prepared according to procedure GR1 or GR5 of SANS 3001. Only material passing the 2mm sieve is used. 100g of sandy material, 50g of silty material or 30g of clayey material are weighed out and used for analysis. Thirty three grams of sodium hexametaphosphate and 7g of sodium carbonate are dissolved in distilled water to make up 1ℓ volume. One hundred and twenty-five millilitres of the dispersant solution is used to treat the 100g, 50g or 30g sample. Stirring with a rod is required until the material is fully wetted. The material is then left to soak for not less than 16hrs. After the soaking period, more water is added to make up roughly 400ml solution. The solution is mechanically stirred for 15min. Immediately after stirring, material is transferred to the sedimentation cylinder and water is added to make up 1ℓ. A blank solution of 125ml of dispersing agent and distilled water to make up 1ℓ is prepared in a second cylinder. A rubber stopper is placed on top of the cylinder containing the treated sample and the cylinder is then shaken, turning repeatedly upside down, for 1 min. The sedimentation cylinder and blank solution are put in a convenient place, which may be a temperature controlled water bath. A set of readings is taken after 1hr. The cylinders are then shaken, turning repeatedly upside down for 1 min. Readings are then taken at 40s, 2min and 12min. without removing the hydrometer. The hydrometer is taken out, rinsed in filtered water, and immediately placed into the blank solution. If sizes of 0.002 mm or smaller are required, a further set of readings is taken after 12h. The hydrometer is inserted 20s before the scheduled readings are to be taken. After the final hydrometer reading, the material from the cylinder is washed through the 425µm and 75µm sieves until the wash water is clear. Retained material is oven dried between 105 ºC and 110 ºC. Dried material is weighed and recorded. The hydrometer readings are corrected using factors for different temperatures. The percentage of material in suspension is then calculated. SANS categorise clay sized particles as particles smaller than 2µm.

2.2.2 TMH 1 – A6

TMH1 was replaced with SANS 3001. TMH1 specified:

i. ASTM hydrometer 152H

ii. Sodium silicate

iii. Sodium oxalate

Samples are prepared as per SANS 3001 but the dispersant, sodium hexametaphosphate and sodium carbonate, is replaced by sodium silicate and sodium oxalate as specified in TMH1. TMH1 soaking is specified to be not less than 2 hours or preferably overnight. Readings are taken at 18s, 40s, and 1hr. In TMH1, clay sized particles are particles smaller than 5µm.

2.2.3 British standard, BS 1377: Part 2: 1990

The British standard specifies sieves 425µm, 300µm, 212µm, 150µm and 63µm. Wet-prepared material passing 63µm is used for hydrometer testing. The standard points out that oven drying of tropical soils can change particle size properties, therefore such soils should not be dried in an oven.

BS 1377 specifications:

i. Hydrometer M100, 250mm.

ii. Hydrogen peroxide (where appropriate)

iii. Sodium hexametaphosphate (33g)

iv. Sodium carbonate (7g)

Thirty grams of clay is specified for the test, 150 ml of hydrogen peroxide is used for overnight pre-treatment (of organic soils only) before dispersing with the specified agent. Dispersing treatment is minimum 4hrs or overnight. Readings are taken after 0.5min, 1min, 2min, 4min, etc (Up to 24hrs). Clay sized particles are specified as “< about 2µm”.

2.2.4 American standard, ASTM: D 422-63:1990

Particle fraction smaller than 2mm is assessed by the sedimentation process, using either ASTM hydrometer 151H or 152H.

i. 151H or 152H Hydrometer

ii. Air-jet dispersion cup

iii. Sodium hexametaphosphate (40g)

iv. Sodium carbonate (amount of sodium carbonate is not specified, however the chemical is mixed with Na^+ hexametaphosphate to adjust pH to between 8 and 9)

Fifty grams of clay soil is treated with 125ml of sodium hexametaphosphate (40g/ℓ), soaking for 16hrs. After soaking, air jet or mechanical stirring is used following which readings are taken at intervals 2, 5, 15, 30, 60, 250 and 1440min. ASTM gives clay size fraction as particles less than 5µm and silt size between 0.074mm to 5µm. ASTM D 422-63 was withdrawn in 2016 and replaced by ASTM D 7928-17.

CHAPTER 3 : STUDY DESIGN

3.1 Introduction

The study design was developed to test some aspects of hydrometer theory. A settlement container was developed for the purpose of the study (see Section 4.3.2); it was designed to be waterproof while having a removable cover sealed by a waterproof paper gasket. It also has a removable top cover to prevent dust contamination. An optical microscope equipped with a 9-megapixel digital camera was used to examine settled sediments. In the process of evaluating the reliability of the hydrometer, soil behaviour when treated with different dispersing agents was also evaluated.

3.2 Chemicals

The chemicals used in this research project, together with their formulas is given in the table below. Chemicals number 1 to 6 were used as dispersing agents.

Table 3. 1: Chemical formula

No.	Name	Referred as	Formula
1	Sodium hexametaphosphate	Calgon	$(NaPO_3)_6$
2	Sodium carbonate		Na_2CO_3
3	Sodium silicate	Sodium Silicate/Oxalate	Na_2SiO_3
4	Sodium oxalate		$Na_2C_2O_4$
5	Tetrasodium diphosphate decahydrate	TSPP	$Na_4P_2O_7$ * 10 (H_2O)
6	Sodium pyrophosphate		$Na_4O_7P_2$ * 10 (H_2O)
7	Methylene blue	MB	C16H18ClN3S

3.3 Dispersing agents

The function of a dispersing agent is to assist the mechanical treatment in separating particles drawn together by electrostatic forces and prevent flocculation of dispersed particles during the test. Stott and Theron (2016) described three ways that dispersants work:

i. Replacing multi-valent ions by monovalent ions at the clay surface.

ii. Reacting with multi-valent ions to form chemical complexes, making them unavailable for attraction to clay surfaces.

iii. Forming functional groups which act as spacers between the clay particles, effectively preventing them from approaching each other.

The process involved in dispersing soils for testing is a combination of mechanical and chemical dispersion. In practice, it is common that after chemically treating samples they are mechanically agitated using a motor driven stirrer. It is, however, important to note that the chemical dispersion aspect has not been resolved and no currently available theory accounts for all aspects of dispersion.

3.3.1 Calgon

The dispersant specified in SANS is sodium hexametaphosphate and sodium carbonate commonly known as Calgon. Thirty three grams of sodium hexametaphosphate and 7g sodium carbonate (33:7) were dissolved in distilled water to make up 1ℓ volume of solution. One hundred and twenty-five millilitres of this solution was added to each sample.

3.3.2 Sodium silicate plus Sodium oxalate

Sodium oxalate is supplied in a solution and sodium silicate as a solid. They were mixed as per TMH1 (1986). Sodium silicate was dissolved in filtered water until the hydrometer gave a reading of 36 at 20ºC, the solution was filtered and stored for future use. Five millilitres of each component were mixed to form the dispersant mixture.

3.3.3 Tetrasodium pyrophosphate

Thirty six grams of TSPP was mixed with distilled water to bring to 1ℓ volume of solution. Following the procedures of Kaur and Fanourakis (2016a), 20ml of this solution was used for each specimen.

3.3.4 Methylene blue

Methylene blue A.R. grade was used for this research, 98.5% pure in a 5g/l aqueous solution. As the investigation was not quantitative, a lower quality grade could have been used. Without MB staining of samples before examination under the microscope, it was not obvious whether clay minerals were present or not as seen in Figure 3.1 and Figure 3.2.

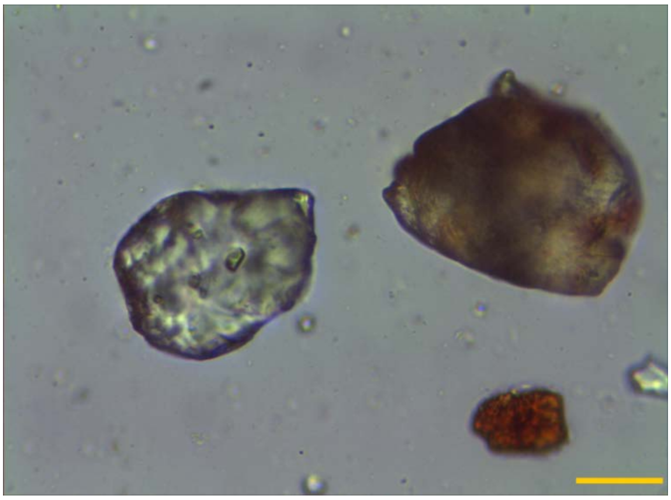

Figure 3. 1: Typical dispersed sand layer MS photographs before MB addition, 40x objective. Scale bar 30 µm x 2 µm

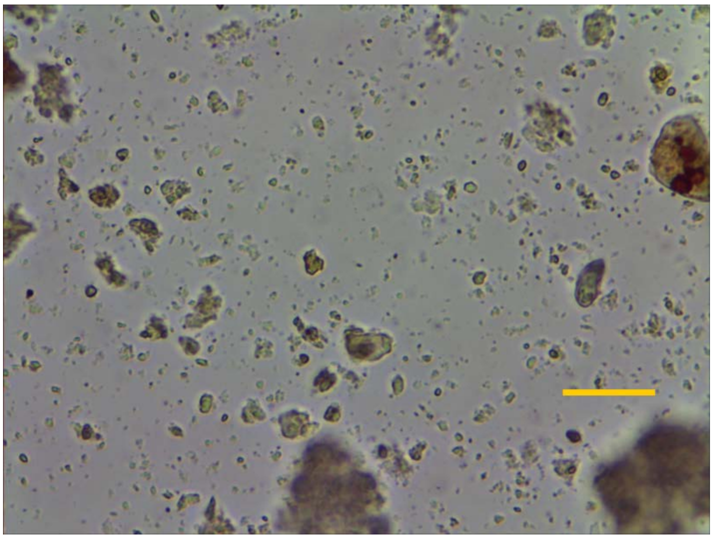

Figure 3. 2: Typical dispersed silt layer MS photographs before MB addition, 40x objective. Scale bar 30 µm x 2 µm

Highly active clays, like smectites, have high CEC and SSA. Such clays exchange cations with MB readily and become stained easily. Less active clays like kaolinite and halloysite have low CEC and SSA and high concentration of MB is required for them to become visibly blue stained. (Stott *et al.* 2016). For the purpose of this study, MB solution with a concentration of 5g/ℓ was used. Samples were examined before MB addition. Thereafter, a small quantity of MB solution was progressively added to the soil suspensions and samples were extracted for microscopic examination after each addition.

CHAPTER 4 : METHODOLOGY

4.1 Introduction

To assess Savage's assertion that many types of clay are not fully de-floculated (dispersed) at the time of testing, a pilot study was conducted where clay samples were immersed in the dispersant and then assessed with a Particle Size Analyzer using laser diffraction and ultrasound analysis. Microscopic examination proved that a significant clay fraction was still attached to silt-sized particles and that most of the samples were not fully dispersed.

Hydrometer results for the investigated samples are included. Results from soil 1 samples are from a commercial laboratory. Results from soil 2 to 6 samples are from the Central University of Technology's soil mechanics research group. Results are in included in table 4.1. The commercial laboratory and the research group followed TMH1 methods A1 to A6.

This study does not make numerical comparisons of clay fraction with the hydrometer or any other method, since no meaningful standard appears to be available - as noted in section 2.1, following Table 2.1 from Farouzan (2017). This study only investigates some reasons for the test being unreliable and gives graphic illustration of the correctness of some of the suggestions which several researchers have made as possible explanation for this lack of reliability.

Soil samples used for the study represent typical expansive soils from the central Free State. The central Free State is underlain by rocks of the Beaufort Group and Adelaide Groups of the Karoo Supergroup. The Karoo Supergroup covers a large area of Southern Africa. There are frequent intrusions of dolerite sills and dykes. Mudrocks of the Beaufort Group, as well as dolerite, are known to weather into expansive clays under the sub-humid and semi-arid conditions. Soil samples (Table 4.1) were from the following sites: Soil 1, a road project at Thaba Nchu intersection (N8 and Hoog Street). Soil 2 from the M10 roads project 6km from the Bram Fischer airport and a Heidedal, Bloemfontein, building project (Soils 3 to 6).

Table 4. 1: Soil Samples description

Sample Name	Location	(LL)	(LS)	(PI)	(w%)	Clay(%)	Clay Activity
Soil 1	Thaba Nchu	60	13	36	8	36%	Very high
Soil 2	M10	63	17	39	13	35%	Very high
Soil 3	Heidedal Building project	Non Plastic					
Soil 4		37	11	22	16	11%	Low
Soil 5		47	12	32	13	13%	Low
Soil 6		54	13	35	16	17%	Low

4.2 Apparatus

The following apparatus was used as per SANS 3001.

i. Settlement container
ii. Weighing balance up to 200g
iii. Sieves: 2 mm, 425 μm, 75 μm, pan and cover
iv. Glass jar with a lid
v. Stopwatch
vi. Thermometer
vii. Mechanical stirrer rotating at 1570 rpm (Ryobi pedestal drill press used as a stirrer)
viii. Drying oven
ix. Microscope
x. MB solution
xi. TSPP
xii. Calgon
xiii. Sodium silicate/oxalate.
xiv. Stirring wire.

4.3 Method procedure

4.3.1 Sample preparation

All tests soil samples were prepared in the same way except for dispersant volume, which is dispersant-dependent. A dry preparation of samples was followed. Wet preparation often gives a significantly higher clay fraction for coarse soils. The samples tested had so little coarse material that wet preparation would have made a negligible difference to these qualitative analyses. The material was transferred from sample bag to a mortar with rubber tipped pestle for disaggregation. The material was sieved and 50g mass of material which passed the 425µm sieve was used for testing. Dispersing preparations and procedure are discussed in chapter 3. SANS 3001 calls for fines to be covered by 125ml dispersant for 16hours minimum. After soaking, distilled water was added to make up 400ml of solution before stirring. The dispersed mixture was mechanically stirred for 15min then transferred to a settlement container (Figure 4.3), distilled water was added to the cylinder to fill. The dispersed solution (Figure 4.1) was agitated using a wire stirrer and then left to settle for four days. After four days, the water was syphoned off using medical tubing. The sample was then dried in two phases. The first phase of drying was in an oven at 45^o C until visible water had evaporated (Figure 4.2) and the sediment was close to the plastic limit. In the second phase, the top of the sample was supported by packing, the cylinder was turned horizontal with the cover uppermost, the cover was removed and the sample was allowed to air dry. Finally the individual layers within the sedimented sample were physically separated. Samples were then ready for microscopic examination.

The observed layering of the sediment was not always the same. Samples treated with Calgon tended to fall into one of two categories. In the first category, the samples separated into three layers: sand, silt and a very thin clay layer which was too thin to be examined with the microscope). In the second, notably for soil 3 to soil 6, the sediments separated into five layers; coarse sand, fine sand, coarse silt, fine silt and a very thin clay layer. Sodium silicate/oxalate sediments followed the Calgon pattern but with a thicker clay layer - sufficient to examine under the microscope. TSPP sediments separated into three layers; sand, silt and a reasonably thick clay layer sufficient to examine. Microscope images of all samples before addition of MB had pale pinkish, cloud-like patches as illustrated by Figures 3.1 and 3.2. These figures show various particles attached to sand grains. MB was added progressive and in small quantities. Methylene blue staining indicated that the pinkish clouds are small, almost transparent, high CEC particles.

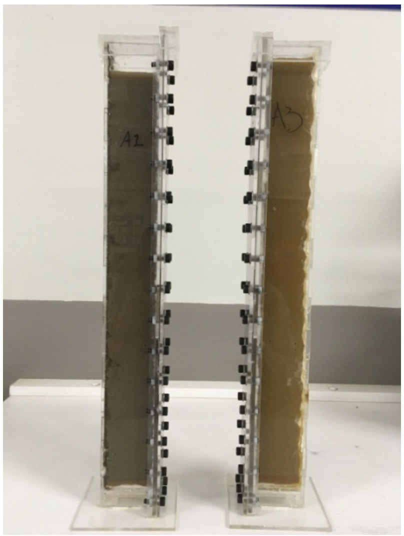

Figure 4.1: Dispersed soil solution after agitation.

Figure 4.2: Typical example of settled semi-dry sample with four layers; sand, coarse silt, silt and thin layer of clay.

4.3.2 Settlement container

The settlement container was developed for the purpose of this research. It was a waterproofed container having one side removable for the extraction of settled samples. A paper gasket and silicone grease were used to create a waterproof seal. Figure 4.3 displays two of these containers, before and after assembly, with gasket and front cover in place. Cyanoacrylate glue was used to bond perplex pieces together and filling powder was used to close gaps.

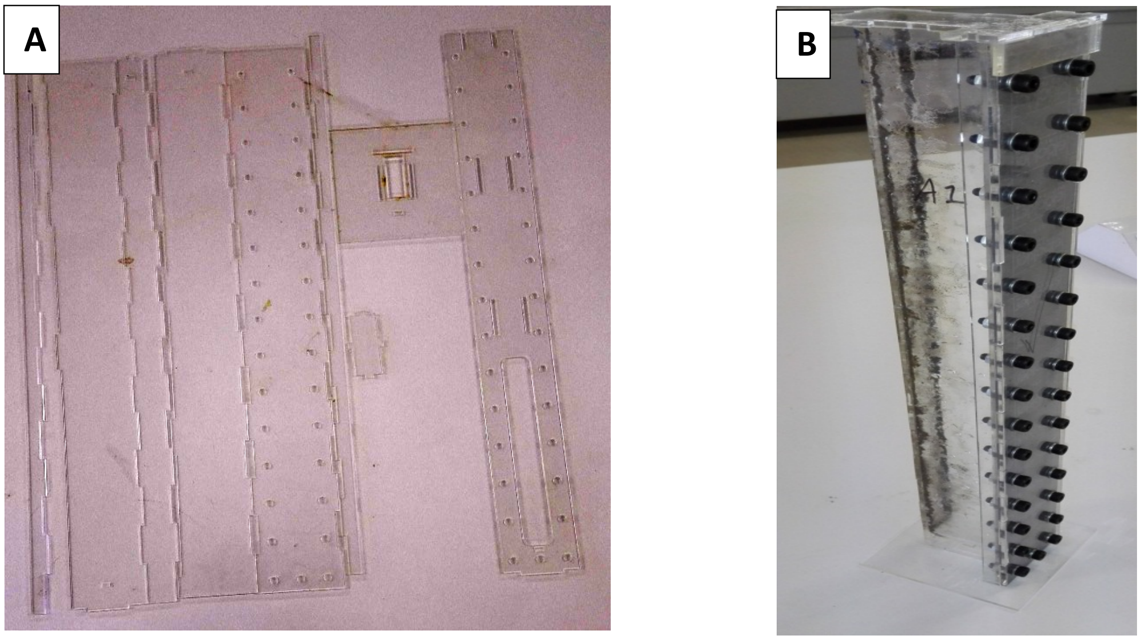

Figure 4.3: 0.43 ℓ Settlement container: 430x50x20mm: A, settlement container before assembling; B, complete settlement container with cover and paper gasket.

4.3.3 Microscopic examination

An optical microscope (Figure 4.4), with 10x, 40x, 60x and 100x objectives, was used for this study as it is suitable for examining material in suspension. The microscope was equipped with a 9-megapixel digital camera. The combined optical and digital magnification was assessed by measurements on a micro ruler. Magnification can be expressed in various ways, for example the spacing between 10-micron graduations on the micro ruler was 60 mm on the viewing screen when using the 60x objective, indicating a magnification of 6000 times. Alternatively, the magnification can be expressed as the number of pixels per micron (22.48 for the same objective). All microscope photographs used were taken with 40x objective unless otherwise stated, as the depth of focus produced better photographs to analyze. The most useful way of indicating magnification - a scale on photographic results - was included, all photographs shown here have a scale rectangle, usually with dimensions 30µm x 2µm.

After removal of sediment from the container and separation of the visible layers, small samples of each layer were suspended in de-ionized water. A drop of that suspension was placed on a clean microscope slide and covered with a similarly clean cover glass. Several slides were prepared and examined. A small quantity of MB was added to the suspension and new slides prepared and examined. This process was repeated until the staining of samples became too dark to reveal useful information.

Figure 4.4: UB200i series biological microscope with the 9 megapixel camera.

CHAPTER 5 : RESULTS AND DISCUSSION

5.1 Soil 1- Sand Layer

5.1.1 Calgon

The sand layers, which accumulated at the lower part of the containers at the start of the sedimentation process, were expected to contain small quantities of silt and clay. This expectation was met, as it will be observed throughout the sand layers for different dispersing agents and all soil samples.

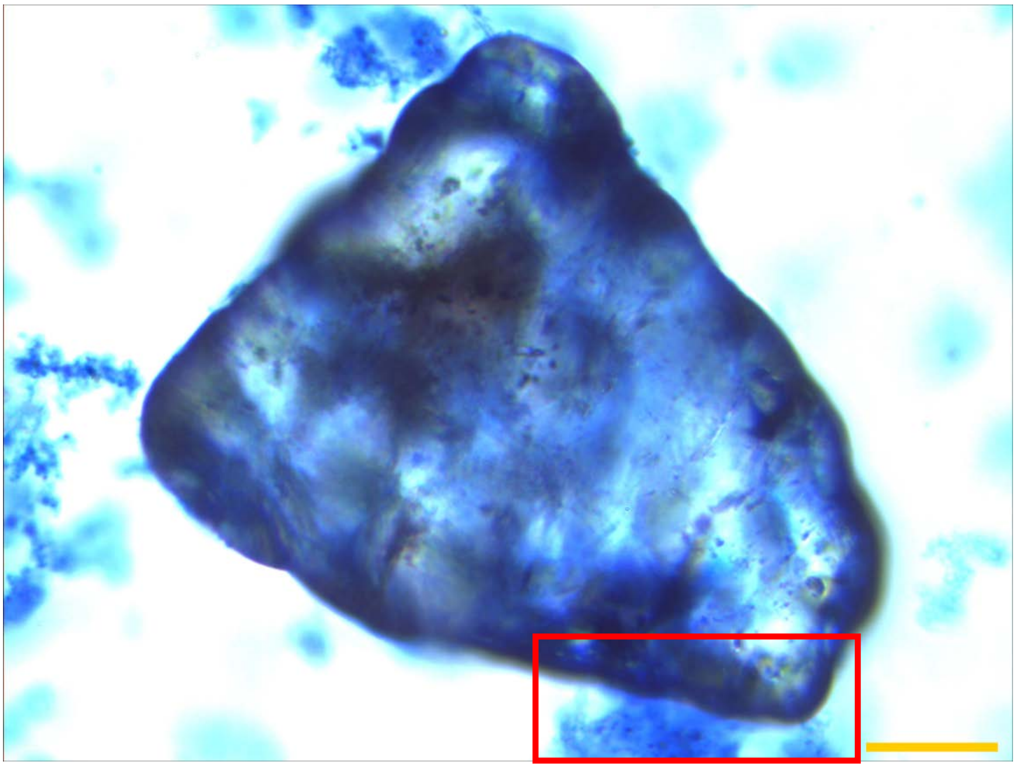

Figure 5.1: Sand layer from soil 1; Calgon - 1.

Figure 5.1 is a view of a coarse sand grain which is completely covered with extremely small, blue-stained, high CEC particles. Patches of blue stained, extremely small particles are also visible around the sand grain. Within the red rectangle a silt-size agglomeration (which appears to be made of small clay particles) is attached to the sand grain.

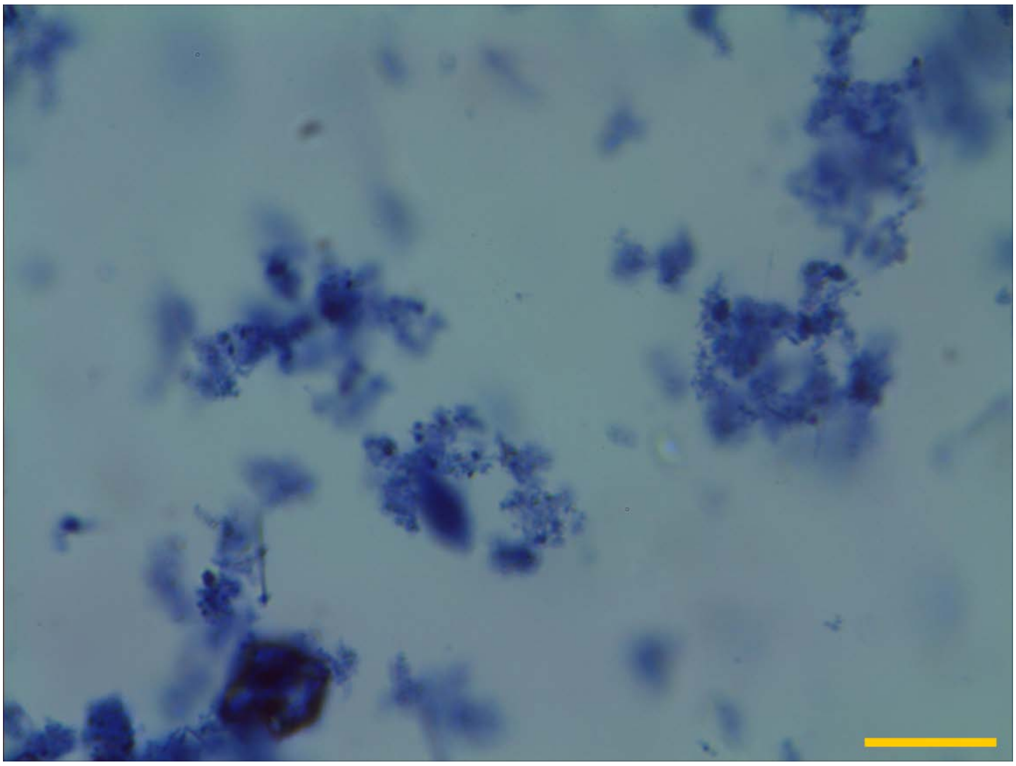

Figure 5.2: Sand layer from soil 1; Calgon - 2.

Figure 5.2 shows agglomerations of clay-sized particles some with silt cores and some without. The string-like structures are completely blue stained.

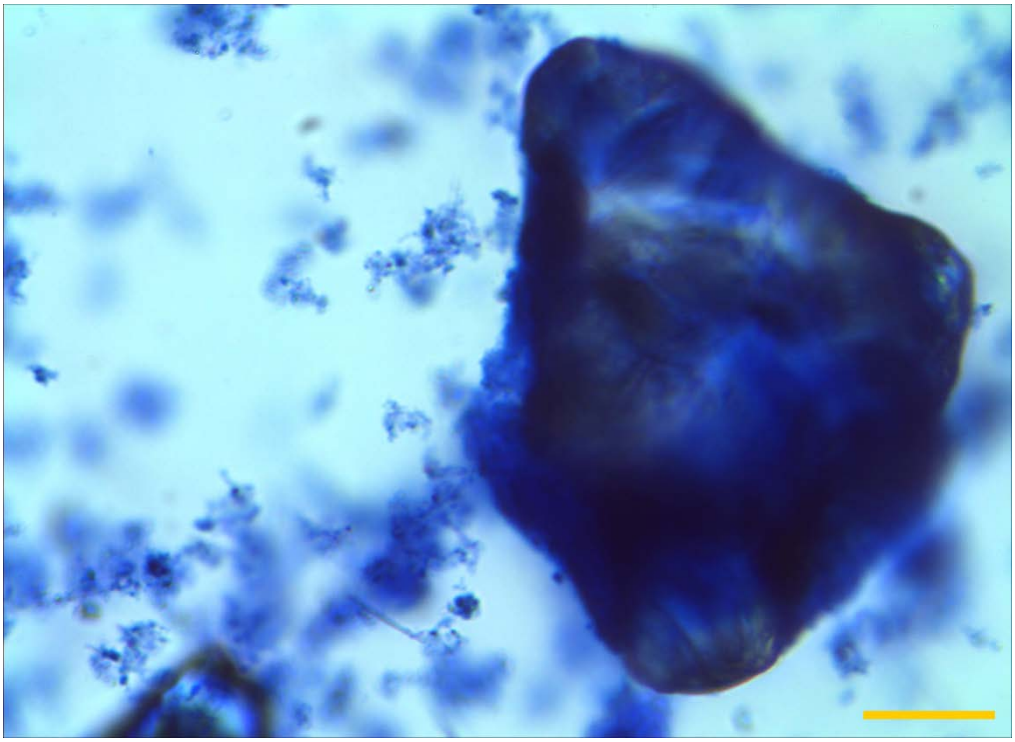

Figure 5.3: Sand layer from soil 1; Calgon - 3.

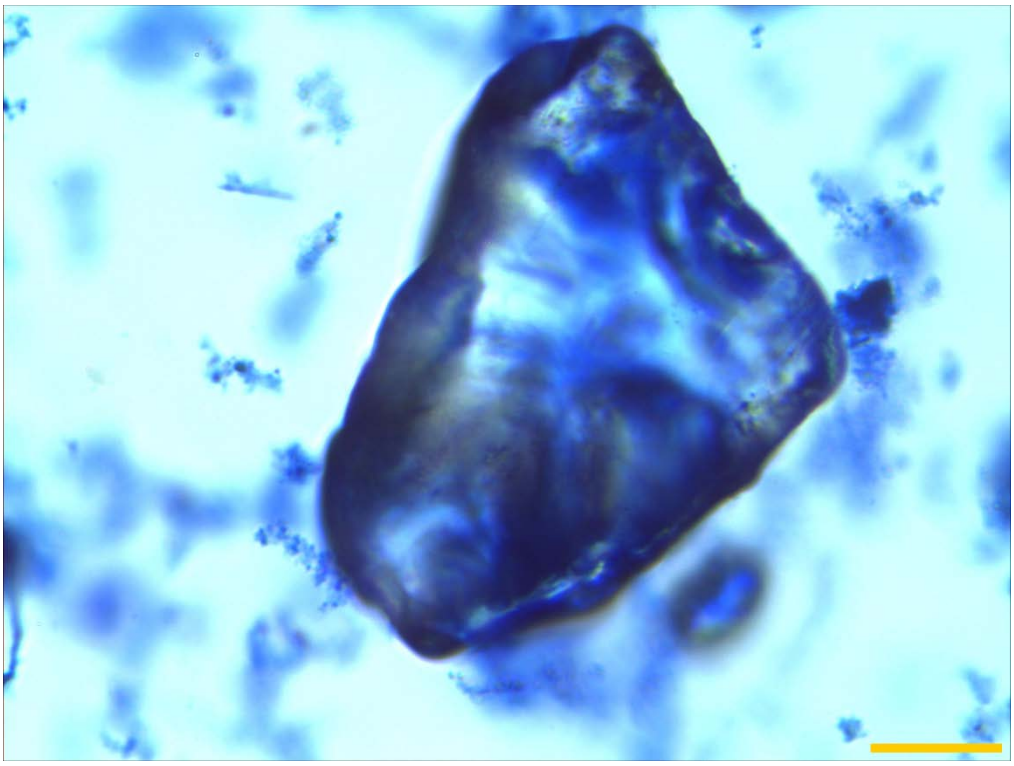

Figure 5.4: Sand layer from soil 1; Calgon - 4.

Figure 5.3 and 5.4 have a similar pattern to Figure 5.1. String-like structures mainly, without silt cores, are attached to the sand grain. All of the particles are completely blue-stained, consisting of minute particles of high CEC minerals. The sand grains in all the Calgon-treated soil 1 sand layer show the fuzzy blue-stained edges, consisting of very small, deeply stained, clay particles around the grain. From this, it is concluded that dispersion was not complete at the time of testing.

String-like structures of high CEC particles are present in all the photographs. The sand grains are completely blue stained, and silt and clay sized particles are visible in agglomerations. In conclusion, the sand layer from soil 1 treated with Calgon contains a significant (non-negligible) fraction of particles which should not be classified as sand.

5.1.2 Sodium Silicate/Oxalate

Some of the silt- and clay-sized particles in Figure 5.5 are independent and not attached to each other. However, silt and clay-sized particles are present in an agglomeration which is completely blue stained. The sand grain appears to be a dark-coloured mineral. However, in the middle of the grain, deeply stained small particles are apparent. Below the 30x2 micron scale bar there is a silt-sized particle which also appears to be a dark mineral completely covered with very small deeply stained clay particles.

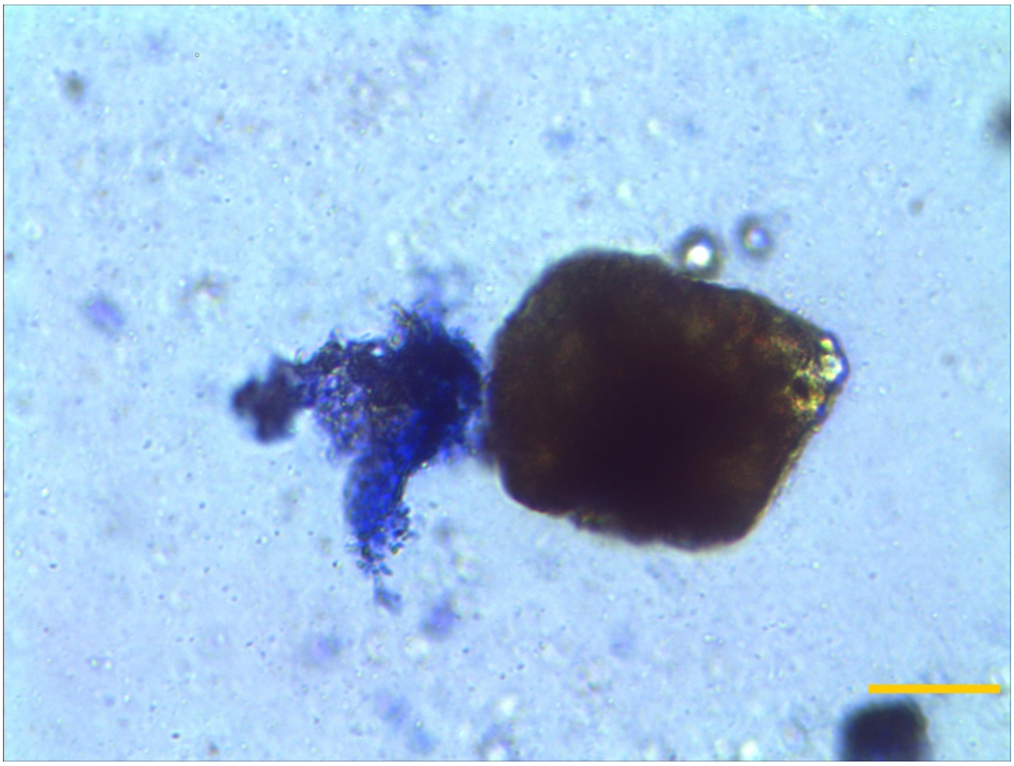

Figure 5.5: Sand layer from soil 1; Sodium Silicate/Oxalate -1.

Figure 5.6 shows two sand grains covered with deeply blue-stained clay particles forming a bridge between them. Clay particles without a silt core can be seen attached to the sand grain. Free silt and clay particles which are blue stained are visible in the photograph’s background.

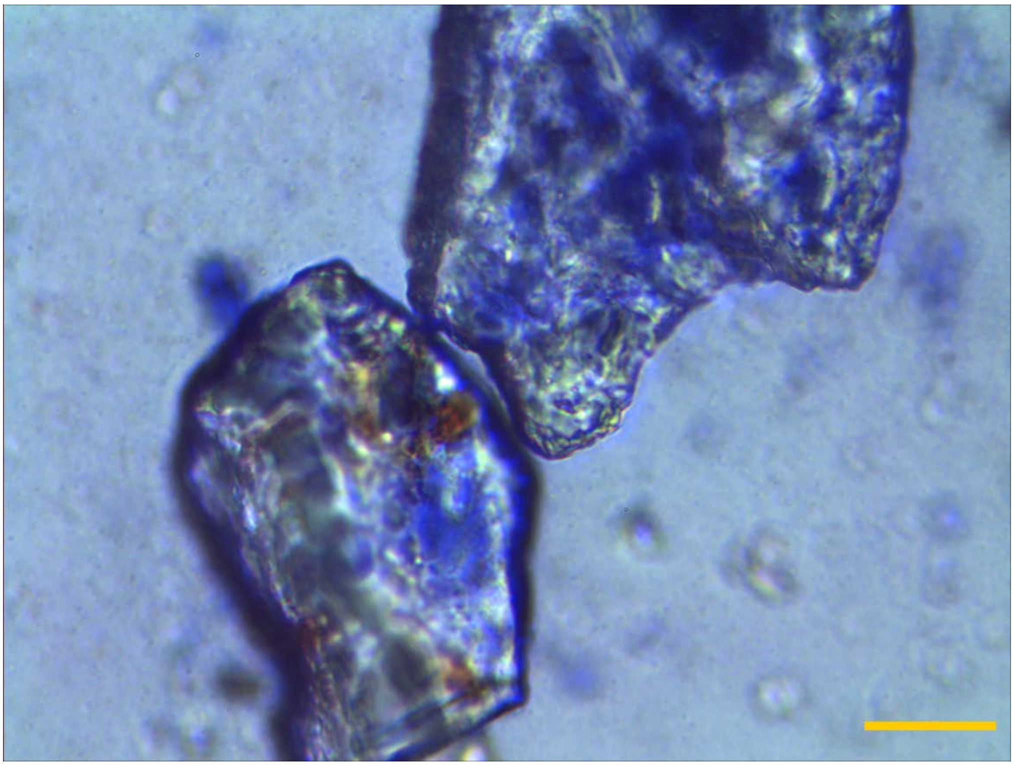

Figure 5.6: Sand layer from soil 1; Sodium Silicate/Oxalate -2.

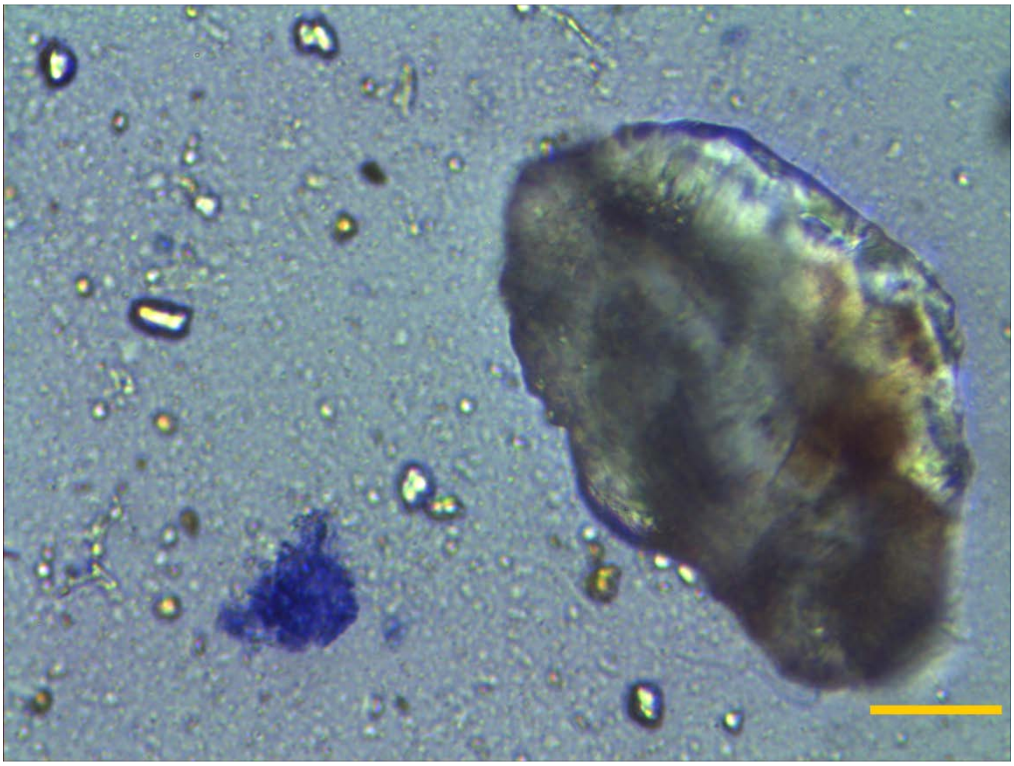

Figure 5.7: Sand layer from soil 1; Sodium Silicate/Oxalate -3.

Figure 5.7 and 5.8 have sand grains which are partially covered by small, deeply blue stained particles. Agglomerations of silt and clay particles are visible in both photographs.

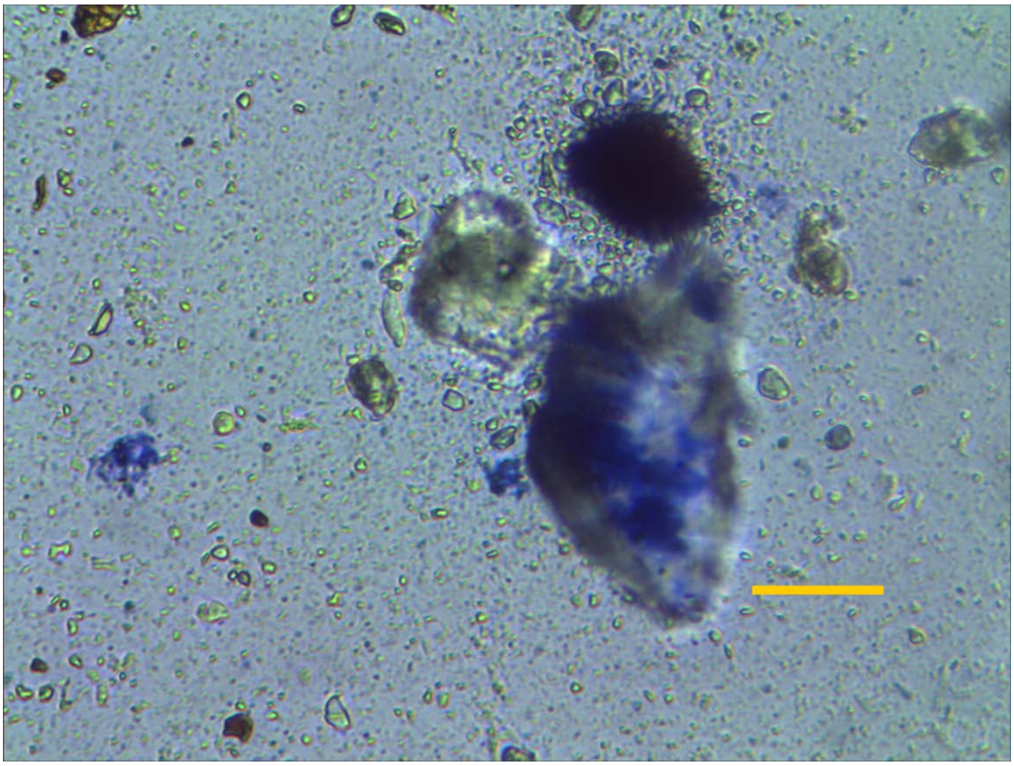

Figure 5.8: Sand layer from soil 1; Sodium Silicate/Oxalate -4.

The Sodium silicate/oxalate appears to leave more silt sized particle not bound together by clay to form agglomerations. Although agglomerations are visible in all the photographs, they are less prominent compared to the Calgon solution. It appears that there are more, unattached silt and clay sized particles visible in the sand layer as compared to Calgon.

5.1.3 TSPP

Figures 5.9 and 5.10 have free silt-sized particles with deep blue stains on the edges. The background has extremely small blue stained clay particles. The sand grains have stained clay particles clinging to them. A cluster of extremely small particles without silt core in Figure 5.10 is evident in a red circle.

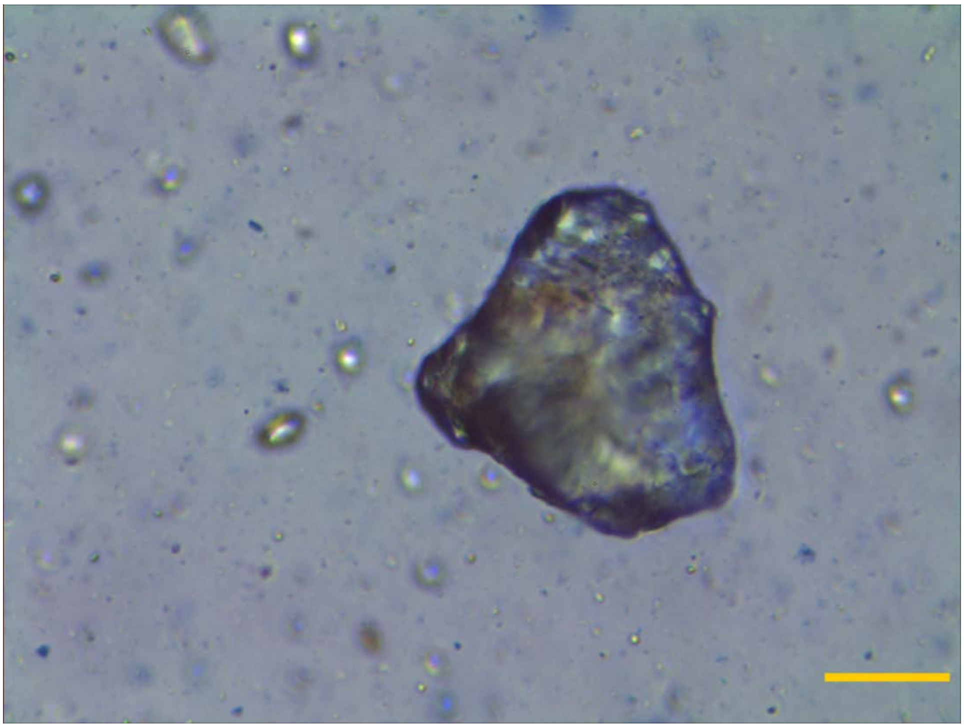

Figure 5.9: Sand layer from soil 1; TSPP. -1.

Figure 5.10: Sand layer from soil 1; TSPP -2.

Free silt sized particles and extremely small clay particles are present in the sand layer. The agglomerations inside the red circle appears to consist only of extremely small clay particles without silt cores. It is noted that the sand grains also have extremely small stained clay particles attached to them in both photographs.

5.2 Soil 1 - Silt layer

5.2.1 Calgon

It is not clear whether the deeply blue stained agglomeration of small, high CEC particles in Figure 5.11 has a silt core. Very small high CEC particles absorb MB more rapidly than low CEC particles and this could explain the unstained clay-sized particles in the photographs.

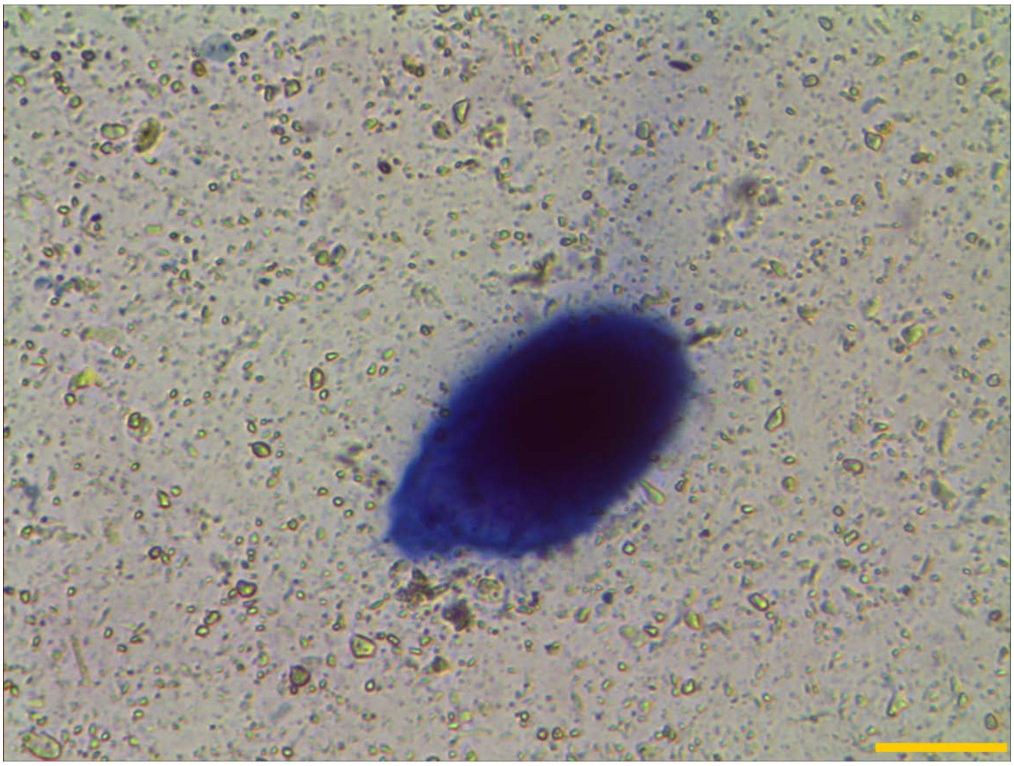

Figure 5.11: Silt layer from soil 1; Calgon -1.

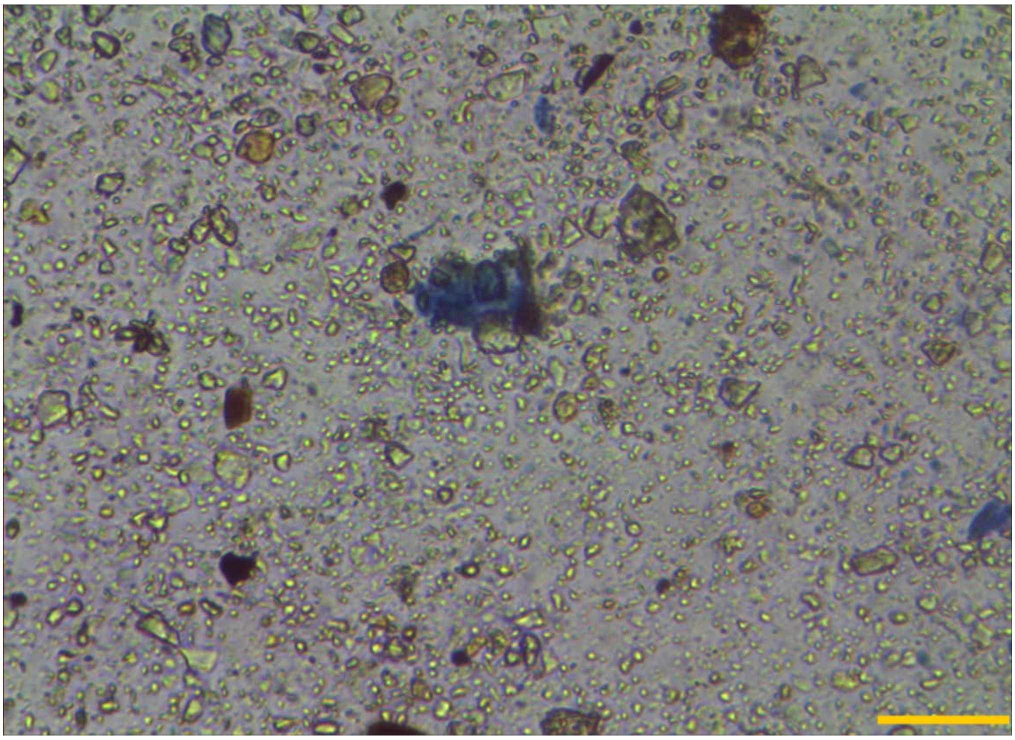

Figure 5.12: Silt layer from soil 1; Calgon -2.

Figures 5.12 to 5.14 have the same pattern as Figure 5.11 with agglomerations of high CEC particles and the yellowish coloured particles that seems to be attached to silt sized particles.

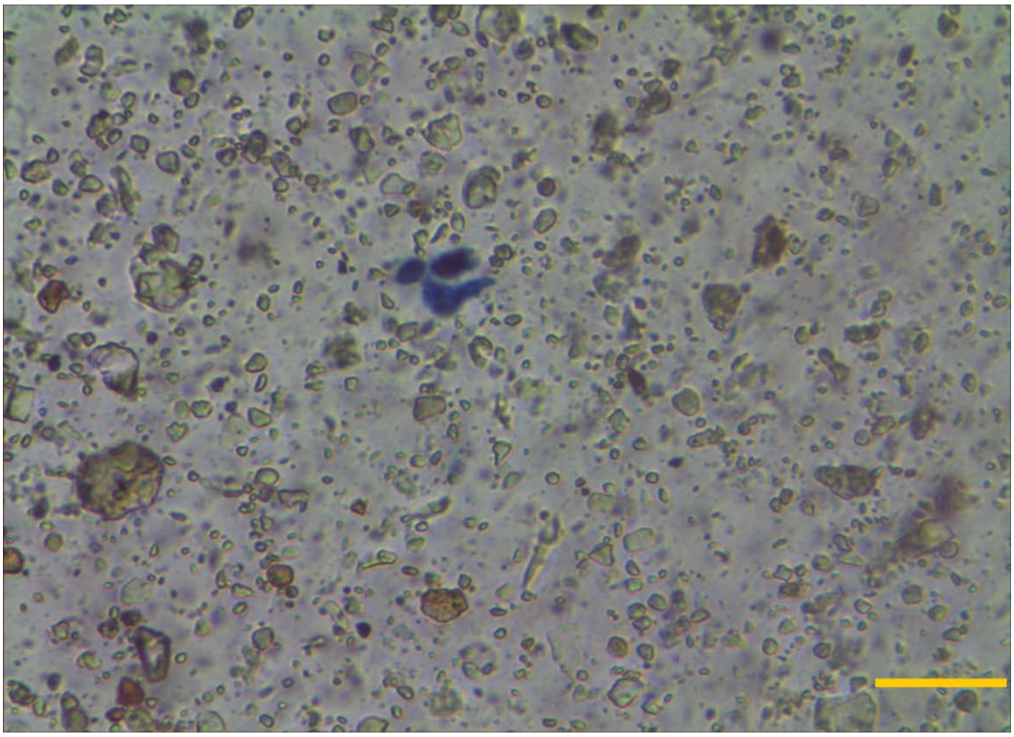

Figure 5.13: Silt layer from soil 1; Calgon -3.

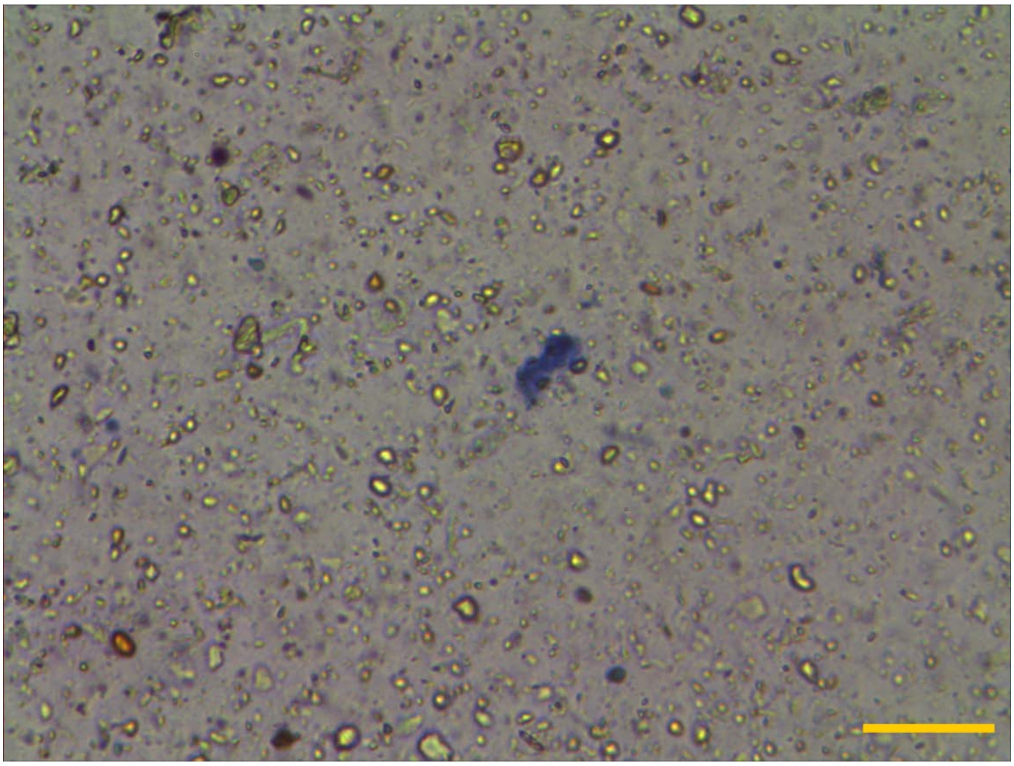

Figure 5.14: Silt layer from soil 1; Calgon -4.

In summary the photographs of the silt layer treated with Calgon show that they all have clay sized particles of about 2microns, many of them in the form of fine silt sized groups of clay particles. Large numbers of clay particles and groupings of them where not prominent in the Calgon treated sand layer.

5.2.2 Sodium Silicate/Oxalate

The deeply blue stained agglomerations in Figure 5.15 appear to be silt-sized particles with clay bonding the silt cores together. The silt particle cores appear to have low CEC particles attached to them. Yellowish coloured particles attached to silt cores are more visible than with the Calgon-treated samples.

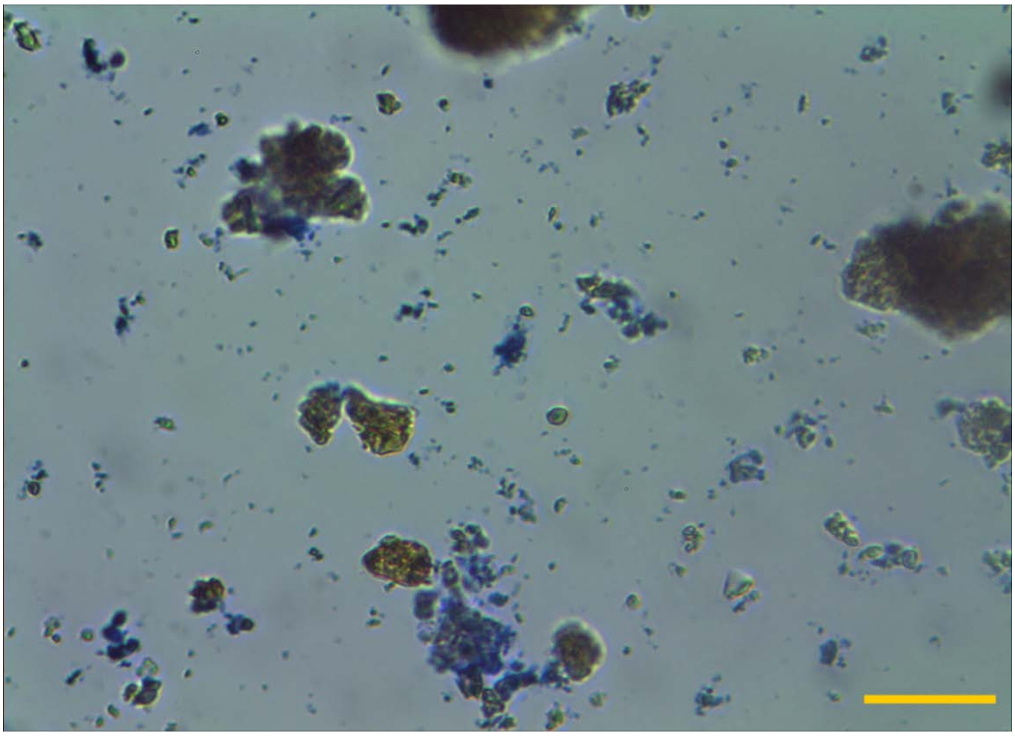

Figure 5.15: Silt layer from soil 1; Sodium Silicate/Oxalate -1.

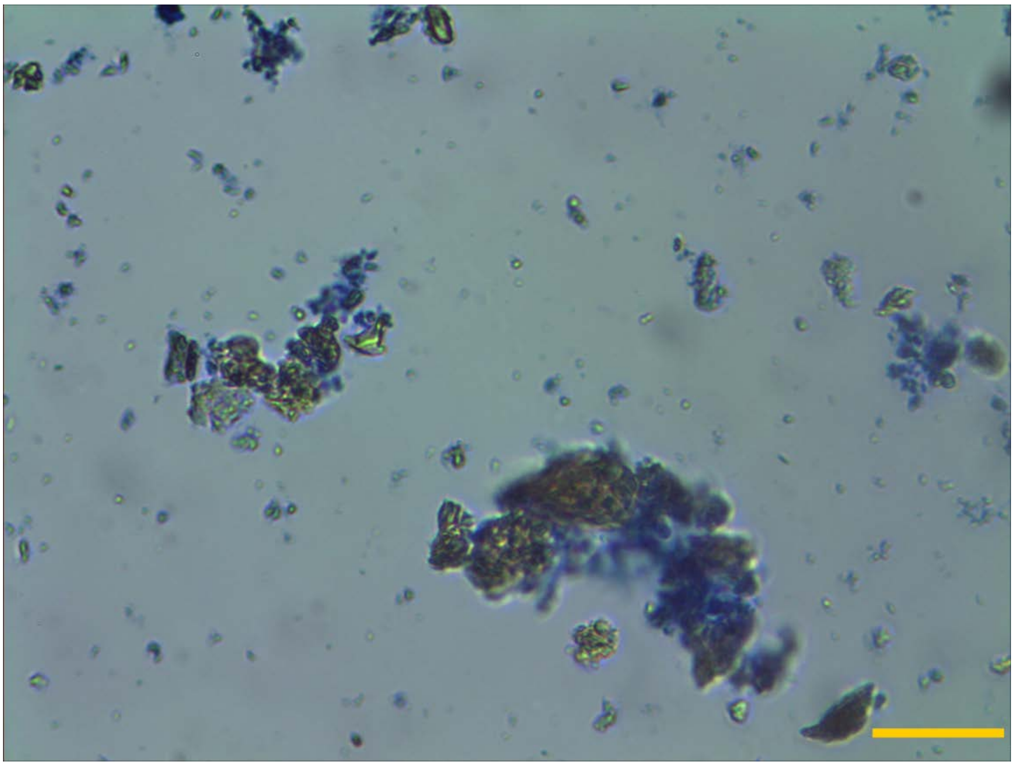

Figure 5.16: Silt layer from soil 1; Sodium Silicate/Oxalate-2.

Figures 5.15 to 5.18 shows agglomerations with low and high CEC particles attached to silt cores forming bridges.

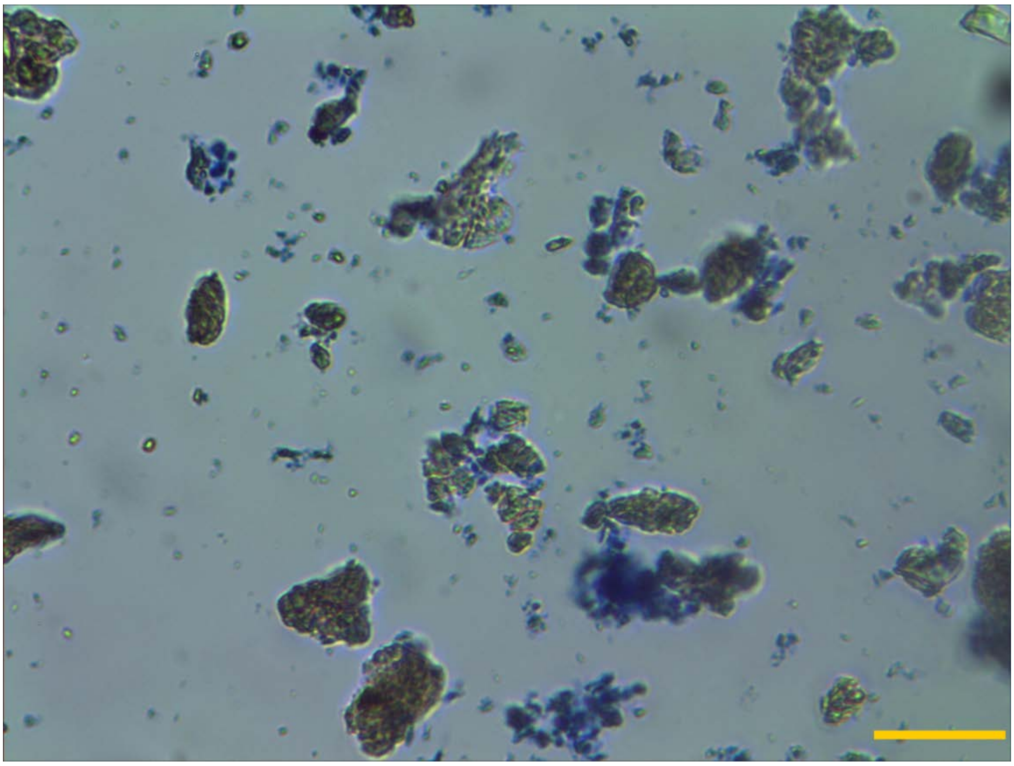

Figure 5.17: Silt layer from soil 1; Sodium Silicate/Oxalate-3.

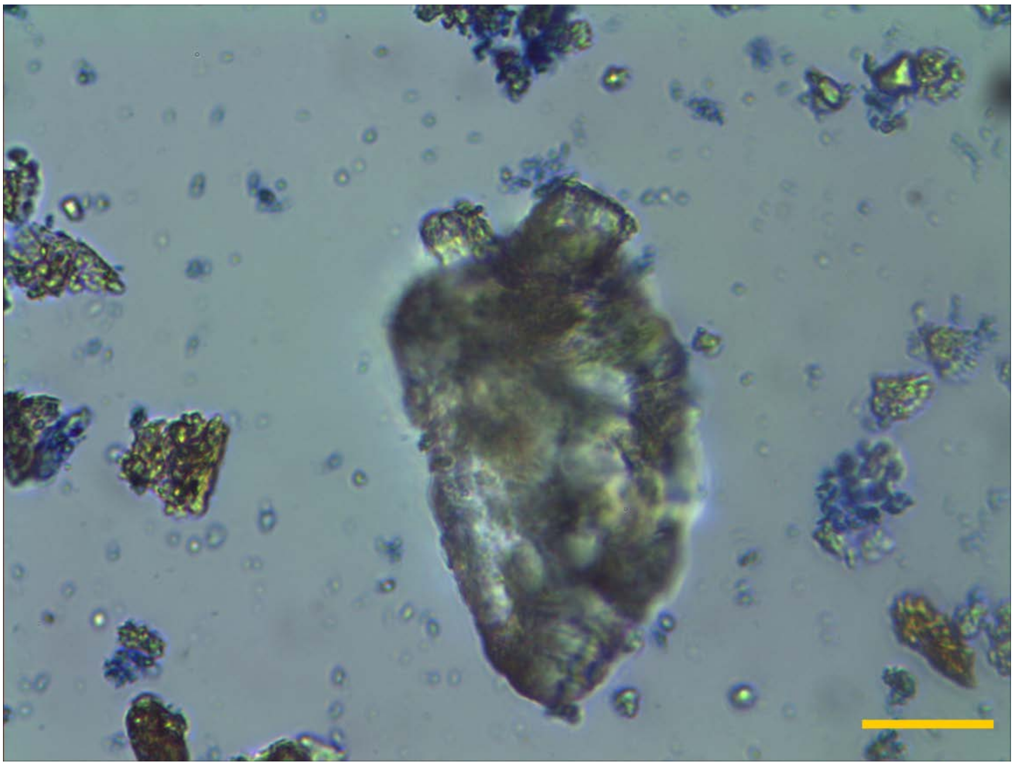

Figure 5.18: Silt layer from soil 1; Sodium Silicate/Oxalate-4.

There seem to be more agglomerations and less free clay sized particles in the silt layer than in the sand layer of the same sample treated with Calgon.

5.2.3 TSPP

Dispersing using TSPP looks more successful and consistent through the sand and silt layers. Most of the particles are completely blue stained. Low CEC particles are not stained and display yellowish in colour, example of yellowish particles are within the red oval shapes.

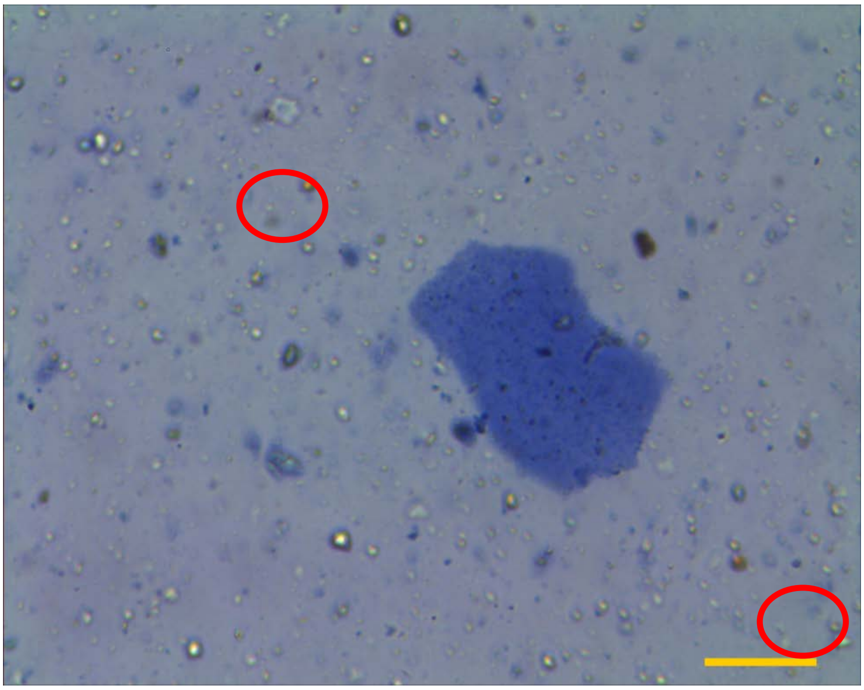

Figure 5.19: Silt layer from soil 1; TSPP -1.

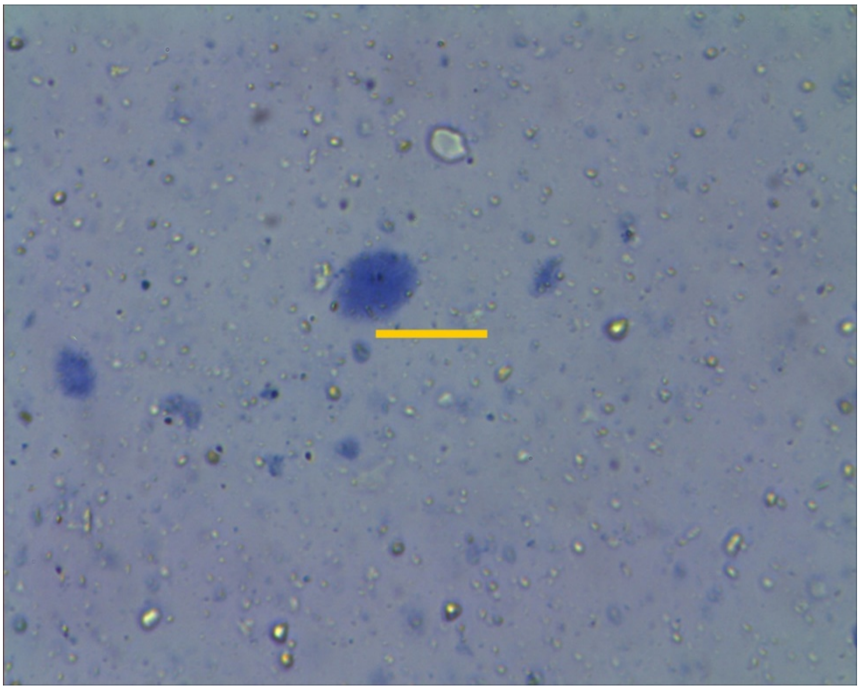

Figure 5.20: Silt layer from soil 1; TSPP -1.

For TSPP, agglomerations of silt sized particles were not observed, there are very few clean silt sized particle visible in the photographs. Agglomerations in this layer appears to be only clay particles.

5.3 Soil 2 - Sand Layer

5.3.1 Calgon

The sand grain in Figure 5.21 is completely covered with small, blue stained particles. A sand-sized agglomeration of high CEC particles with silt cores is shown within the red rectangle.

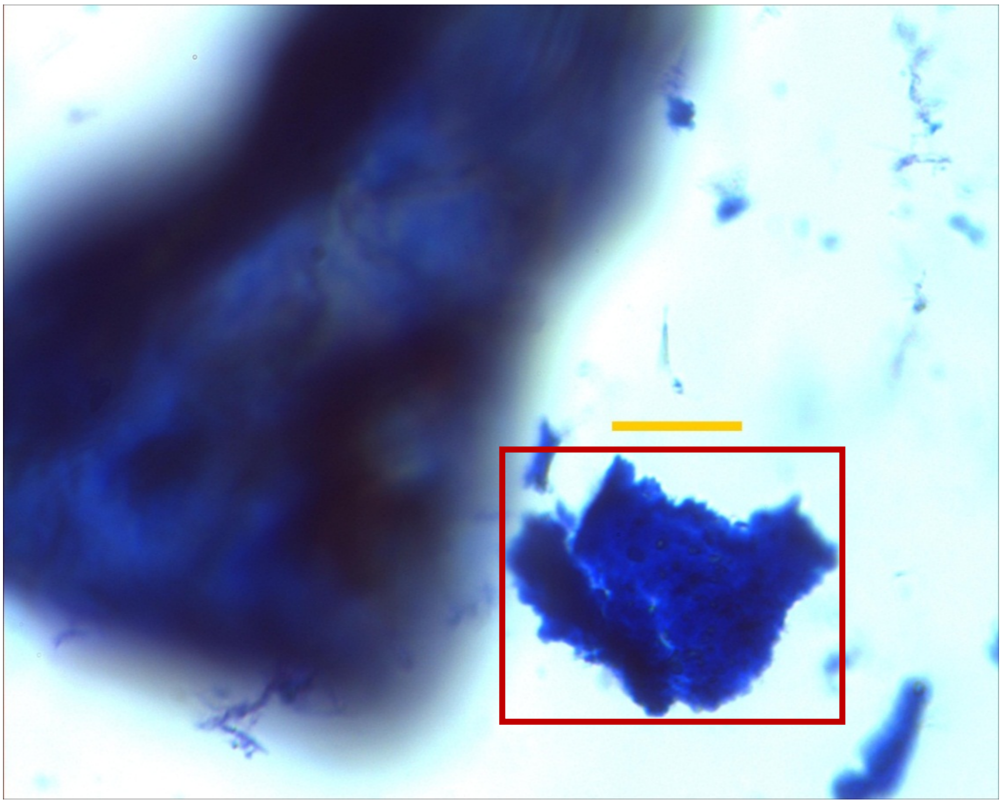

Figure 5.21: Sand layer from soil 2; Calgon -1.

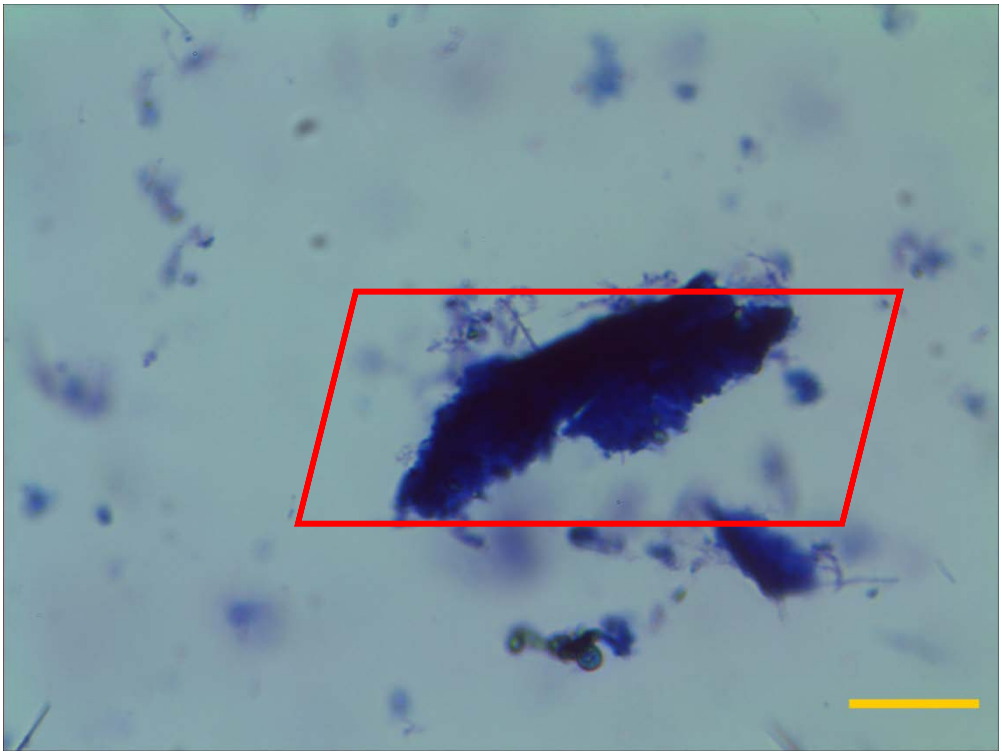

Figure 5.22: Sand layer from soil 2; Calgon -2.

In Figures 5.22 and 5.23, all particles are completely blue stained. An agglomeration of low and high CEC particles is visible in the red parallelogram.

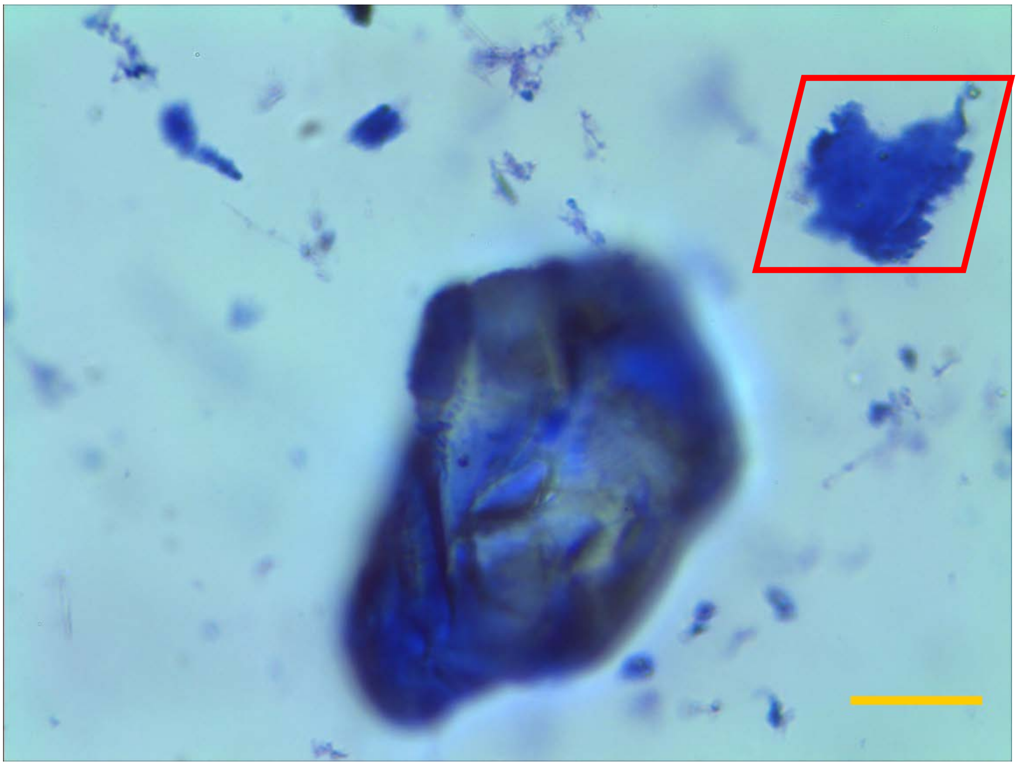

Figure 5.23: Sand layer from soil 2; Calgon -3.

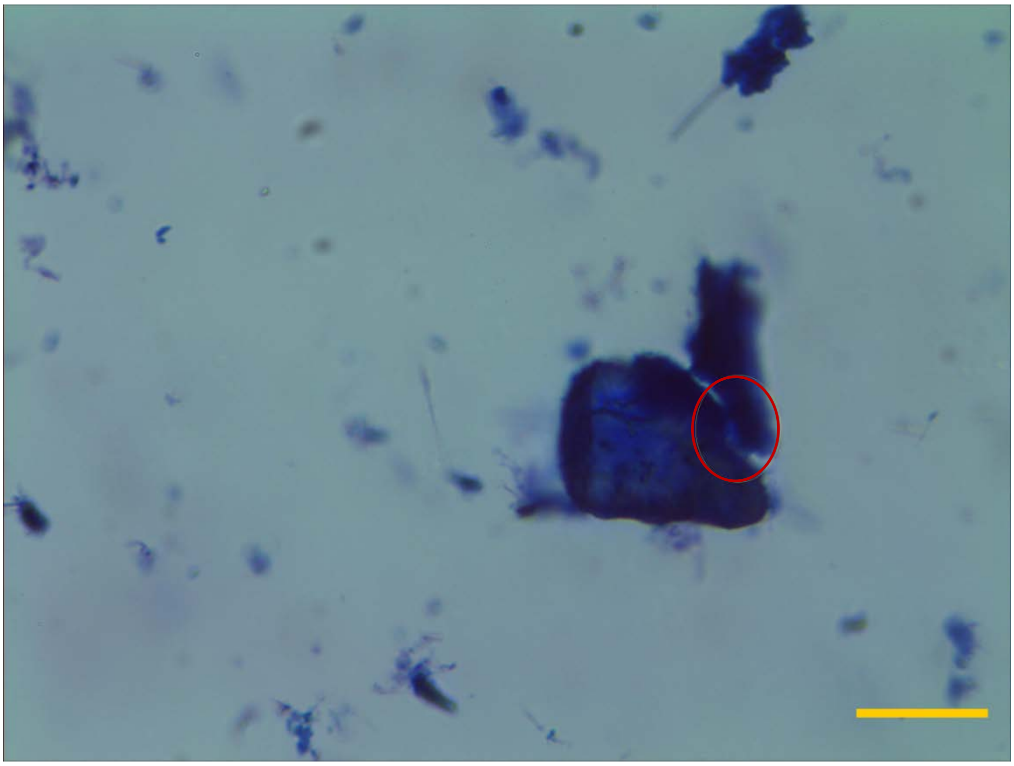

Figure 5.24: Sand layer from soil 2; Calgon -4.

The red circle in Figure 5.24 indicates a bridge of blue stained, high CEC particles joining two silt-sized particles which are completely covered with clay.

In all the photographs, there are deeply blue stained clay particles grouped into sand sized particles.

5.3.2 Sodium Silicate/Oxalate

In Figure 5.25, the big sand grain is a dark colored mineral and has small clay particles attached to it. Above the big sand grain, within the red rectangle, a group of silt and clay size particles are attached to each other and completely stained to form a sand sized particle.

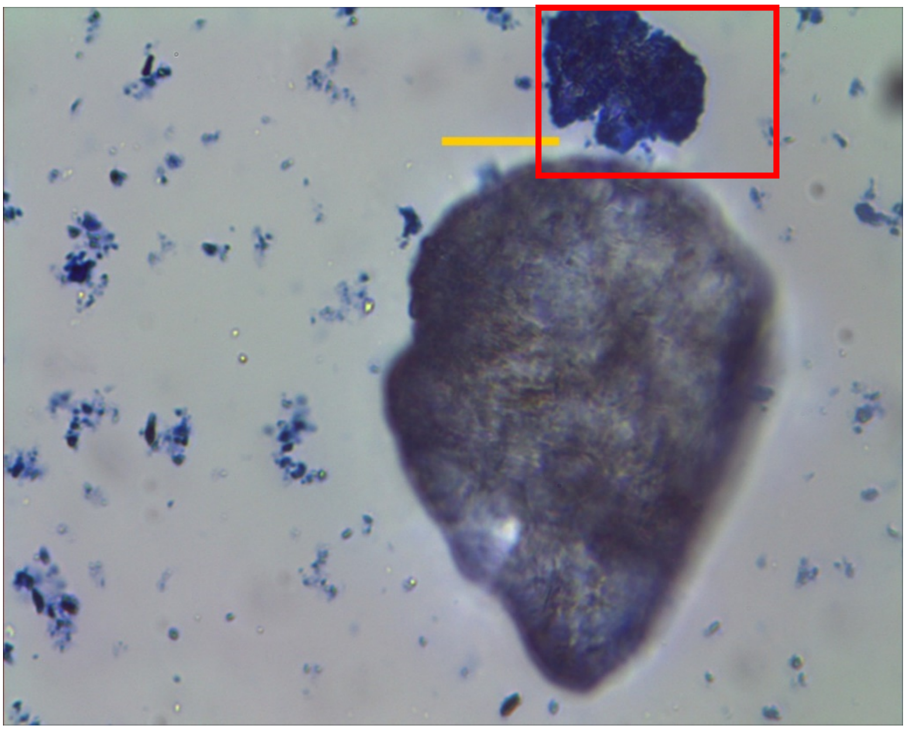

Figure 5.25: Sand layer from soil 2; Sodium Silicate/Oxalate-1.

Figure 5.26 shows a sand grain of a dark mineral completely covered with small clay particles, and silt-sized agglomerations of high CEC particles with silt cores. The silt-sized agglomerations are high CEC particles.

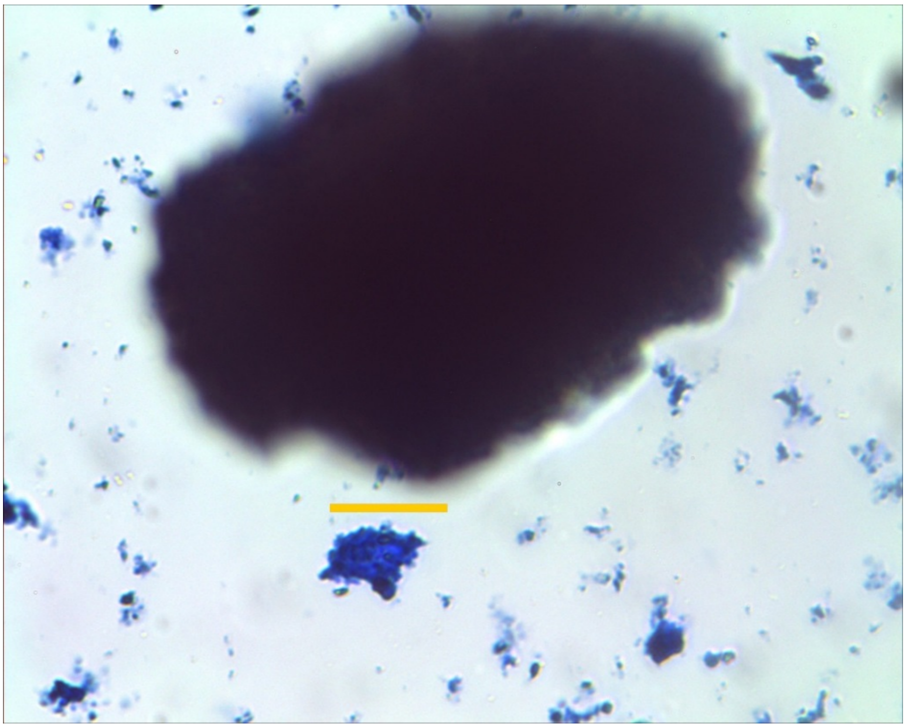

Figure 5.26: Sand layer from soil 2; Sodium Silicate/Oxalate -2.

5.3.3 TSPP

Figure 5.27 shows a grain of transparent mineral, completely covered with high CEC particles too small to be resolved with the optical microscope.

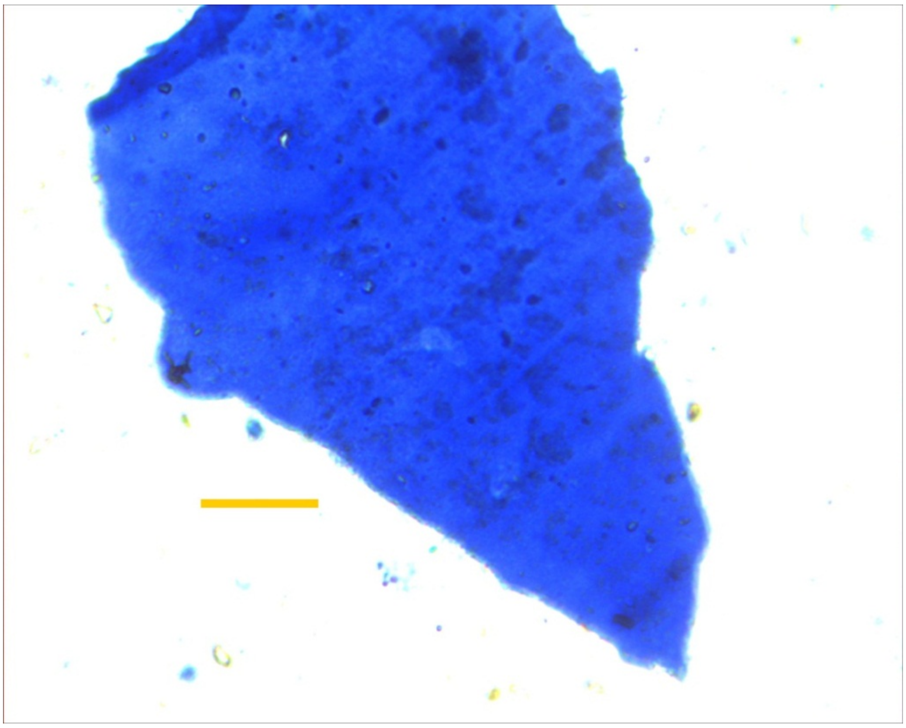

Figure 5.27: Sand layer from soil 2; TSPP -1.

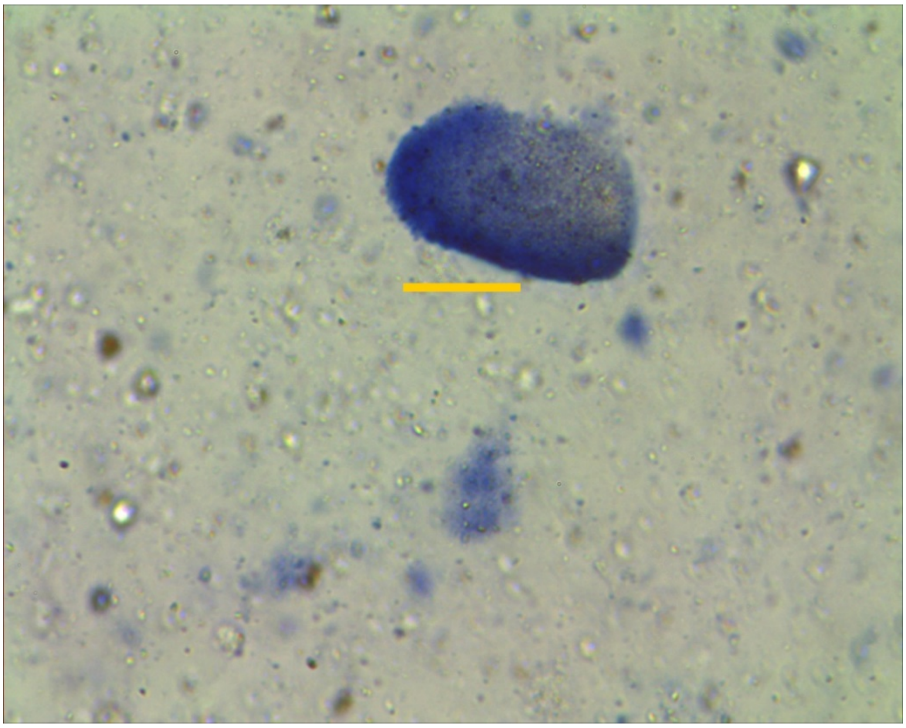

Figure 5.28: Sand layer from soil 2; TSPP -1.

Figure 5.28 shows a small sand particle with a covering of high CEC particles, several silt size agglomerations and some apparently clean, small silt particles. The sand layer appears to contain considerable material which is not actually sand.

5.4 Soil 2 - Silt Layer

5.4.1 Calgon

Figures 5.29 and 5.30 show some clean silt particles, some silt-sized particles which appear to be small agglomerations of low CEC particles, some silt particles with clay particles attached and agglomerations of silt and high CEC particles.

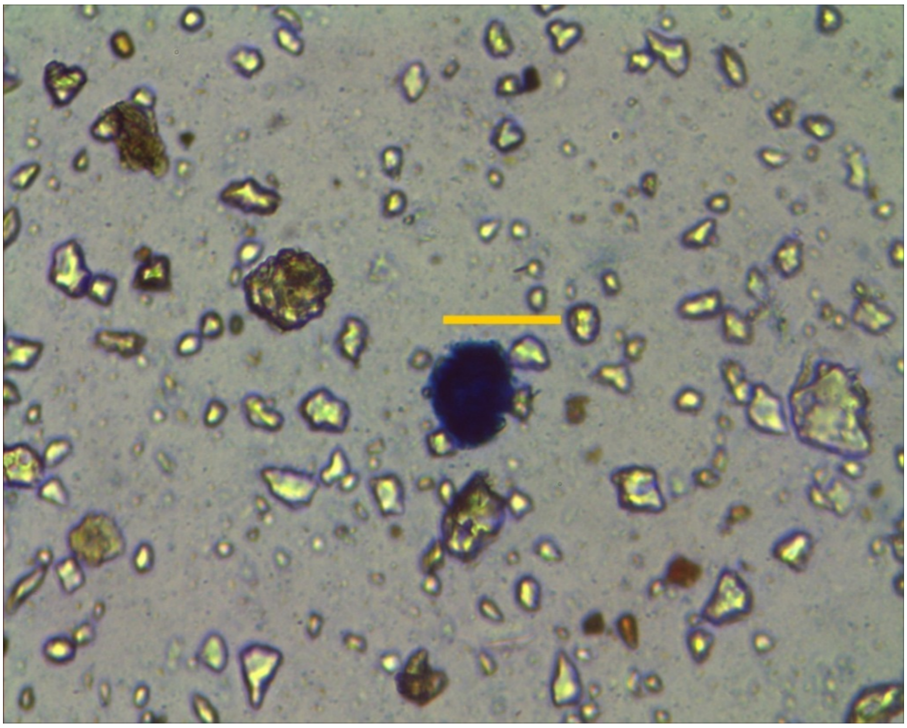

Figure 5.29: Silt layer from soil 2; Calgon -1.

Soil 2 dispersed with Calgon shows less prominent agglomeration of high CEC particles in the silt layer than in the sand layer. This was also observed for Soil 1. The silt layers have fewer agglomerations of high CEC particles and more low CEC particles, some of which are attached to silt cores.

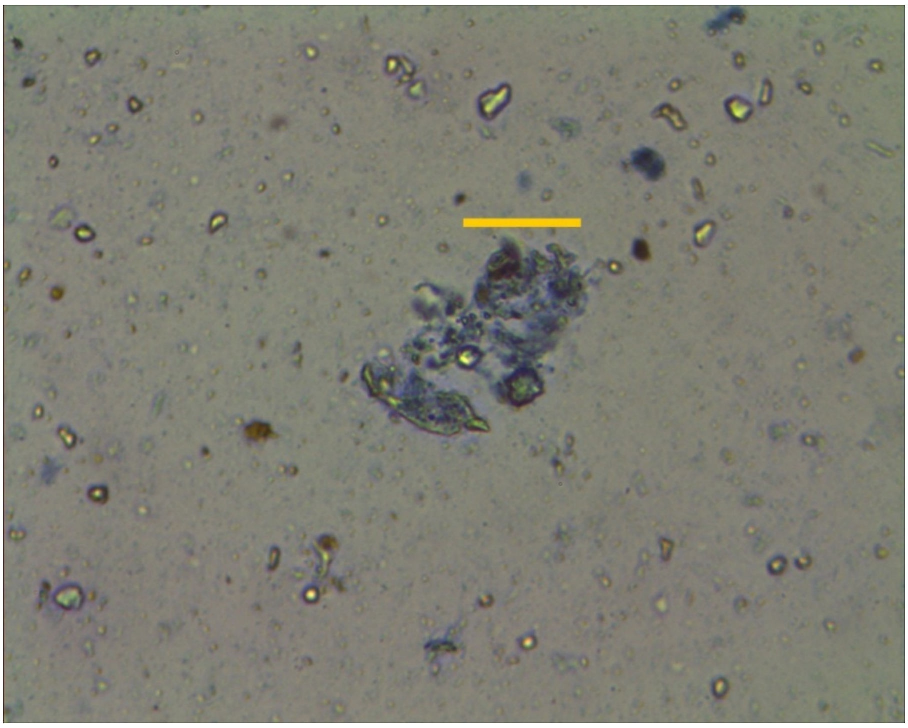

Figure 5.30: Silt layer from soil 2; Calgon -2.

5.4.2 Sodium Silicate/Oxalate

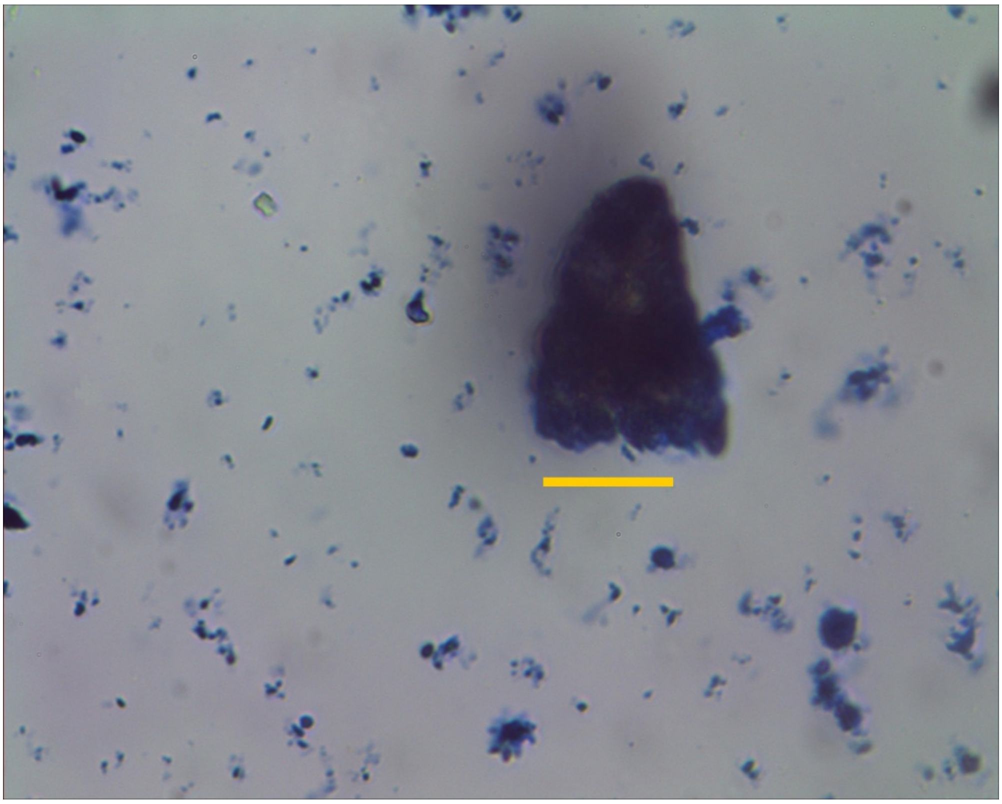

Figure 5.31: Silt layer from soil 2; Sodium Silicate/Oxalate-1.

There are small patches of agglomerations, most without silt cores. The larger particles are of a dark colored mineral with clay particles attached to them (Figure 5.31 to Figure 5.32). Only a few clean silt particles are visible in Figure 5.32. String-like agglomerations are present, approximately 30microns in size. Some structures have silt cores and some are without the silt cores. The structures are all deeply blue stained.

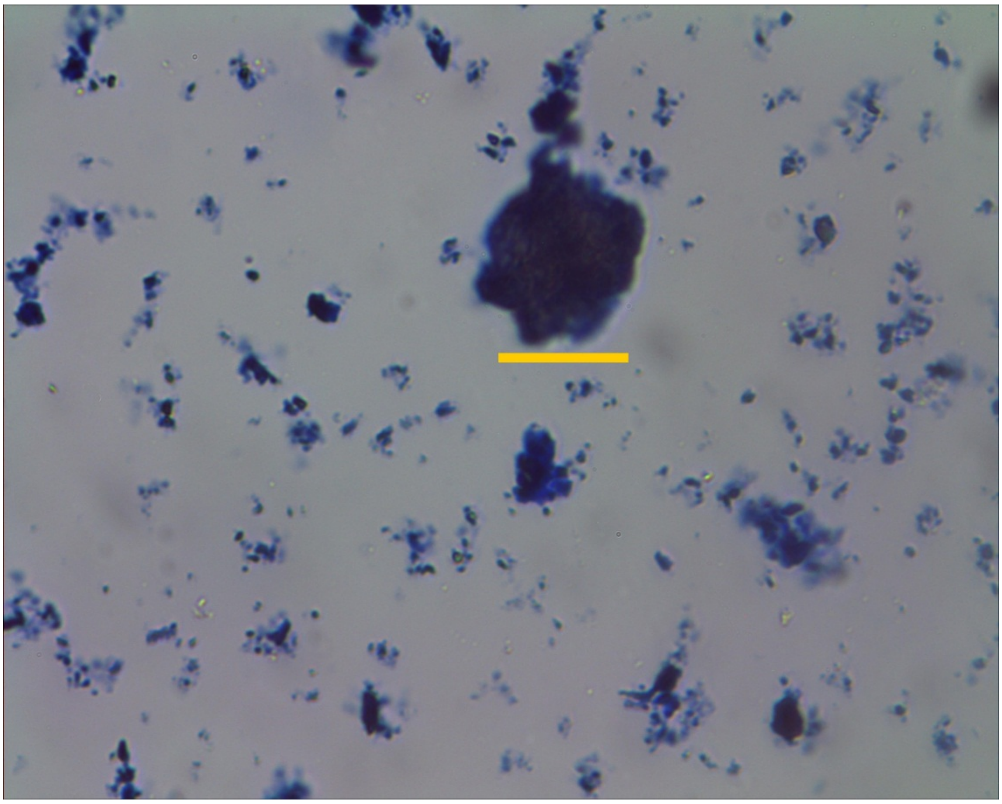

Figure 5.32: **Silt layer from soil 2; Sodium Silicate/Oxalate-2.**

5.4.3 TSPP

The dispersing of TSPP on this layer also appears to be more successful when compared to the Calgon and sodium silicate/oxalate, however, agglomeration of clay and silt particles can be seen in the photographs. The two photographs consist of high and low CEC particles agglomerations. Figure 5.33 agglomerations are of high CEC particles while 5.34 appear to be of low CEC particles. The agglomerations are larger than the clean silt particles.

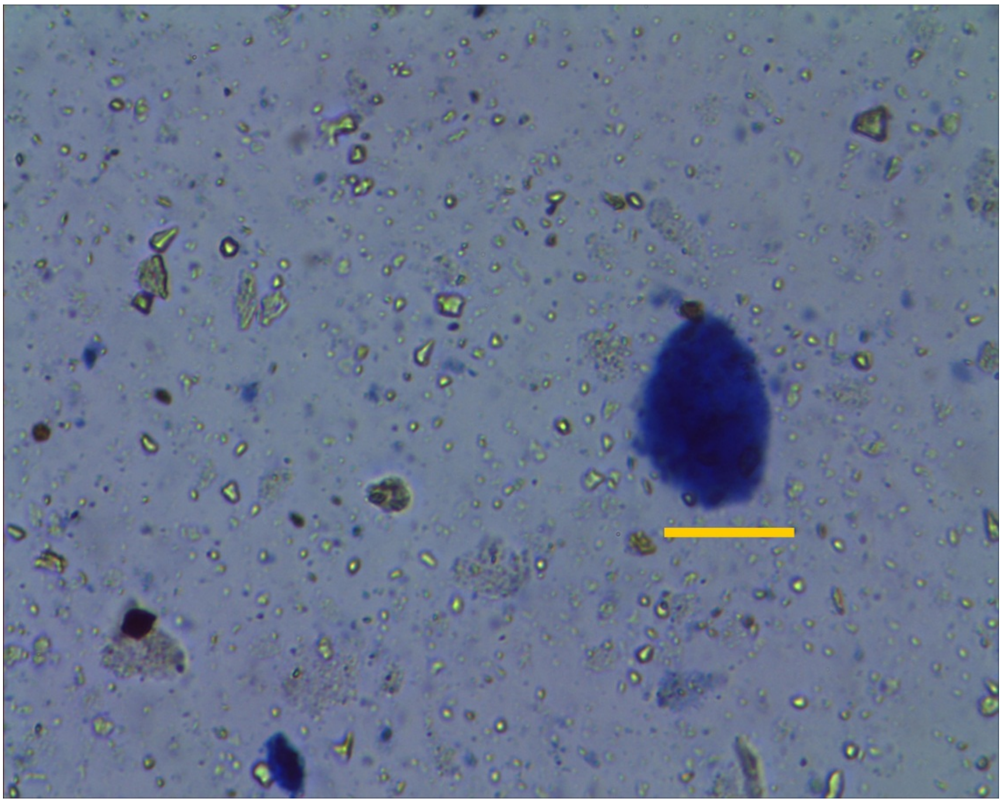

Figure 5.33: Silt layer from soil 2; TSPP -1.

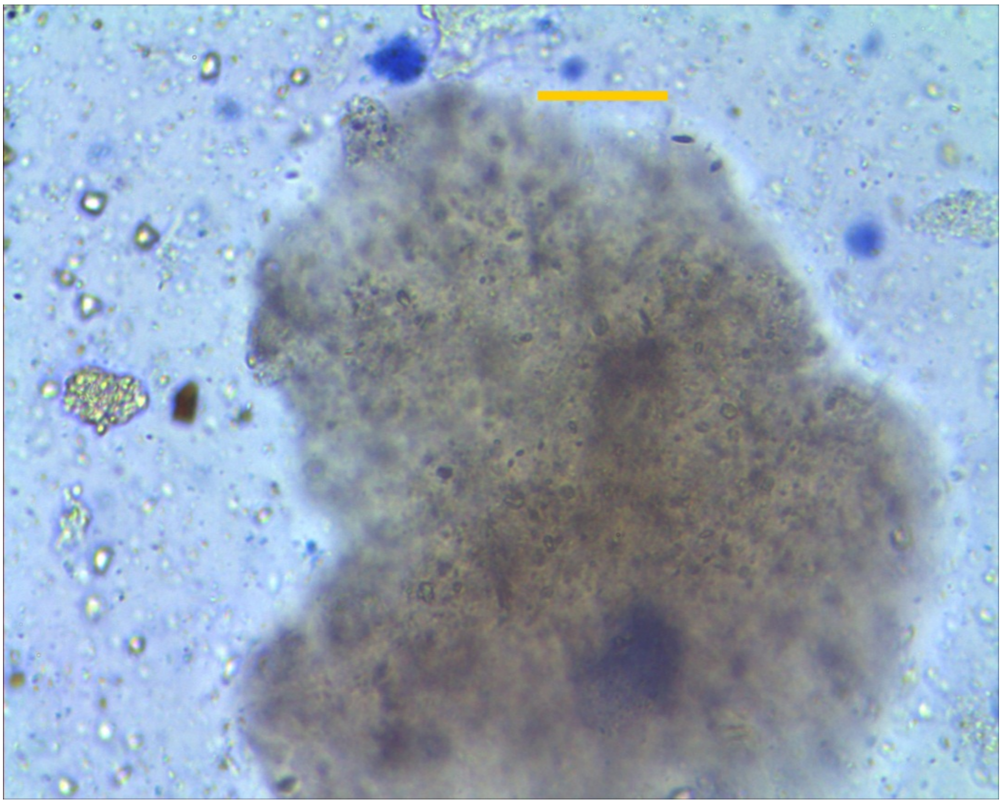

Figure 5.34: Silt layer from soil 2; TSPP -2.

5.5 Soil 3 - Coarse Sand Layer

5.5.1 Calgon

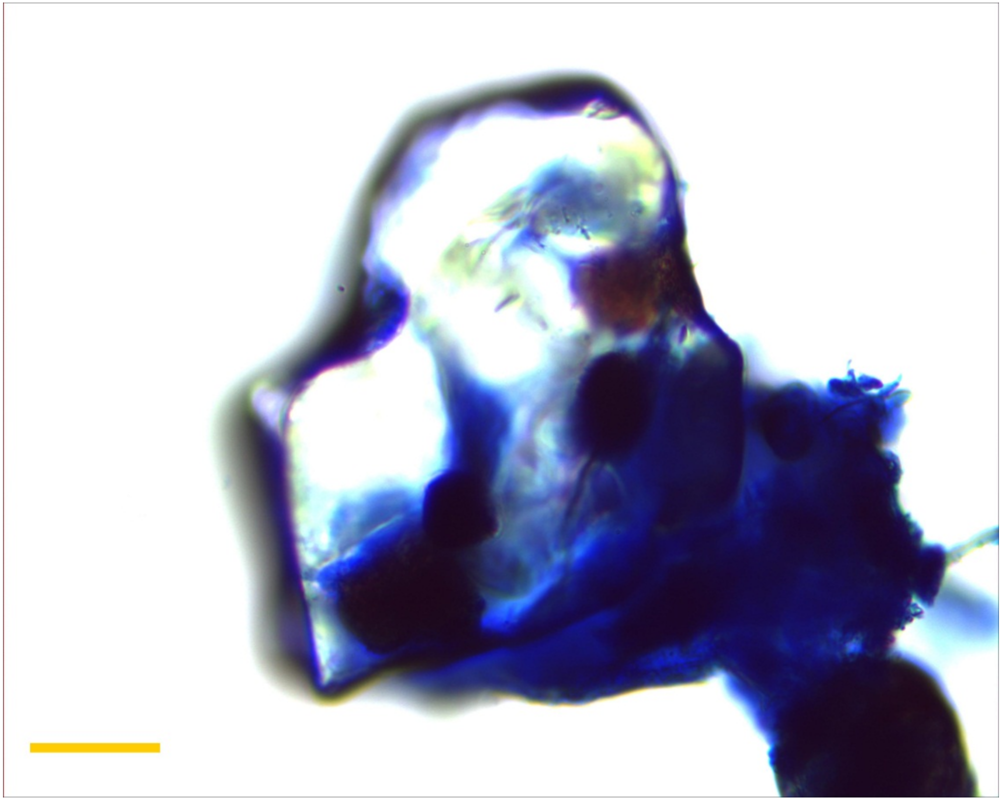

Figure 5.35: Coarse sand layer from soil 3; Calgon -1.

Soil 3 settled into very distinct coarse and fine layers. Figure 5.35 displays two sand grains joined by a solid bridge of high CEC particles. The larger sand grain has clay particles attached to it, the patches attached to the grain are deeply stained. The fuzzy blue edges are projecting away from the particle.

The agglomerations on Figures 5.36 to 5.38 are deeply blue stained and probably consist of montmorillonite. The particles are deeply stained and the two sand grains are joined by extremely small, blue stained particles. Small, deeply blue stained clay particles are apparent in all three photographs.

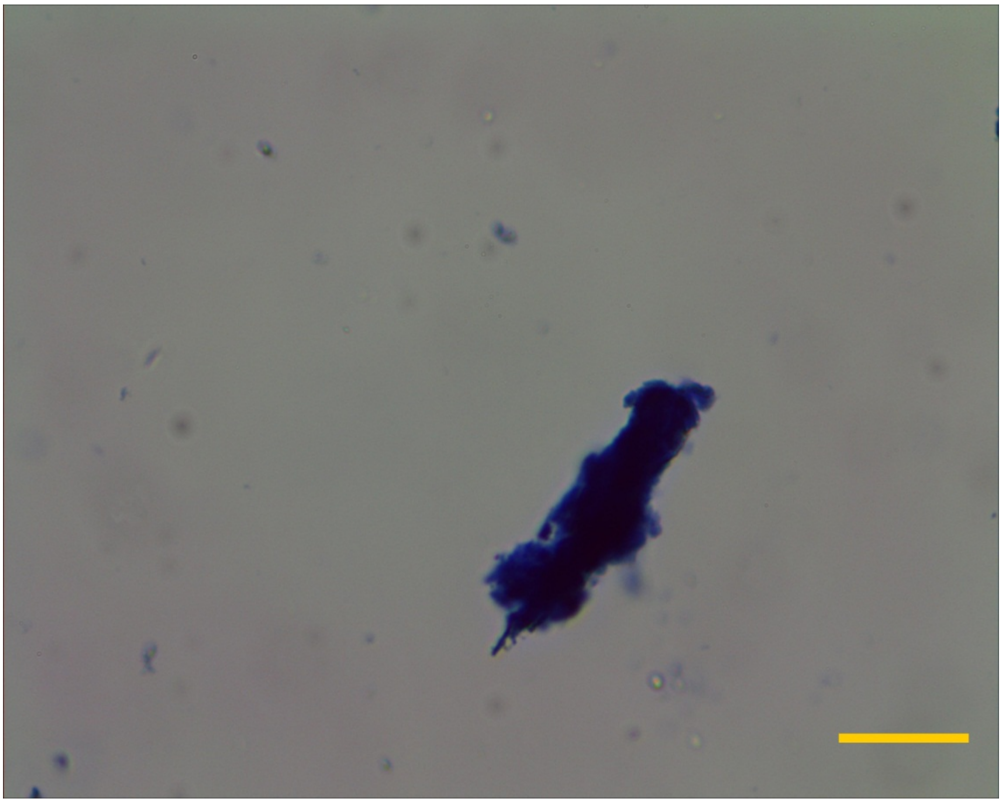

Figure 5.36: Coarse sand layer from soil 3; Calgon -2.

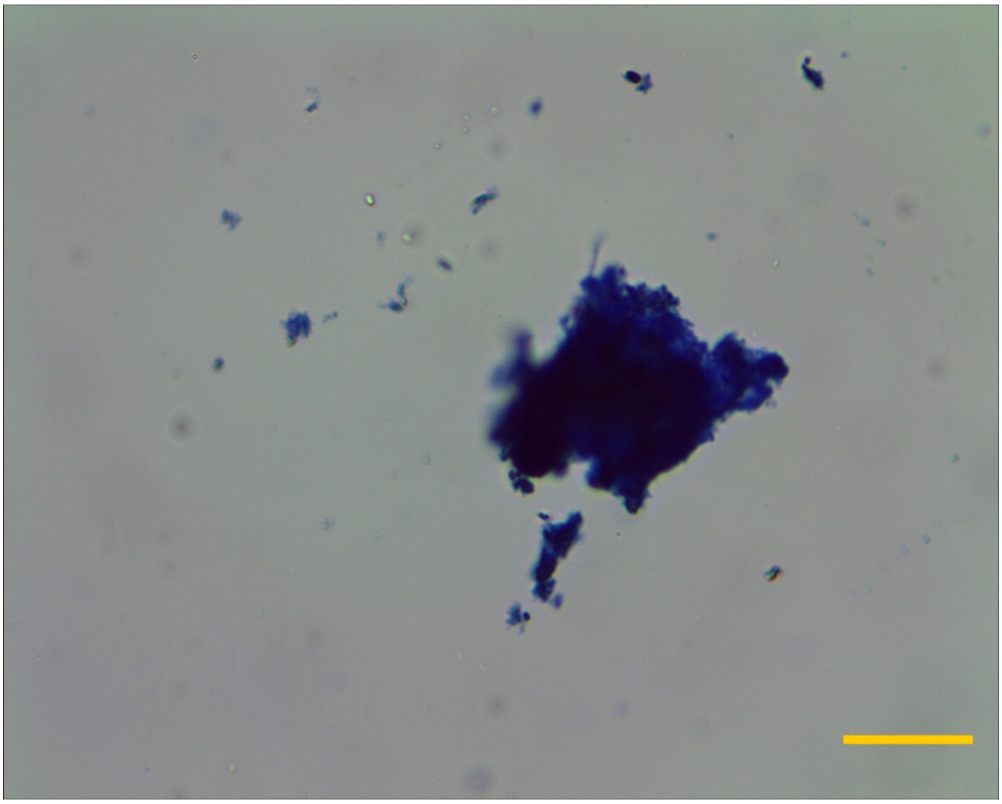

Figure 5.37: Coarse sand layer from soil 3; Calgon -3.

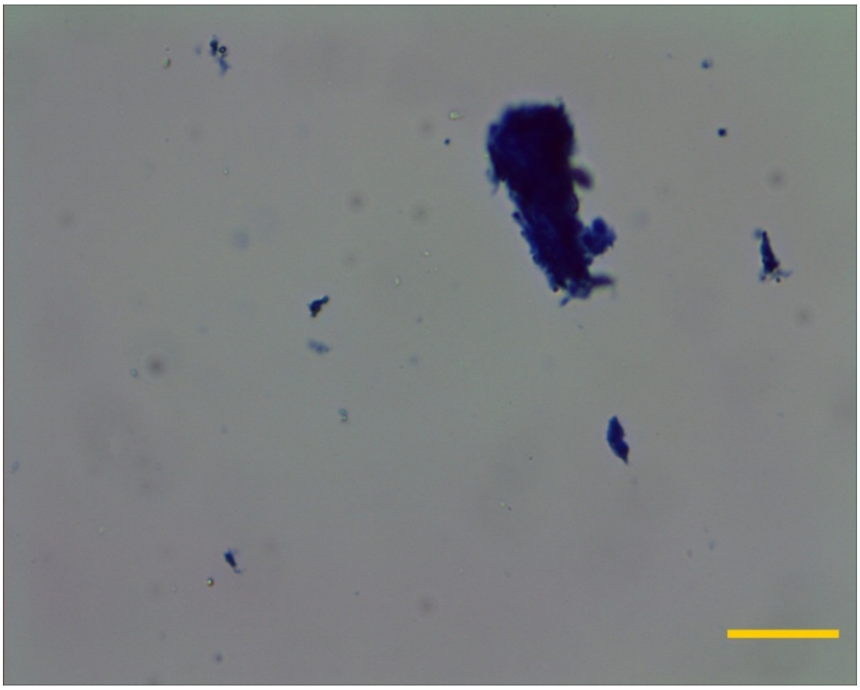

Figure 5.38: Coarse sand layer from soil 3; Calgon -3.

5.5.2 Sodium Silicate/Oxalate

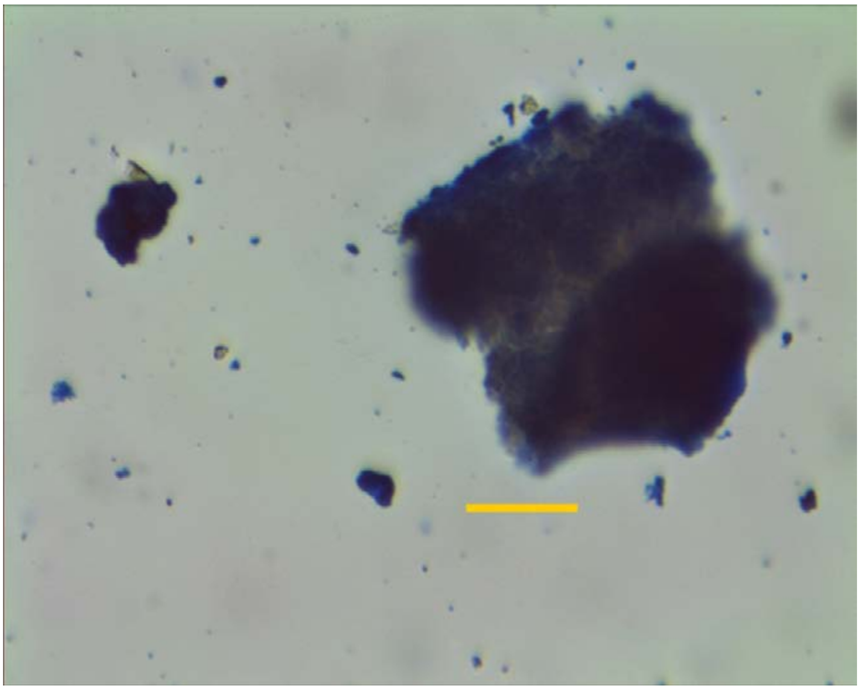

Figure 5.39: Coarse sand layer from soil 3; Sodium Silicate/Oxalate -1.

In Figure 5.39, the sand-sized particle is deeply blue stained. The sand grain appears to be of dark mineral with high CEC particles attached to it. The deep blue stain suggests that high CEC particles are present. Most of the silt-sized particles are deeply blue stained with only a few clean silt particles present in Figure 5.40.

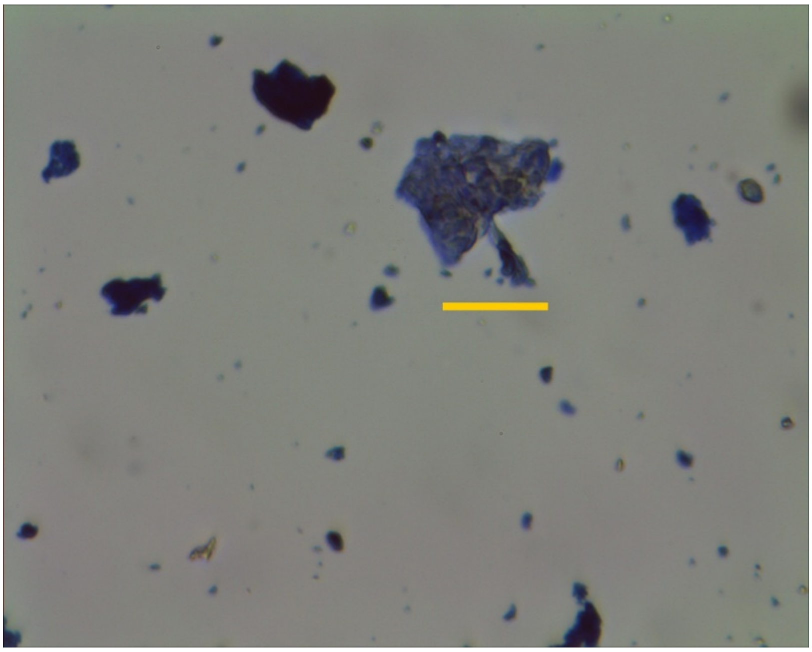

Figure 5.40: Coarse sand layer from soil 3; Sodium Silicate/Oxalate -2.

5.5.3 TSPP

Figures 5.41 and 5.42 show free low CEC particle-sized particles, the two photographs display more clean silt particles when compared to the two previous dispersants. Although the dispersant seems partially effective, agglomeration of high CEC particles is still evident. Within the red oval is the agglomeration of blue stained, silt sized particles.

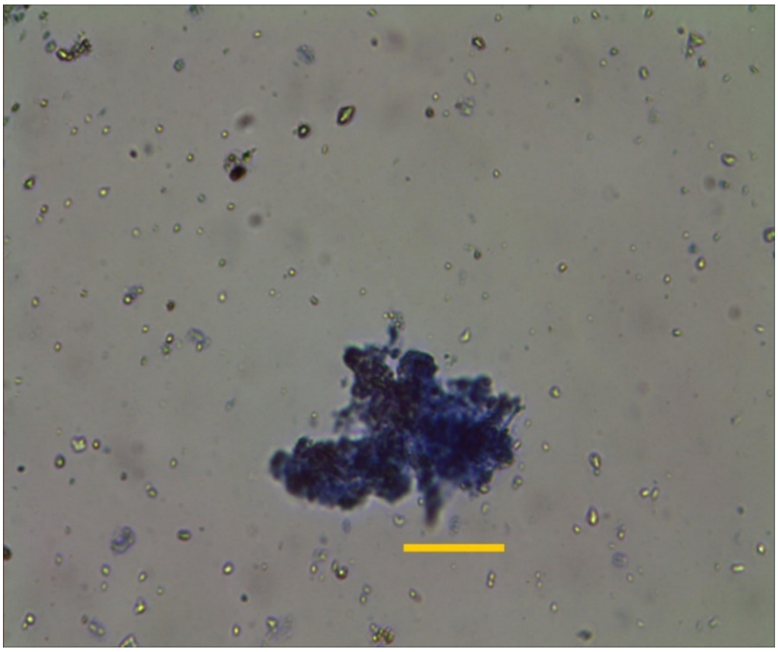

Figure 5.41: Sand layer from soil 3; TSPP. -1.

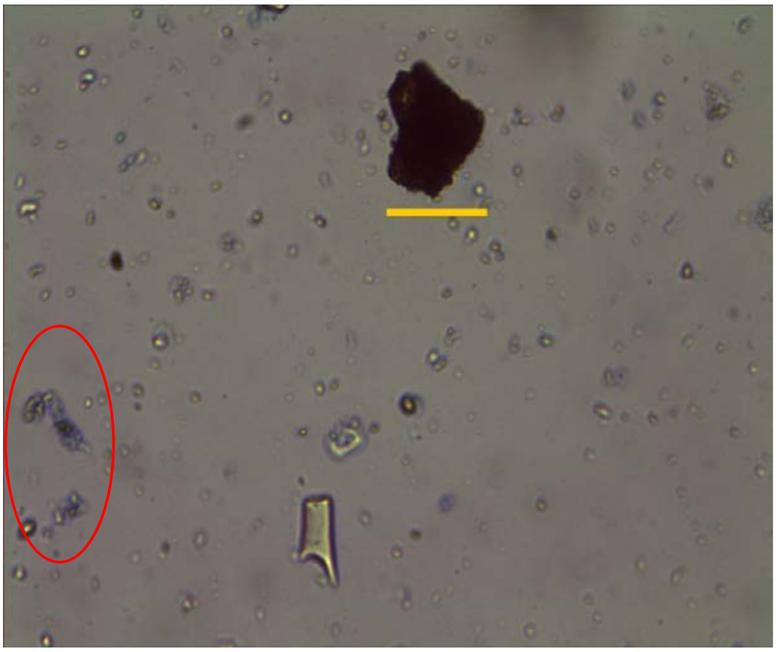

Figure 5.42: Sand layer from soil 3; TSPP. -1.

5.6 Soil 3 - Fine Sand Layer

5.6.1 Calgon

Half of the upper grain of fine sand shown in Figure 5.43 is covered by high CEC particles while the upper edge has fuzzy blue fringe projecting away from the grain. The two grains are joined by a solid bridge of clay and the lower grain is completely covered by high CEC particles.

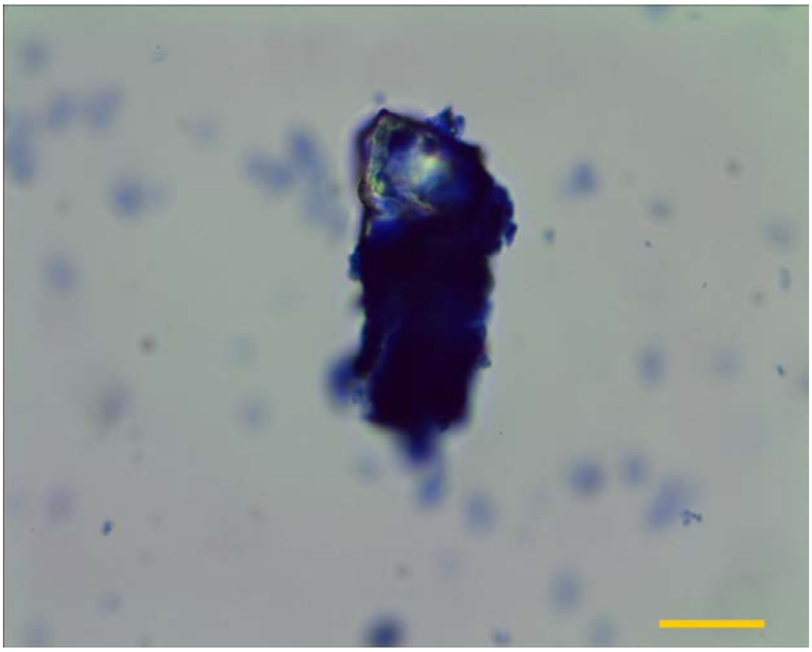

Figure 5.43: Fine sand layer from soil 3; Calgon -1.

Figure 5.44 appears to have agglomerations with and without silt cores. Within the red rectangle on Figure 5.45, two sand grains are joined together by high CEC particles. Free silt particles are not visible in all the photographs. All the particles in the Figures 5.43 to 5.46 are completely blue stained.

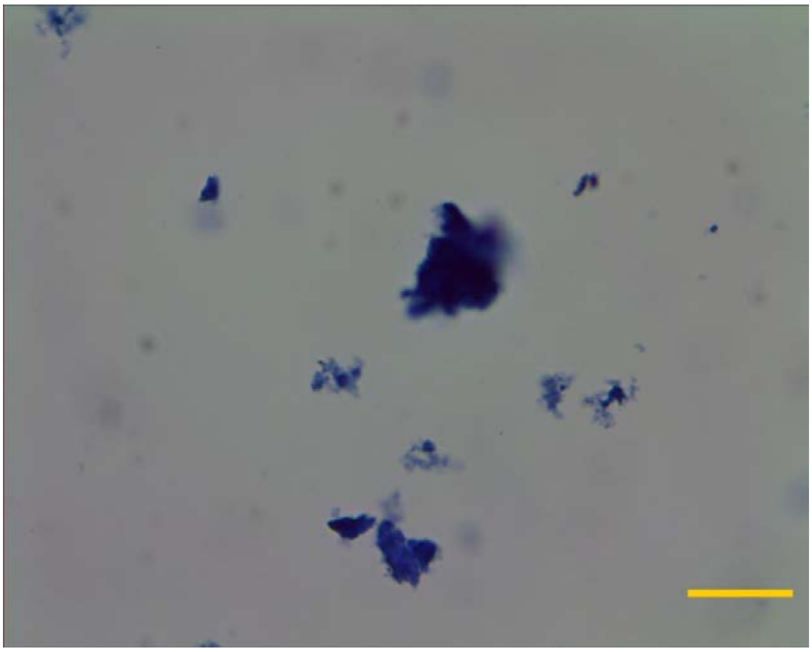

Figure 5.44: Fine sand layer from soil 3; Calgon -2.

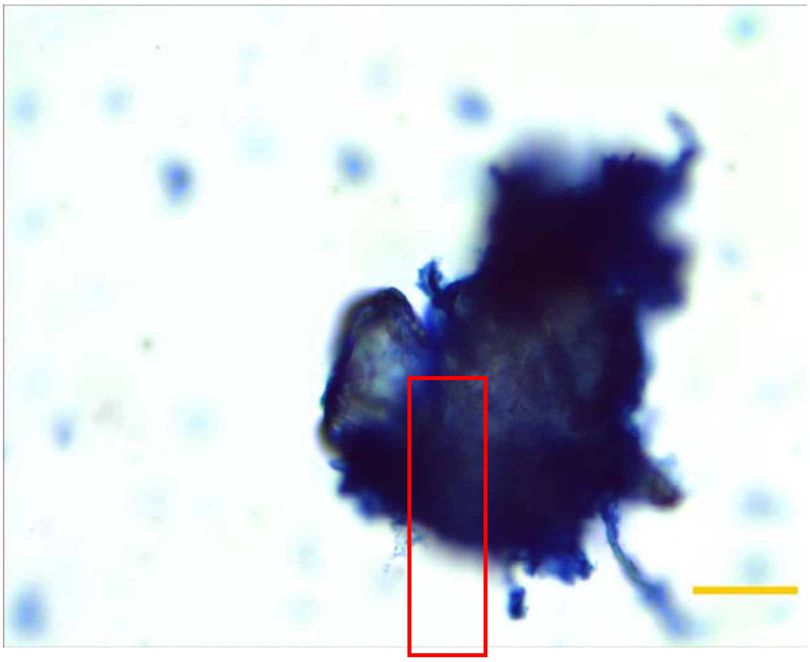

Figure 5.45: Fine sand layer from soil 3; Calgon -3.

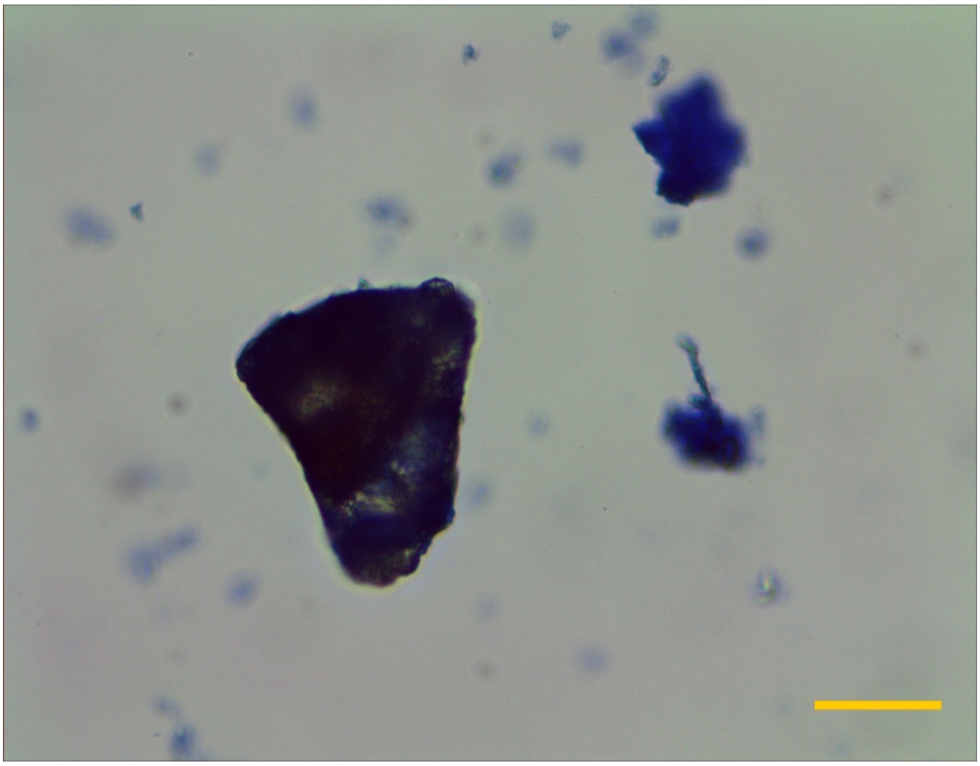

Figure 5.46: Fine sand layer from soil 3; Calgon -4.

5.6.2 Sodium Silicate/Oxalate

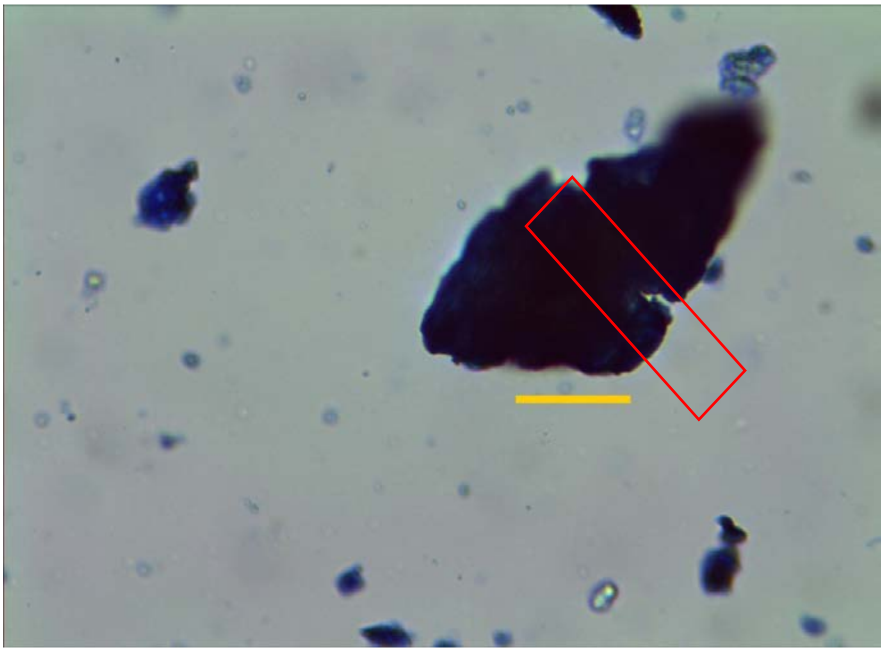

Figure 5.47: Fine sand layer from soil 3; Sodium Silicate/Oxalate -1.

Figure 5.47 shows two sand grains covered with deeply blue stained high CEC particles which are joined together by very small, high CEC particles highlighted within the parallelogram. Silt sized particles are visible and are completely covered with blue stained particles. The bubbles on Figure 5.48 are due to defective cleaning of the cover slip. Figure 5.48 shows low CEC particles attached to each other within the red ovals it also shows high CEC particles attached to sand grains.

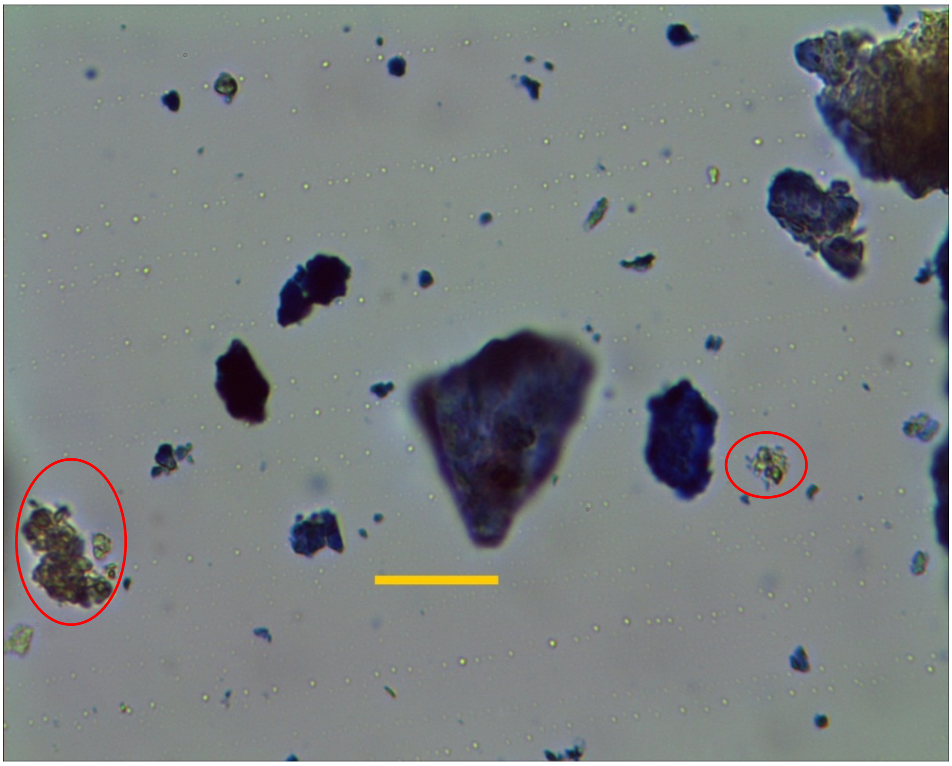

Figure 5.48: Fine sand layer from soil 3; Sodium Silicate/Oxalate -2.

5.7 Soil 3 - Coarse Silt

5.7.1 Calgon

Figure 5.49 shows two coarse silt particles of dark coloured minerals with high CEC particles attached to them. The three silt particles in the bottom part of the photograph are joined together by clay particles. Figures 5.49 to 5.52 shows string like structures and agglomerations with silt cores. High and low CEC particles are attached to larger grains.

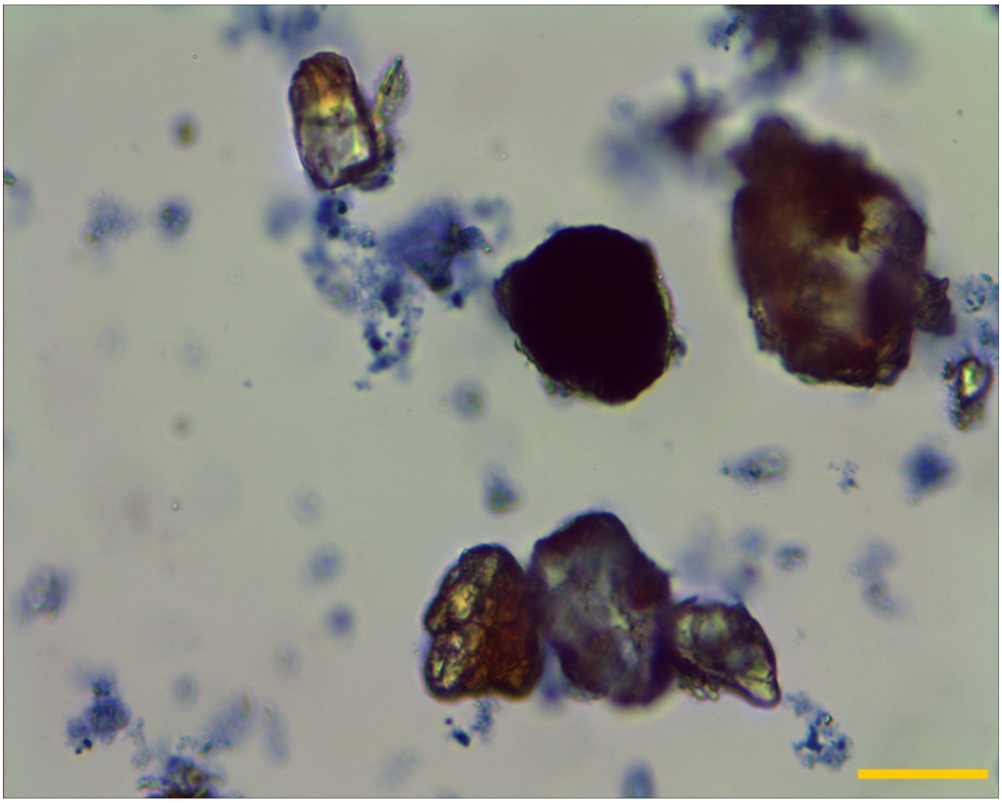

Figure 5.49: Coarse silt layer from soil 3; Calgon -1.

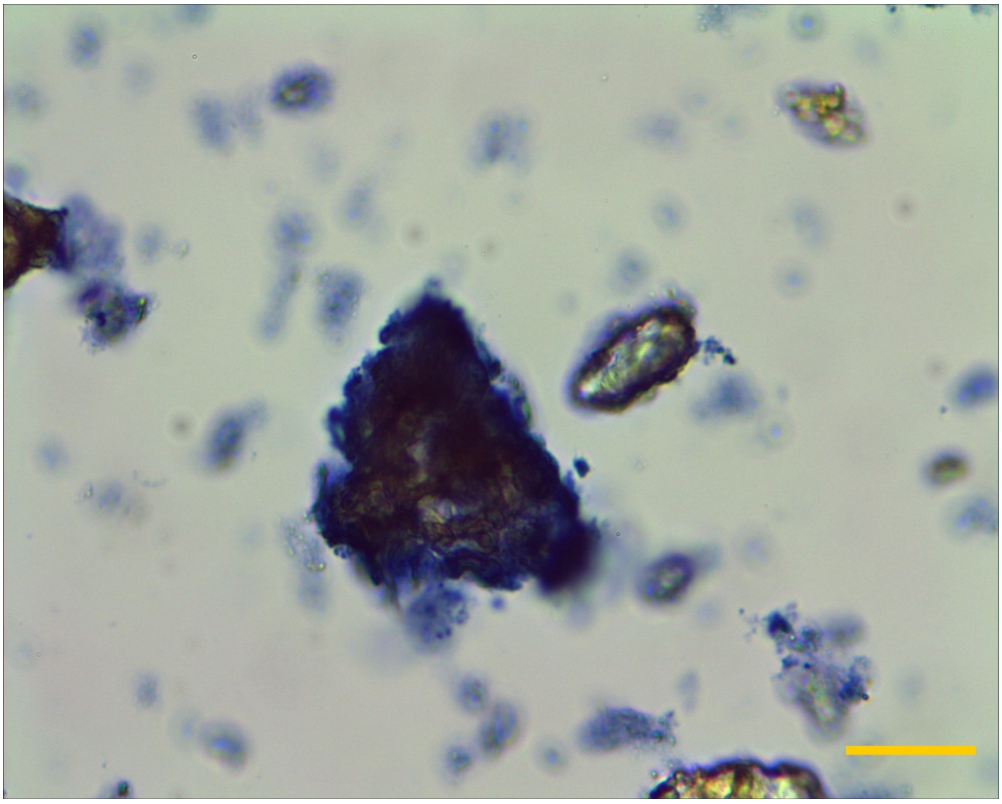

Figure 5.50: Coarse silt layer from soil 3; Calgon -2.

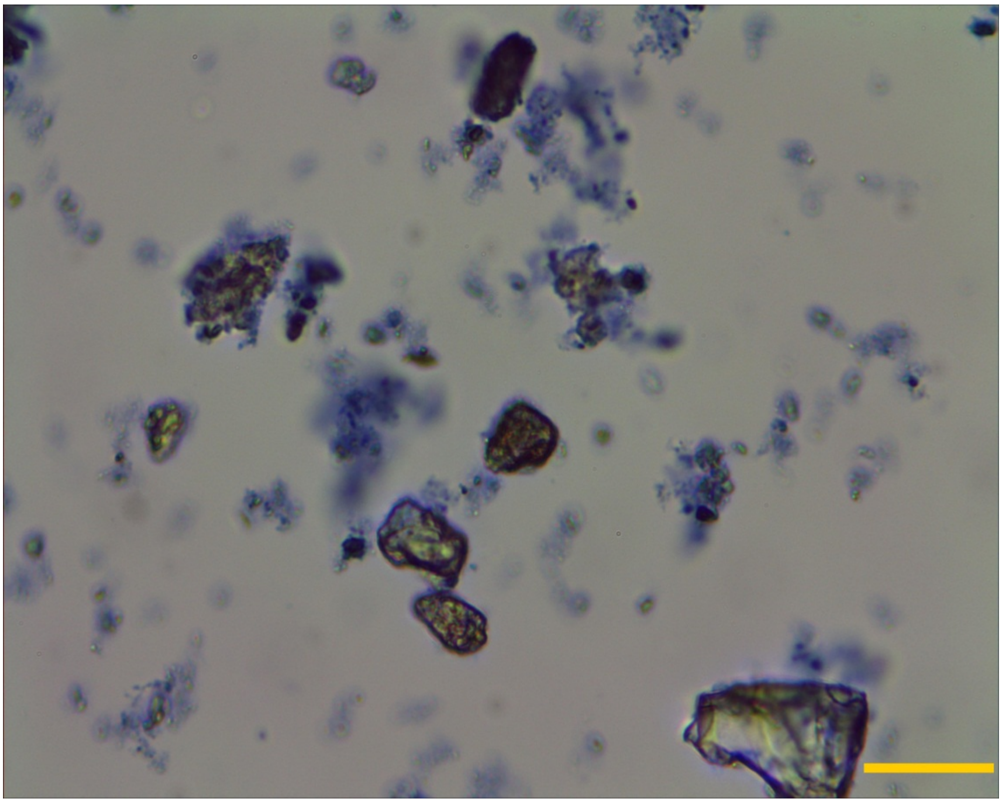

Figure 5.51: Coarse silt layer from soil 3; Calgon -3.

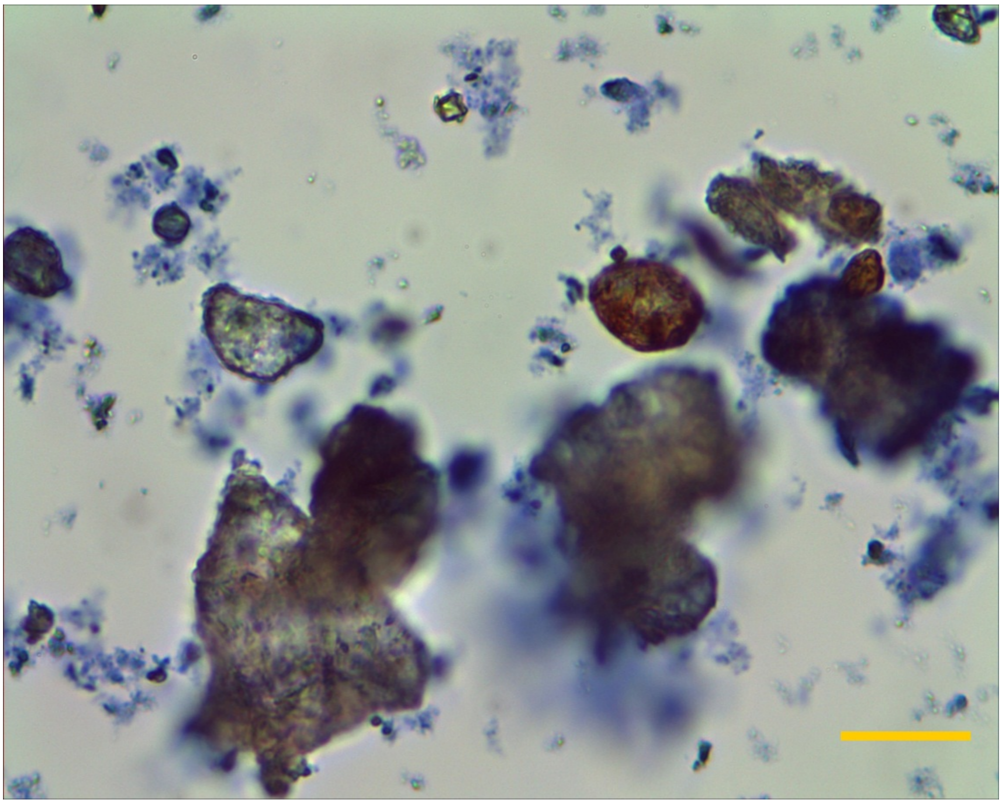

Figure 5.52: Coarse silt layer from soil 3; Calgon -4.

String-like structures are becoming apparent (after addition of MB) in the silt layer and consist of high CEC particles.

5.7.2 Sodium Silicate/Oxalate

Free silt and clay sized particles are visible in Figures 5.53 and 5.54, most particles appear to be deeply blue stained agglomerations. Figure 5.53 within the rectangle could be a dark mineral with high CEC particles attached to it.

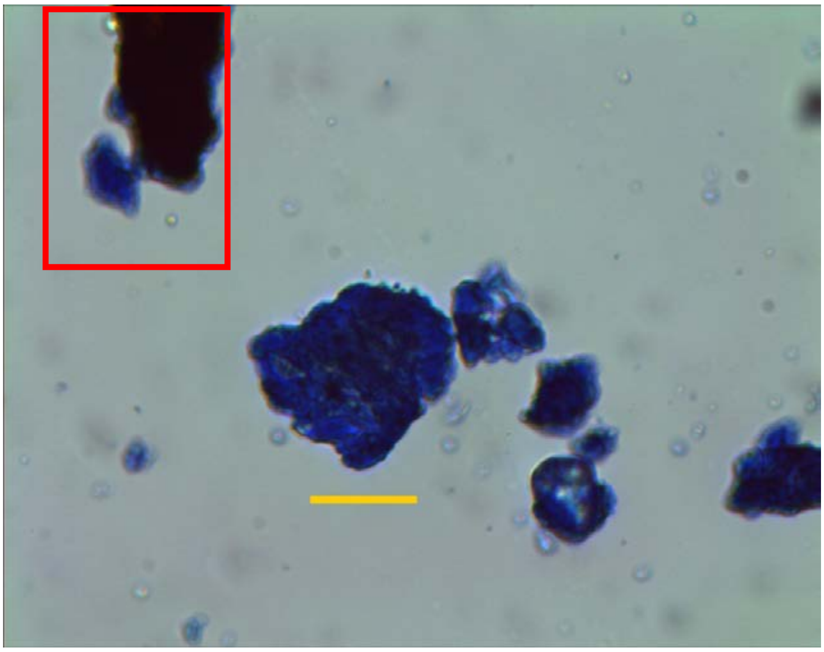

Figure 5.53: Coarse silt layer from soil 3; Sodium Silicate/Oxalate -1.

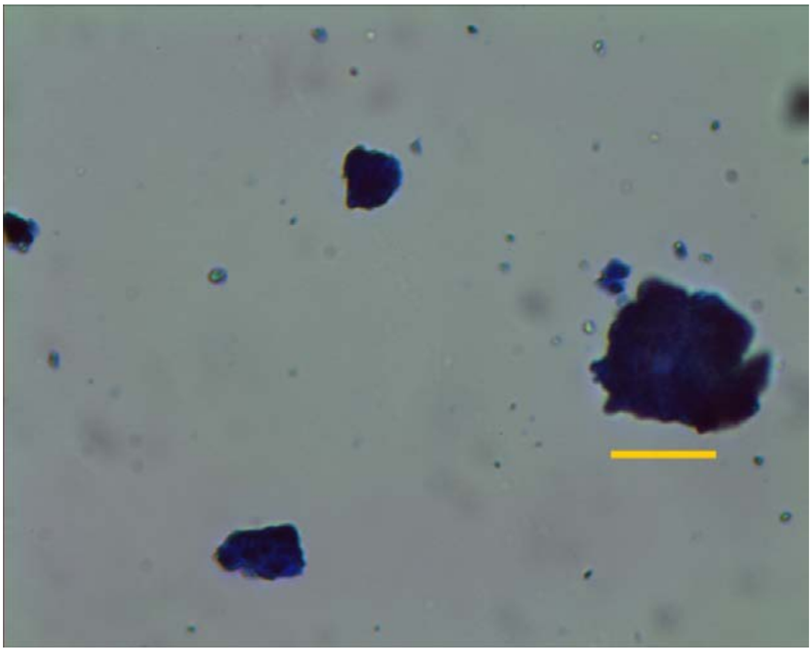

Figure 5.54: Coarse silt layer from soil 3; Sodium Silicate/Oxalate -2.

5.7.3 TSPP

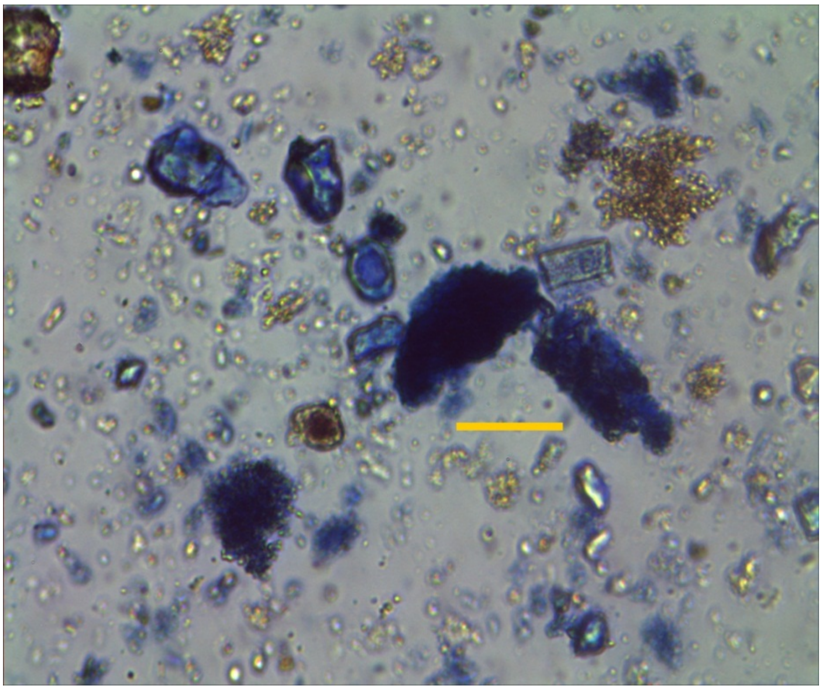

Figure 5.55: Silt layer from soil 3; TSPP -1.

Throughout all the soil types and different dispersant TSPP seems to achieve consistent result. Agglomerations of high CEC particles and low CEC are seen on both Figures 5.55 and 5.56, again these agglomerations are bigger than the genuine silt particles.

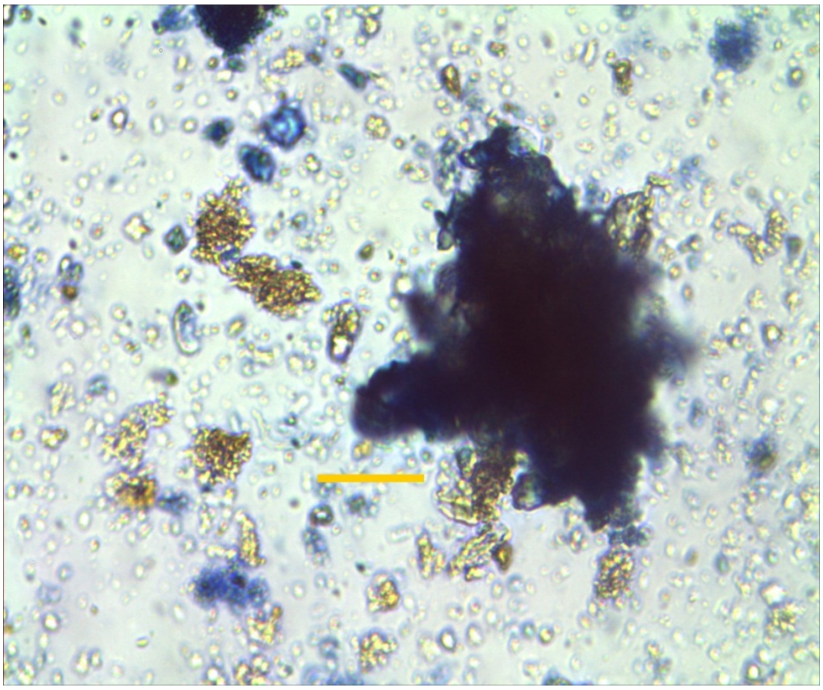

Figure 5.56: Silt layer from soil 3; TSPP -1.

5.8 Soil 3 Fine Silt

5.8.1 Calgon

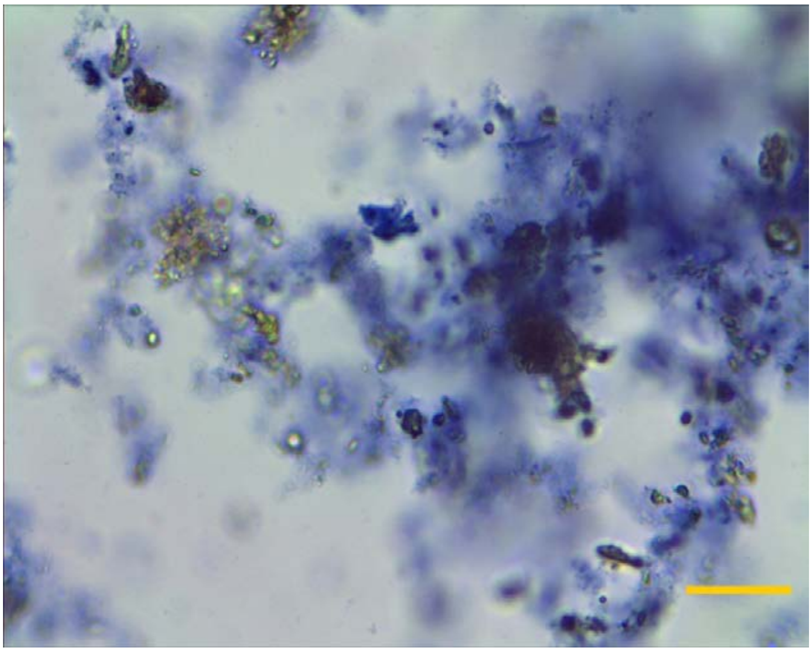

Figure 5.57: Fine silt layer from soil 3; Calgon -1.

Figures 5.57 and 5.58 consist of low and high CEC particles, the string like structures in the fine silt layer are more obvious than in the coarse silt layer. There appears to be more clay than silt.

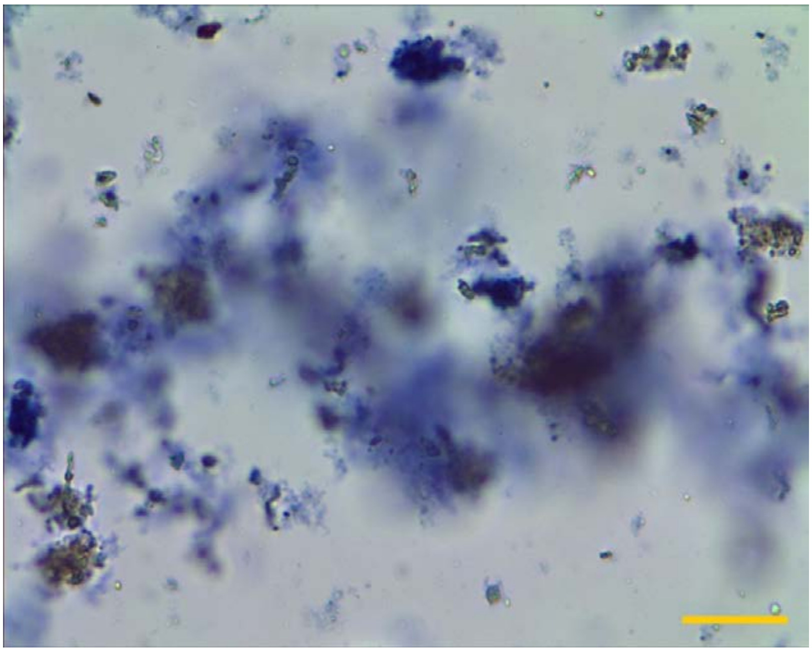

Figure 5.58: Fine silt layer from soil 3; Calgon -2.

5.8.2 Sodium Silicate/Oxalate

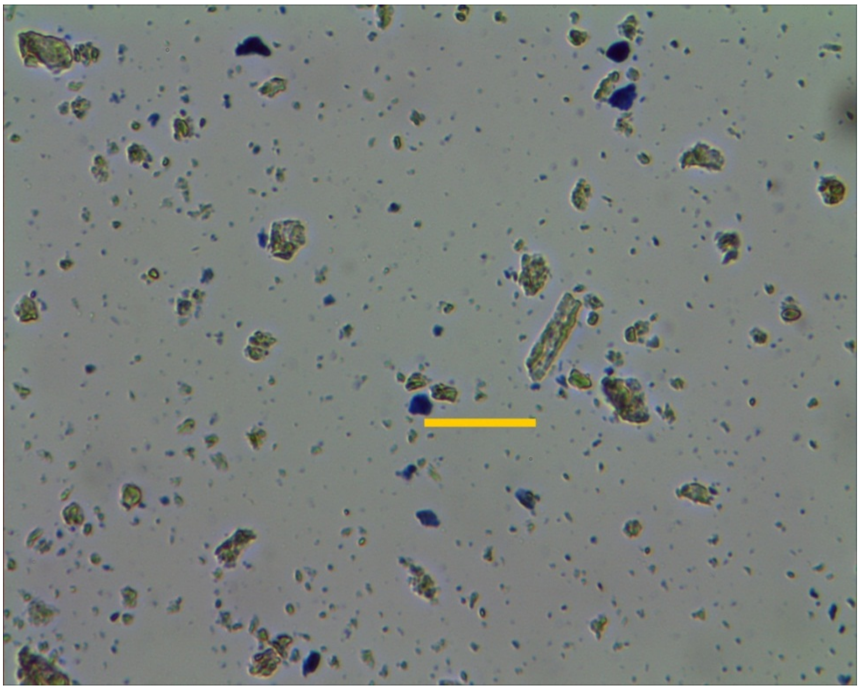

Figure 5.59: Fine silt layer from soil 3; Sodium Silicate/Oxalate -1.

The fine silt layer appears to have less high CEC particles compared to the sand and coarse silt layers. Agglomerations of low CEC particles appear to be bigger in size than those of the deeply blue stained particles (Figures 5.59 and 5.60). There again appears to be more clay agglomeration than silt.

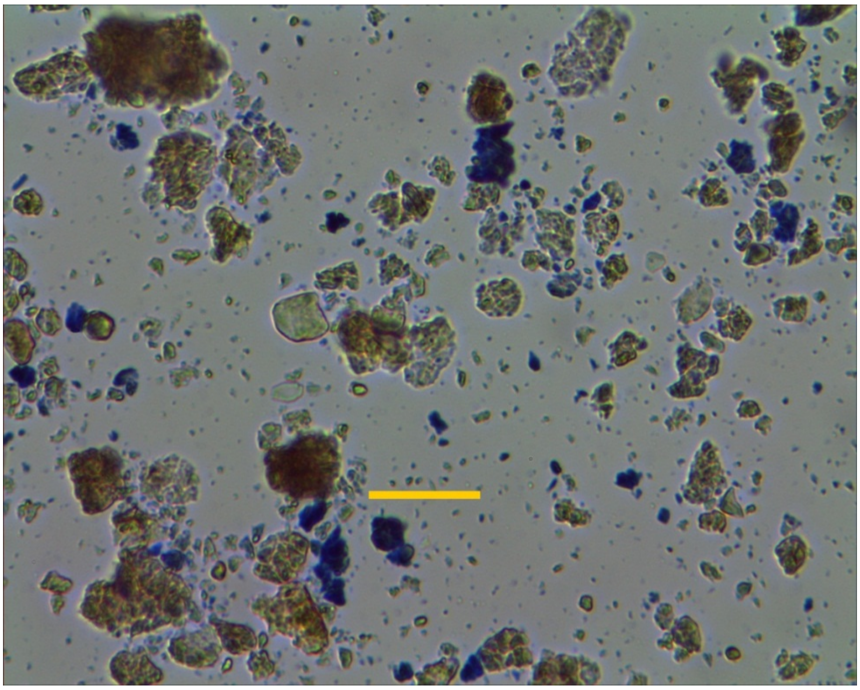

Figure 5.60: Fine silt layer from soil 3; Sodium Silicate/Oxalate -2.

5.9 Soil 4 - Coarse Sand

5.9.1 Calgon

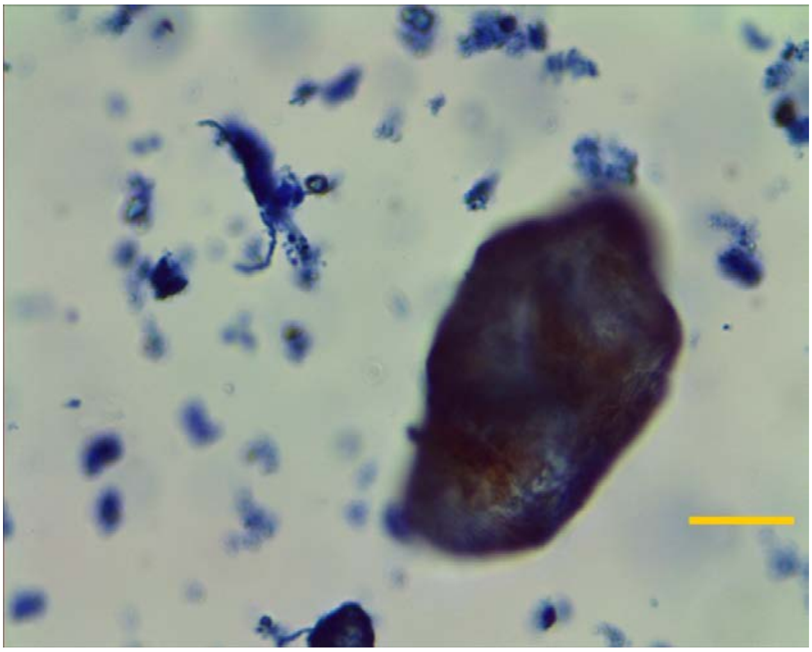

Figure 5.61: Coarse sand layer from soil 4; Calgon -1.

The sand grains in the Figures 5.61 and 5.62 are blue stained. Deeply blue stained silt and clay sized particles are present in both photographs. Small string-like structures are visible in both photographs.

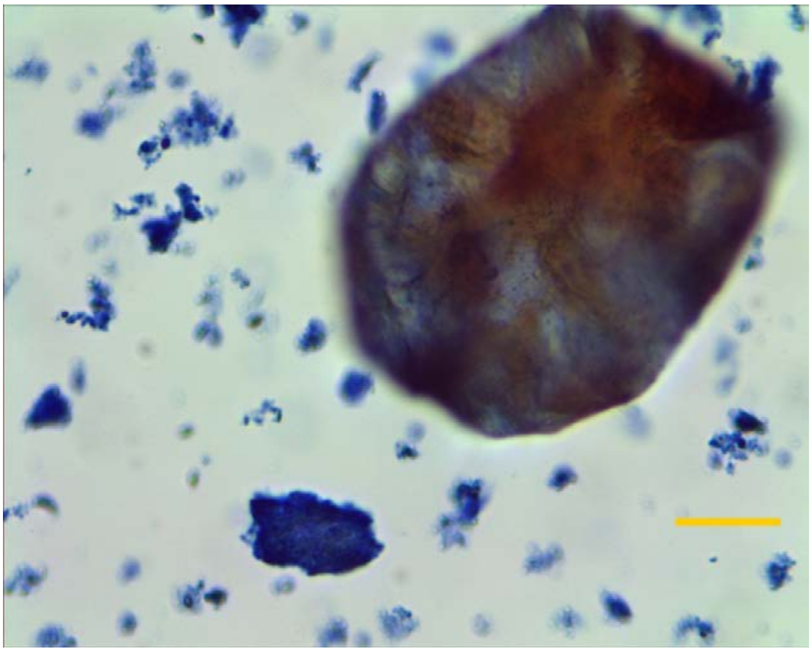

Figure 5.62: Coarse sand layer from soil 4; Calgon -2.

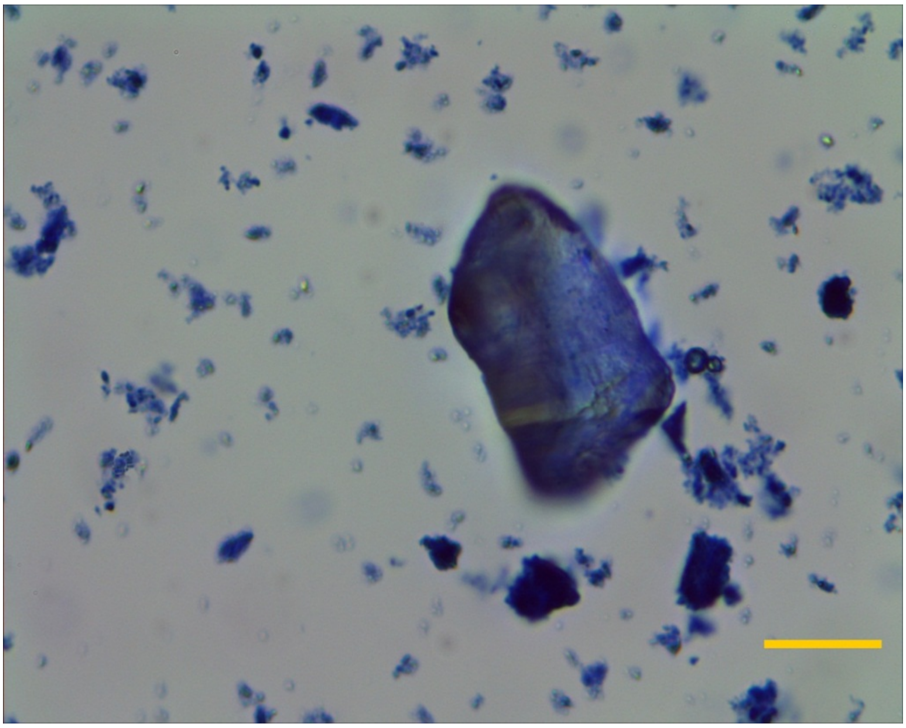

Figure 5.63: Coarse sand layer from soil 4; Calgon -3.

Figures 5.63 and 5.64 have the same pattern as the above photographs. String-like structures are visible in all the photographs and the clay content is significant.

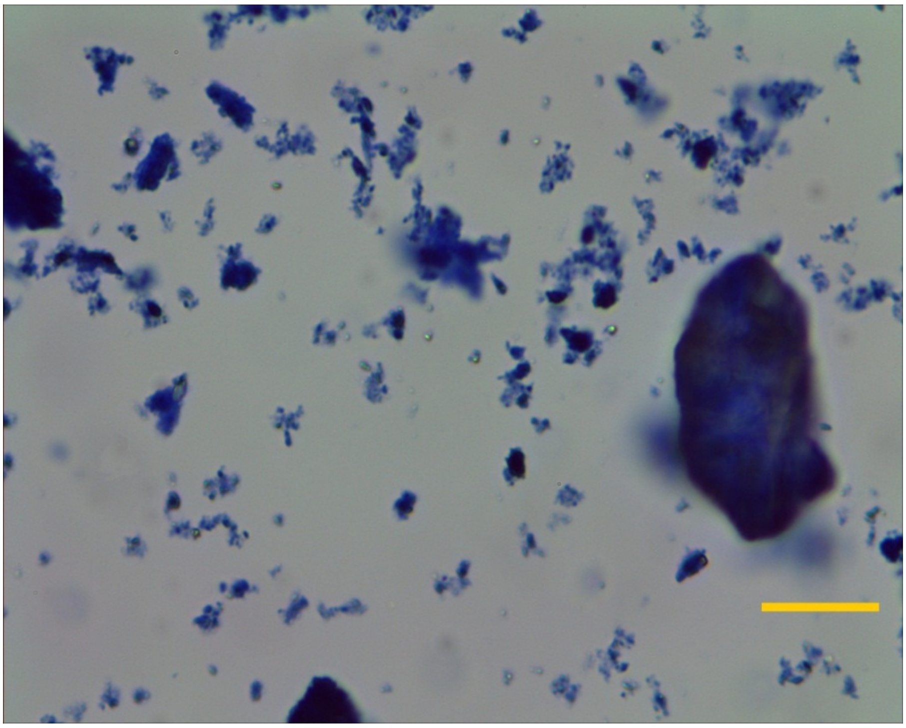

Figure 5.64: Coarse sand layer from soil 4; Calgon -4.

5.9.2 Sodium Silicate/Oxalate

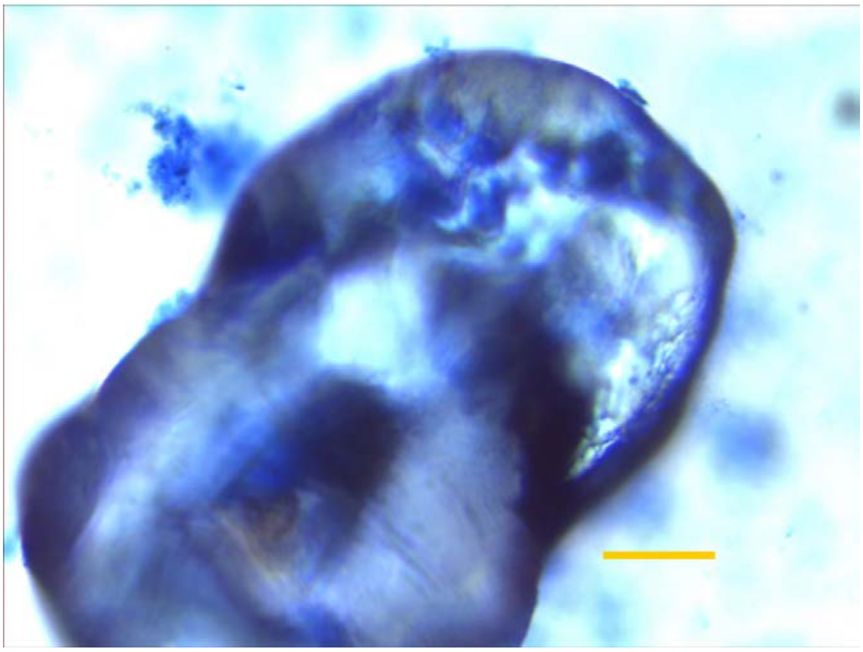

Figure 5.65: Coarse sand layer from soil 4; Sodium Silicate/Oxalate -1.

The photographs are not of the same magnification, in Figure 5.65 the scale bar is 30µm x 2µm as in most of the photographs. Figure 5.66 was taken with the 10x objective and the scale bar is 90µm x 6µm. This photograph shows how widespread the clay agglomerations are. The sand grains have clay agglomerations with no silt cores attached to them.

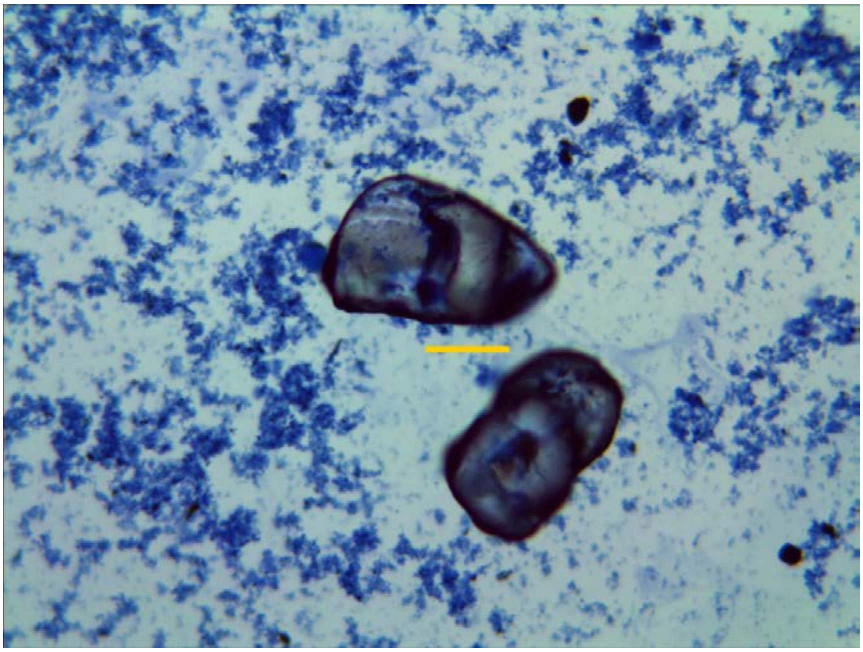

Figure 5.66: Coarse sand layer from soil 4; Sodium Silicate/Oxalate -2. 10x objective and 90x6µm scale rectangle.

5.9.3 TSPP

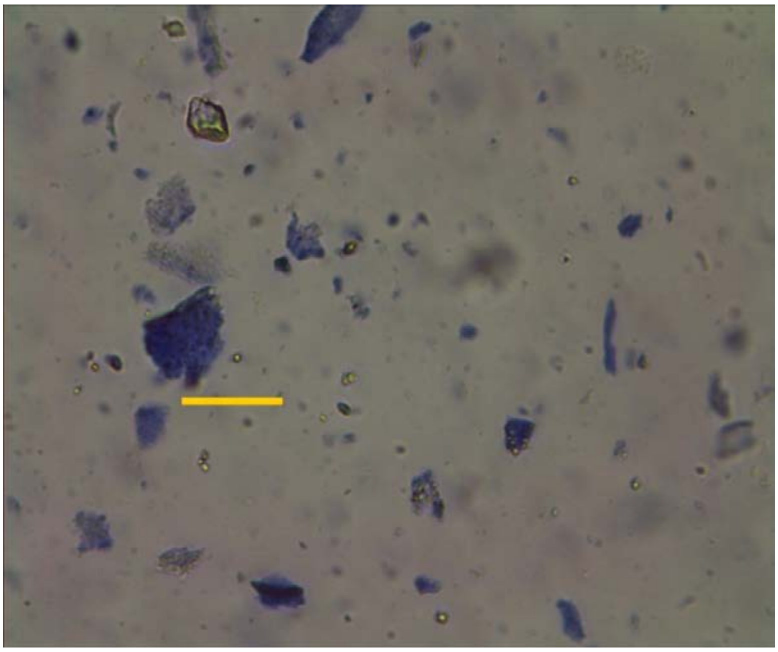

Figure 5.67: Sand layer from soil 4; TSPP-1.

In Figures 5.67 and 5.68, silt sized particles were found in the sand layer. The pattern of settlement for TSPP was not identical to those of Calgon and Sodium silicate/oxalate for this soil; coarse and fine layers were not clearly defined. TSPP continued to show apparently better dispersion compared to the other chemicals, clean silt particles were found with very small clay particles attached to them.

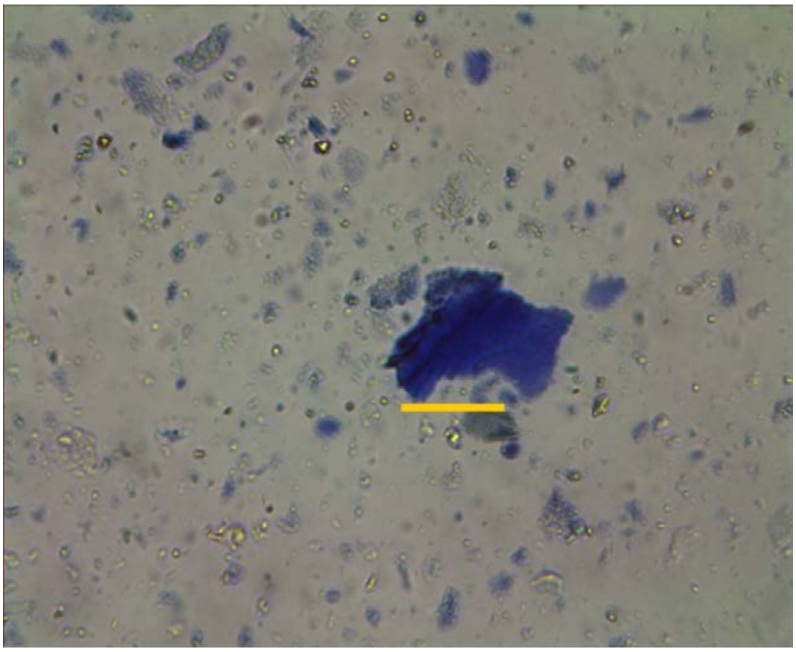

Figure 5.68: Sand layer from soil 4; TSPP-2.

5.10 Soil 4 - Fine Sand Layer

5.10.1 Calgon

The string-like structures are more obvious and larger in size in the fine sand layer. This is consistent with agglomeration occurring during settlement, since the finer sand layers settle at a slower rate. Agglomerations are completely deeply blue stained in all the photographs (Figures 5.69 and 5.70). All the silt sized particles have high CEC particles sticking to them.

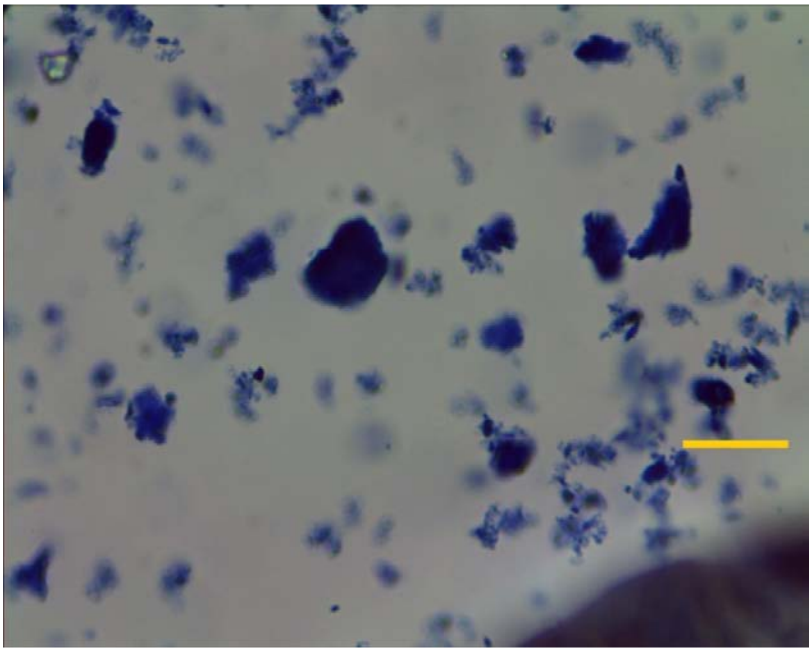

Figure 5.69: Fine sand layer from soil 4; Calgon -1.

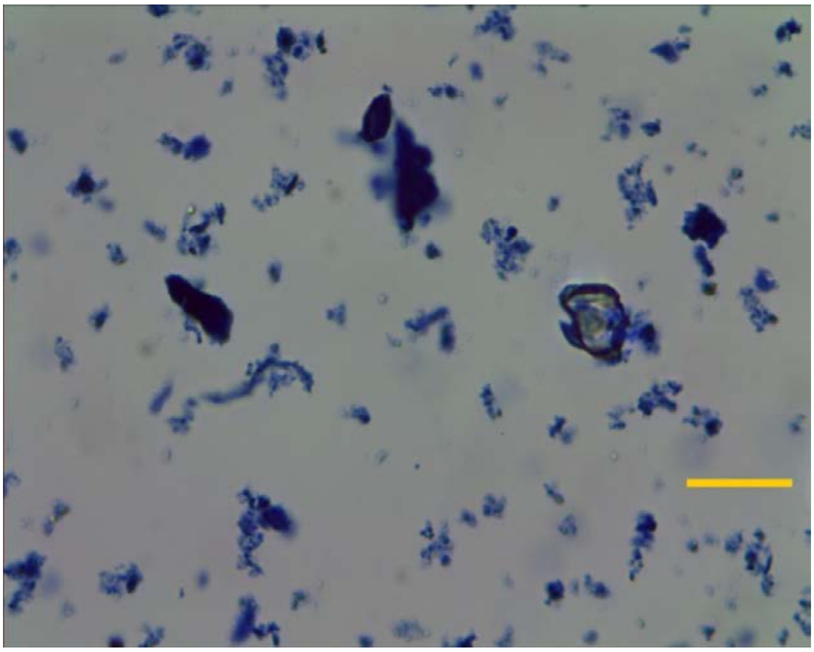

Figure 5.70: Fine sand layer from soil 4 ; Calgon -2.

Much of the material visible is clearly not sand. The particle at the bottom right hand corner in figure 5.69 is a sand grain, the agglomerations are silt sized suggesting they have been carried down by larger particles as Savage (2007) suspected.

5.10.2 Sodium Silicate/Oxalate

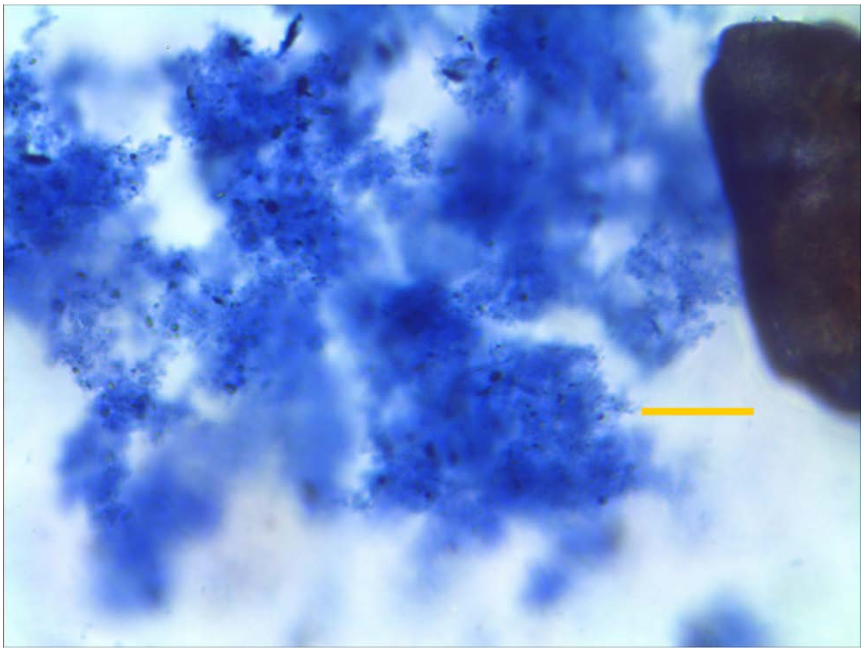

Figure 5.71: Fine sand layer from soil 4 ; Sodium Silicate/Oxalate -1.

The agglomerations in both Figures 5.71 and 5.72 are the same size as a genuine sand grain but are made up of extremely small particles that are completely blue stained. These tenuous nebular structures develop quite soon (a few minutes) after mechanical stirring has finished and appear to be resistant to low-energy agitation.

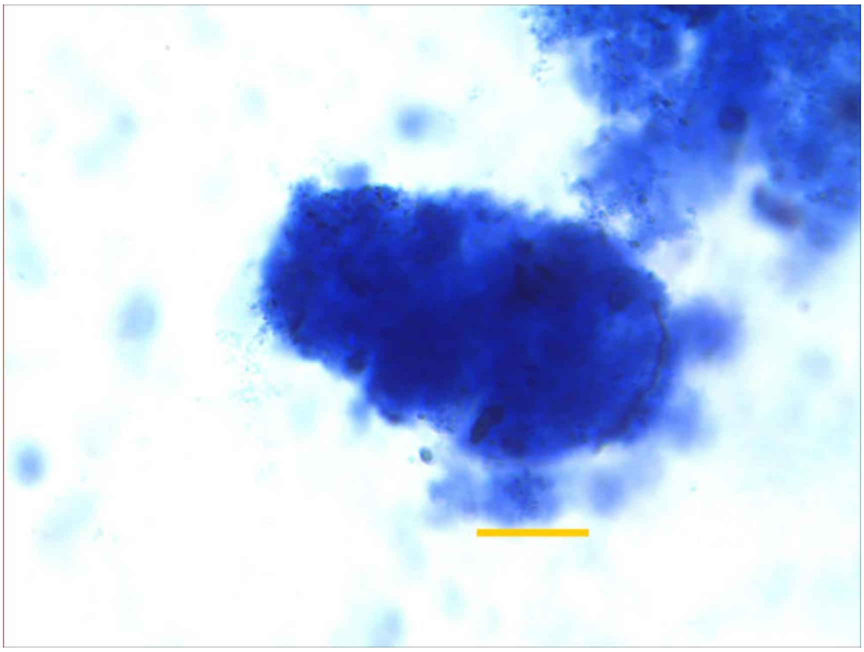

Figure 5.72: Fine sand layer from soil 4 ; Sodium Silicate/Oxalate -2.

More photographs are shown in the appendix A.

5.11 Summary

The study makes no numerical estimate of the inaccuracy of the hydrometer, but the results suggest reasons why the hydrometer is unlikely to give reliable results for clayey soils.

5.11.1 Calgon

Free silt size particles are very scarce in the sand layers throughout the different samples. Agglomerations of deeply blue stained, high CEC particles are present in the sand layer. Throughout the different samples the increase of blue stained clay particles increase in the silt layers. In soils 1 and 2, the sand layers appear to have more high CEC particles in agglomerations compared to the silt layer. The sand grains and silt particles have clay attached to them.

Soils 3 to 6 dispersed with Calgon and Sodium silicate/oxalate, sediments were divided into coarse and fine layers, while TSPP only into sand and silt. String-like structures in the sand layers of soils 3 to 6 became more frequent in the silt layers. For all samples dispersed with Calgon the silt layer appeared to have more clay-sized particles than in the sand layers.

5.11.2 Sodium Silicate/Oxalate

Sand layers contained stained silt sized particles. Agglomerations are evident throughout the layers, coarse and fine. Agglomerations decreased in the silt layers. Sodium silicate/oxalate appeared to leave more high CEC particles in the sand layers than in the silt layers.

5.11.3 TSPP

TSPP dispersing pattern presented more consistency throughout the different layers. Free silt sized particles in the sand layer are more evident when compared to the other two dispersing chemicals. Agglomerations in the sand layer seemed to be about the same size as the genuine sand particles. In the silt layer, agglomerations were found to be larger than the genuine silt particle size for both high and low CEC particles. Summary of the research findings are included in table 5.1 below.

Table 5. 1: Observations summary

Sample	**Layer**	**Dispersing agent**		
		Calgon	**Sodium silicate/oxalate**	**Tspp**
Soil 1& Soil 2	Sand	Agglomeration of deep blue stained particles.	Coarse and fine layers particles agglomerations are evident. High CEC in the sand layer evident.	Agglomerations in the layer were same size as genuine sand particles. Free silt-sized particles evident.
Soil 3 to Soil 6	Coarse sand	String-like structures consisting of clay and silt-sized particles agglomeration.		
Soil 1 to Soil 6	Silt	Increase of string-like structures consisting of stained particles compared to the sand layers. More clay-sized particle content.	Particle agglomeration decreases	Agglomerations larger than genuine silt particles, both high and low CEC.

CHAPTER 6 : CONCLUSION

6.1 General

To be effective, a dispersing agent should give complete separation of particles into sand, silt and clay and reduce agglomeration of particles to a size not larger than the smallest size group desired to be measured. This study illustrates that it is very unlikely that, for many South African soils, the hydrometer test will give reliable results no-matter what dispersant is used. The hydrometer was introduced in northern latitudes where clays are often of glacial origin and the hydrometer may give acceptable results for such soils. None of the soils tested in this investigation were of glacial origin; they are of the soils derived from the sedimentary and igneous rocks of central South Africa. All tests showed significant clay content in layers not supposed to contain a significant proportion of clay. The study confirms Savage’s suspicions that not all soils are fully dispersed at the time of testing and that clay particles are carried down by larger particles. This statement agrees with findings of this investigation, throughout all different samples and dispersing agents. Clay particles were present in all the layers (sand and silt).

The different standards discussed in Section 2.2 define clay sized as particles as particles smaller than 5µm or 2µm. This seems to be acknowledgement that 2µm probably underestimates the clay fraction.

It has not been established that all the stained material is clay. Some could be organic colloids, some could even be groupings of hydrated cations. Even if it is not all clay, it is part of the soil and affects the results of the hydrometer by binding particles into agglomerations which settle at a rate inappropriate for interpretation by hydrometer theory.

Coating of large particles with high-CEC particles was present in all dispersed layers. Generally, the coating on sand particles was quite thin in comparison with the size of the sand grains and therefore unlikely to make up a large fraction of material in the layer. The hydrometer was therefore likely to give an acceptable assessment of the sand fraction in many cases. The exploring of different dispersants also suggested that, although different dispersants show different inadequacies, none appears to be effective enough to be considered reliable.

The available literature on the different dispersant is dealt with in the literature study. It appears that the most relevant study for comparison with this investigation is that of Wintermyer and Kinter (1955). They found that sodium polyphos and sodium tripolyphosphate (chemicals not part of this study) were highly effective for all soil types except laterite soils. Calgon and sodium tetraphosphate (also not part of this study) were found to be less effective. TSPP was found to have high dispersion value for laterite soil. This research did not focus on dispersion, but it did appear to indicate that dispersion of silt and clay particles could be somewhat more successful with TSPP than with the other chemicals used.

Calgon treated samples displayed more clay particles in the silt layers and agglomerations with silt particles forming cloud-like structures. This suggests that smaller particles may be more effective in carrying down clay particles than larger particles. The silt layers were frequently found to have agglomerations made up of fine silt and clay and, in some circumstances, clay without silt cores. These agglomerations are larger than the genuine single particles of the layer, which suggests that the agglomerations settle more slowly than single particles of their aggregate size, but quicker than the individual small particles from which they are made up. The hydrometer analysis for Calgon treated samples appears likely to over-estimate the amount of silt and under-estimate the amount of clay for high clay content soils. Calgon is the dispersant specified by the current South African standard.

Sodium silicate/oxalate showed many fine clay particles to have settled with bigger particles. This could be relevant to the suggestion by Savage (2007) that clay particles may be carried down by larger particles. Sodium silicate/oxalate was the specified dispersant at that time. Wintermyer and Kinter (1955) found the Sodium silicate/oxalate to be amongst the 14 least effective dispersing chemicals out of 19 that were tested.

TSPP dispersing of samples seemed to be better when compared with Calgon and Sodium silicate/oxalate under the microscope, although silt and clay sized particles are still found as agglomerations in the silt and sand layer.

Based on Stokes' law, particles are assumed to be spherical. According to Jansen van Rensburg *et al.* (2012), visual examination under high magnification using SEM and petrographic microscopy showed clay particle to resemble flat discs when dry. Di Stefano *et al. (*2010) found clay particles to be generally platy or tubular in shape. These findings may have little relevance for the hydrometer, which deals with clay suspended in water. In hydrated conditions clay particles become completely surrounded by layers of hydrated cations. Particles and agglomerations observed in this study tended to be rounded, and it may be that the shape of individual dry clay particles is less relevant than generally believed for the accuracy of hydrometer analysis. The shapes evident in completely stained clay agglomerations did not appear to be based primarily on the shapes of the core particles. They appear to depend on how clay particles agglomerate. The shapes observed under the optic microscope were mostly rounded. The major problem for hydrometer analysis appears to be inadequate dispersion

Stott and Theron (2015) mentioned that test methods in SANS 3001 GR3 may be adequate for road construction where granular soils are the norm, but they are not adequate for assessing heave potential under building foundations. It is however important to note that roads also suffer damage from heaving clay particularly in the roadbed. This could be due to underestimation of high CEC particles.

The postulate that agglomerations would not precipitate at the rate expected for clay (Stott and Theron, 2016) was supported by this microscopic investigation. This could explain many of the misleading results which have led to observed failures of both roads and building foundations. Most of the silt particles have a coating of deeply blue stained fine particles. It appeared to be the small, high CEC particles which hold low CEC silt and sand particles together into sand-sized agglomerations. It also seems likely that silt and clay particle agglomerations found in the sand layer were carried down by the sand. Another possibility is that some of the agglomerations were in the water right at the bottom of the cylinder at the start of the test. In the other layers it appears that clean particles precipitate together with agglomerations which are larger than the clean particles themselves. This suggests that agglomerations precipitate more slowly than single particles of their aggregate size, probably due their lower density.

Forouzan's research study (2016) included conducting hydrometer analysis on 100% mixtures of 2 types of clay. Hydrometer estimates for the clay content of these mixtures ranged from 37% to 55%; errors therefore ranged from 63% to 45%. The activity of soil is derived from the clay content. This soil activity is then used for design purposes. Thus, the accuracy of determining clay content is very important. Projects have failed due to expansive clay and such failures will probably continue as long as hydrometer determination of clay fraction is accepted without question. Table 6.1 demonstrates how the research objectives were met.

Table 6. 1: Research Objective

Original objective	**Findings**	**Objective met**
Assess Savage's suggested shortcoming that dispersion maybe incomplete at the time of testing. (Savage, 2007)	The sand particles, which settle within seconds, were coated with clay showing that poor dispersion was present from the time the test started.	Inadequate dispersion confirmed.
Clay particles being partially carried down by larger particles. (Savage, 2007)	The high concentration of clay and clay agglomerations in the silt layers.	Clay carried down confirmed.
Agglomeration may occur during settlement in the hydrometer test (Nettleship, 1997).	The fact that more agglomerations are found in the layers settling out more slowly.	Agglomeration of particles confirmed.
Speculation that the agglomerations which they observed will not settle at the rate of their individual particles. (Stott and Theron, 2015)	Agglomerations being in the wrong layers for the sizes of their constituent particles.	Speculation confirmed.

6.2 Recommendations

The findings from the research are that it is imperative to find a better method to estimate clay content, as the hydrometer may underestimate it. While the research around clay fraction is continuing, and until a better method is available, the recommendation would be the emphasis of appointment of competent geo-practitioners to conduct a thorough geotechnical investigation, and not simply to accept the results of foundation indicator tests without question. Doubts have been raised about the reliability of hydrometer methods, and it appears that in some cases hydrometer assessment of clay fraction can be very misleading. This research has shown that agglomerations may occur while sedimentation is in progress, affecting the accuracy of the results. This implies that if sedimentation procedures based on Stokes' Law are to be successful then they must be completed in a very short time.

6.3 Future research

Possible future research will be to examine a simple method of performing rapid sedimentation tests using relatively simple equipment with the aim to find a method of assessing clay fraction before significant agglomeration can take place.

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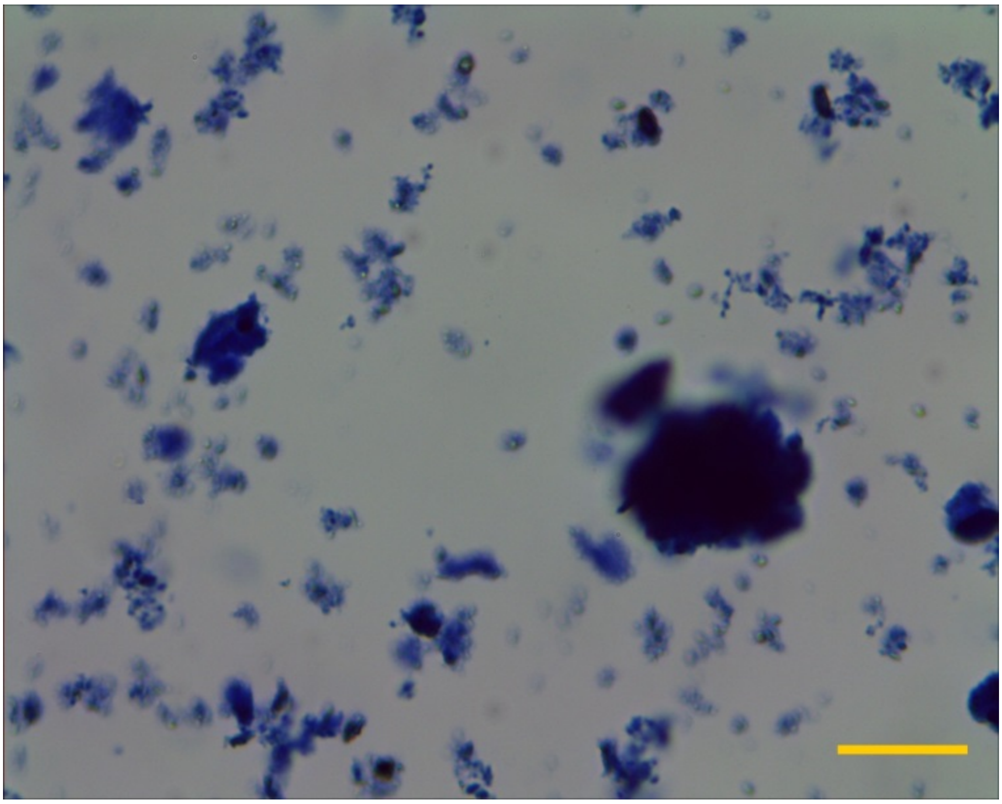
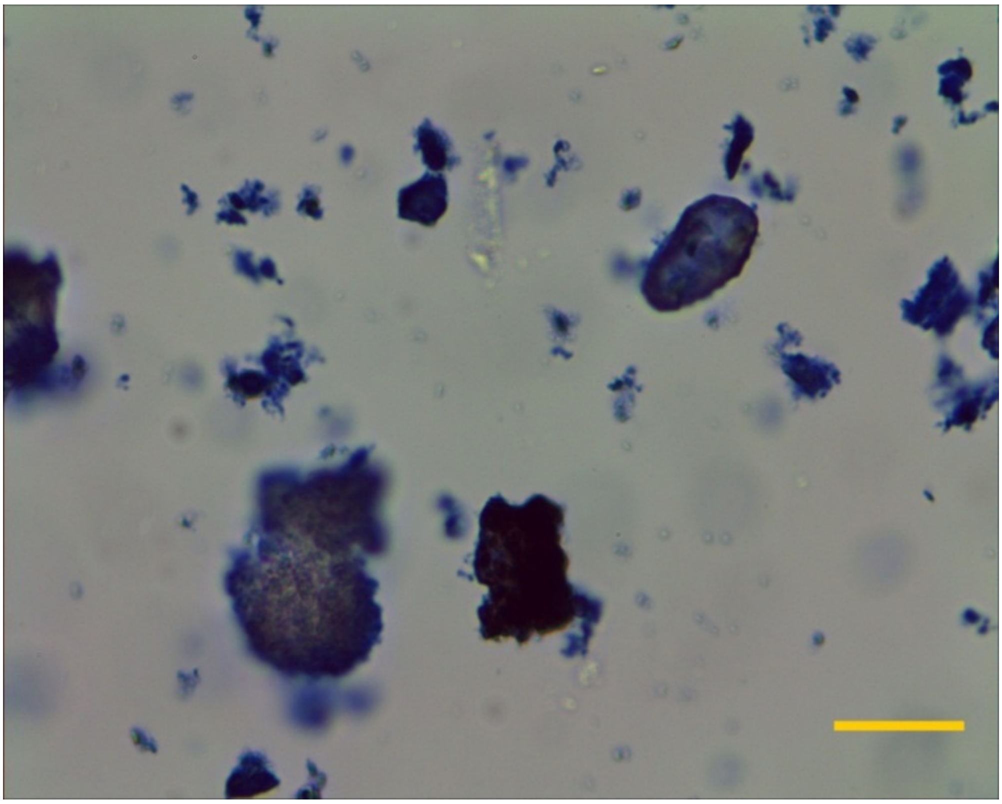
APPENDIX: A PHOTOMICROGRAPHS

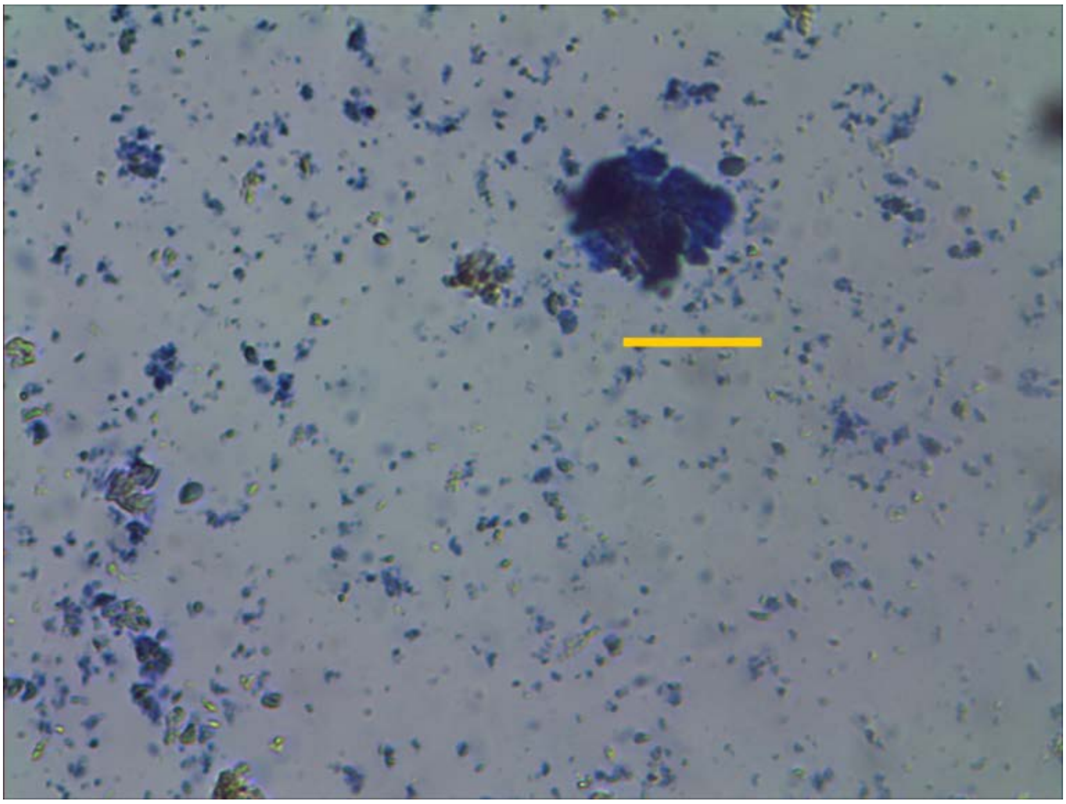
Summary of photographs showing various soil samples and different dispersants



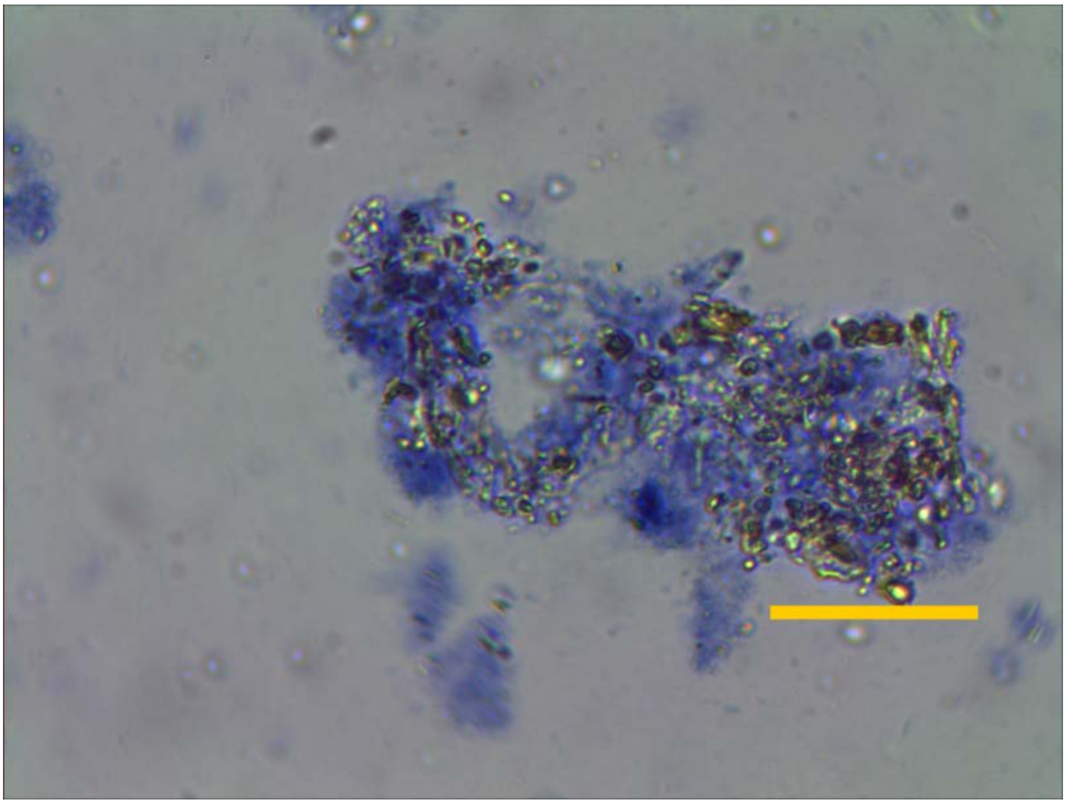
APPENDIX A - Figure 1: Fine sand layer from soil 4 – Calgon



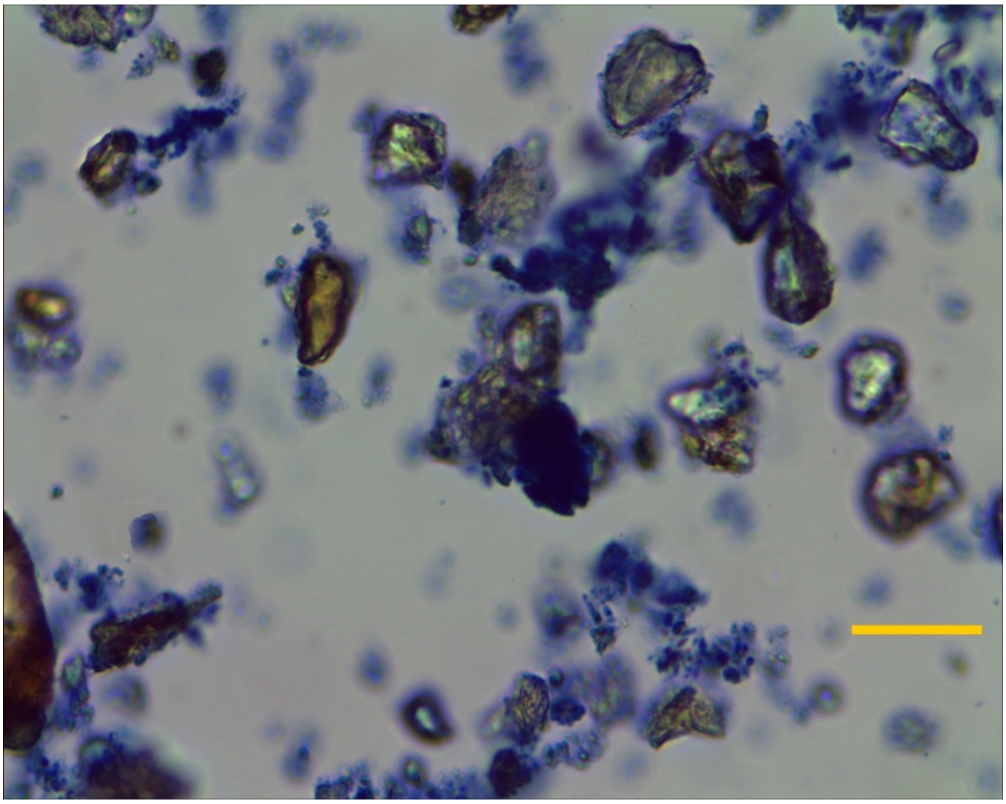
APPENDIX A - Figure 2: Coarse silt layer from soil 4 – Calgon



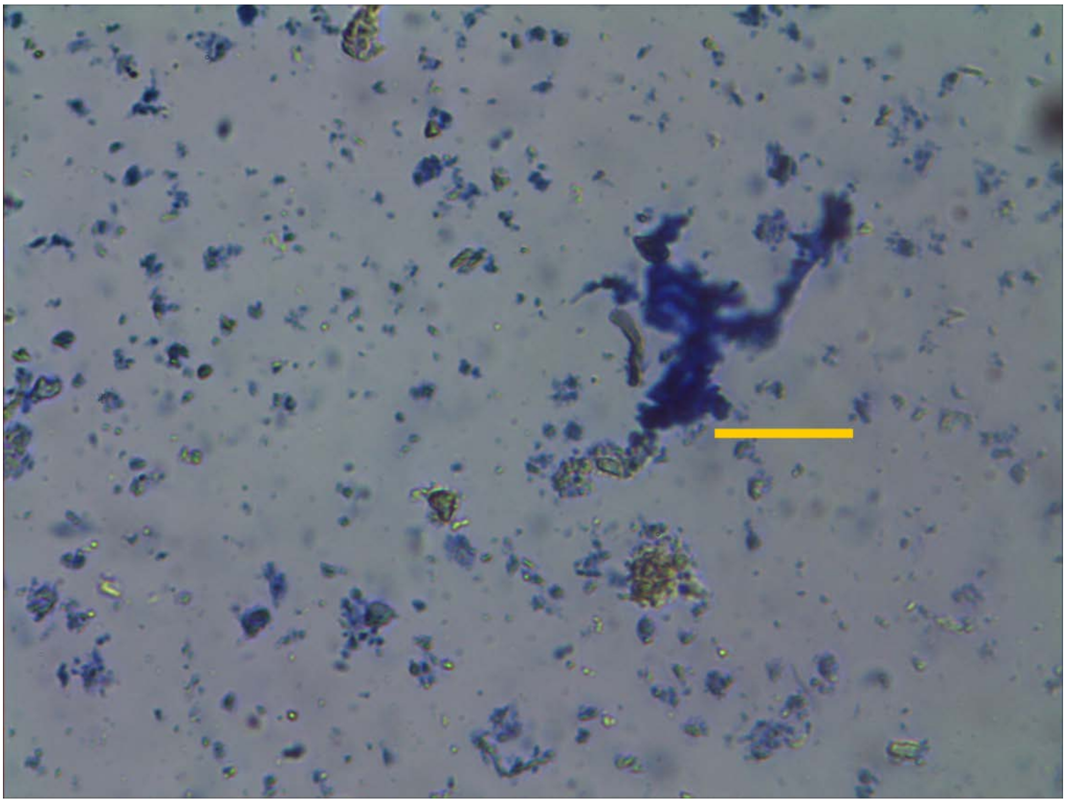
APPENDIX A – Figure 3: Coarse silt layer from soil 4 - Sodium Silicate/Oxalate



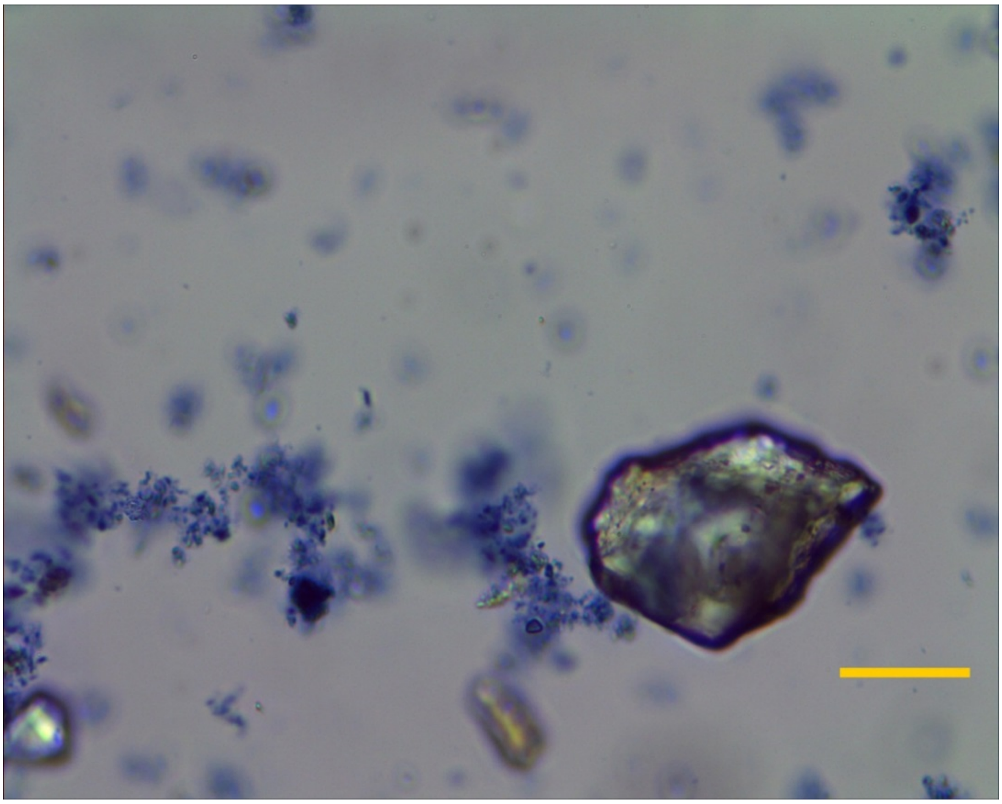
APPENDIX A - Figure 4: Silt layer from soil 4 – TSPP



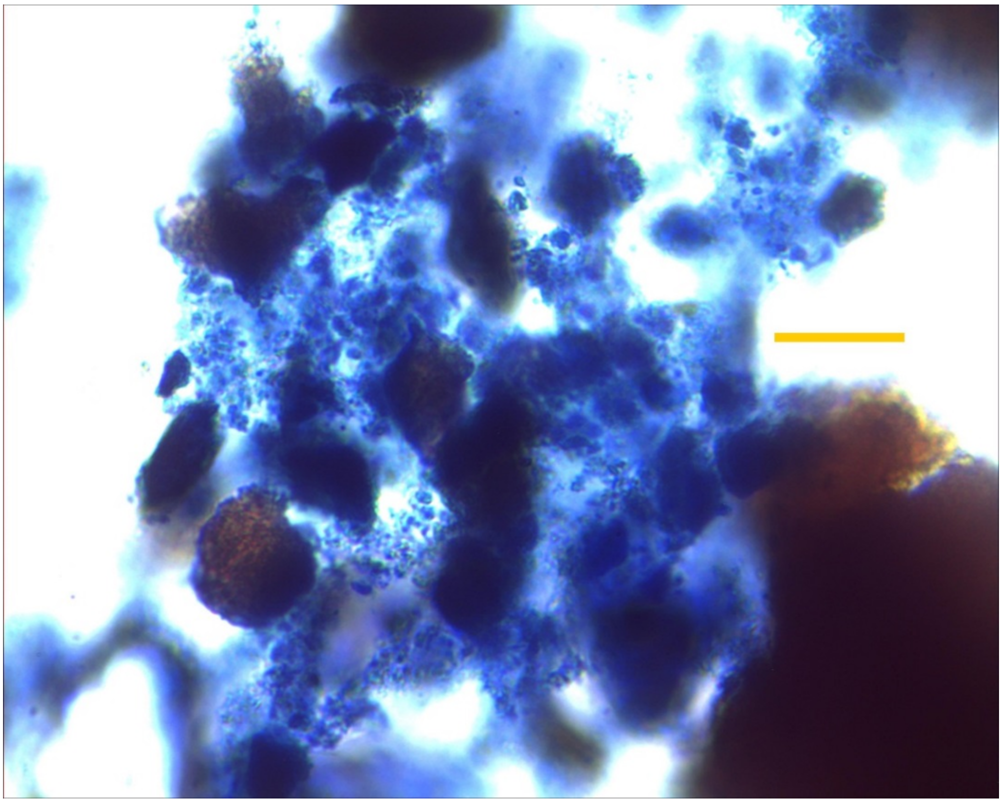
APPENDIX A - Figure 5: Fine silt layer from soil 4 - Calgon



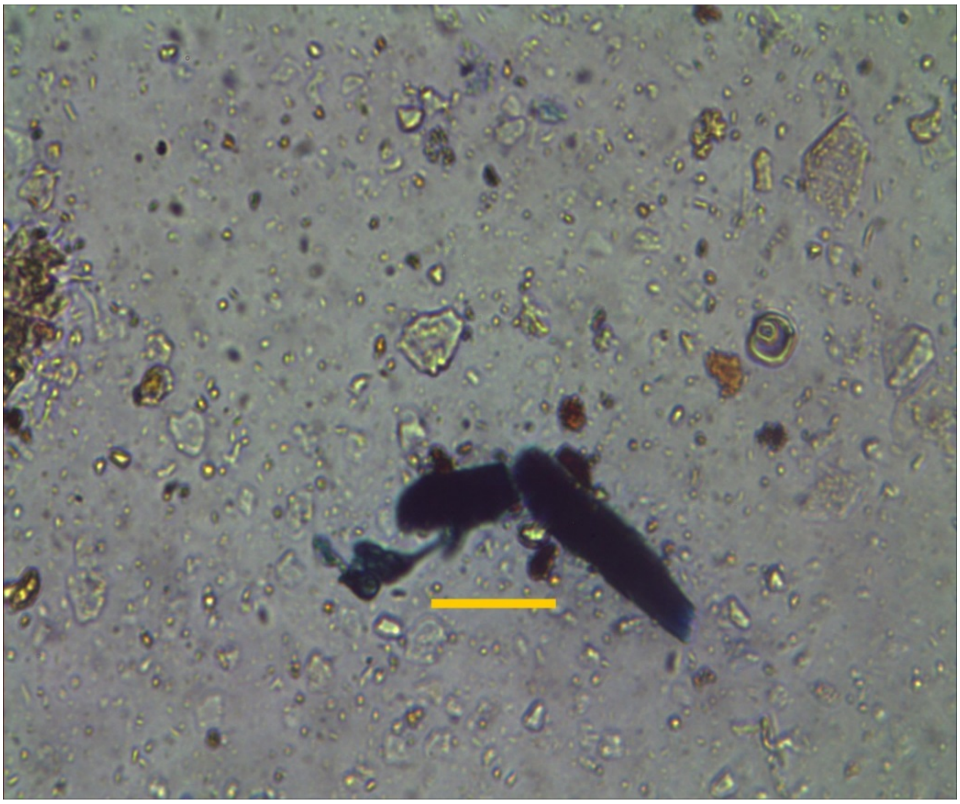
APPENDIX A - Figure 6: Fine silt layer from soil 4 - Sodium Silicate/Oxalate



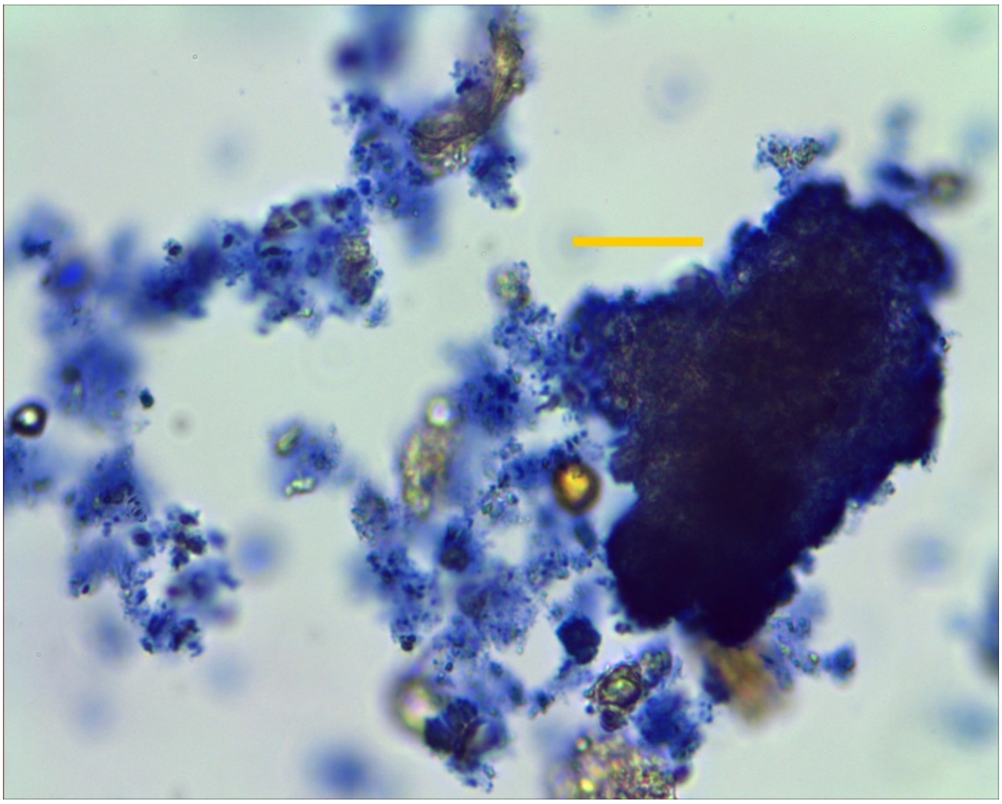
APPENDIX A - Figure 7: Coarse sand layer from soil 5 – Calgon



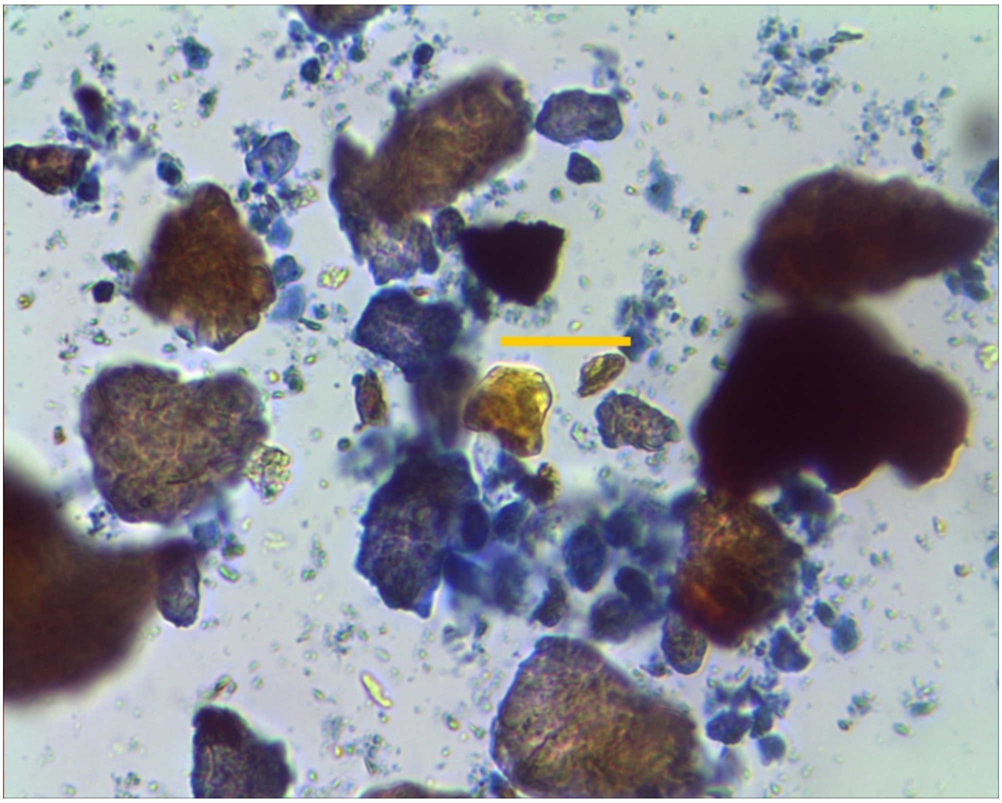
APPENDIX A - Figure 8: Coarse sand layer from soil 5 - Sodium Silicate/Oxalate



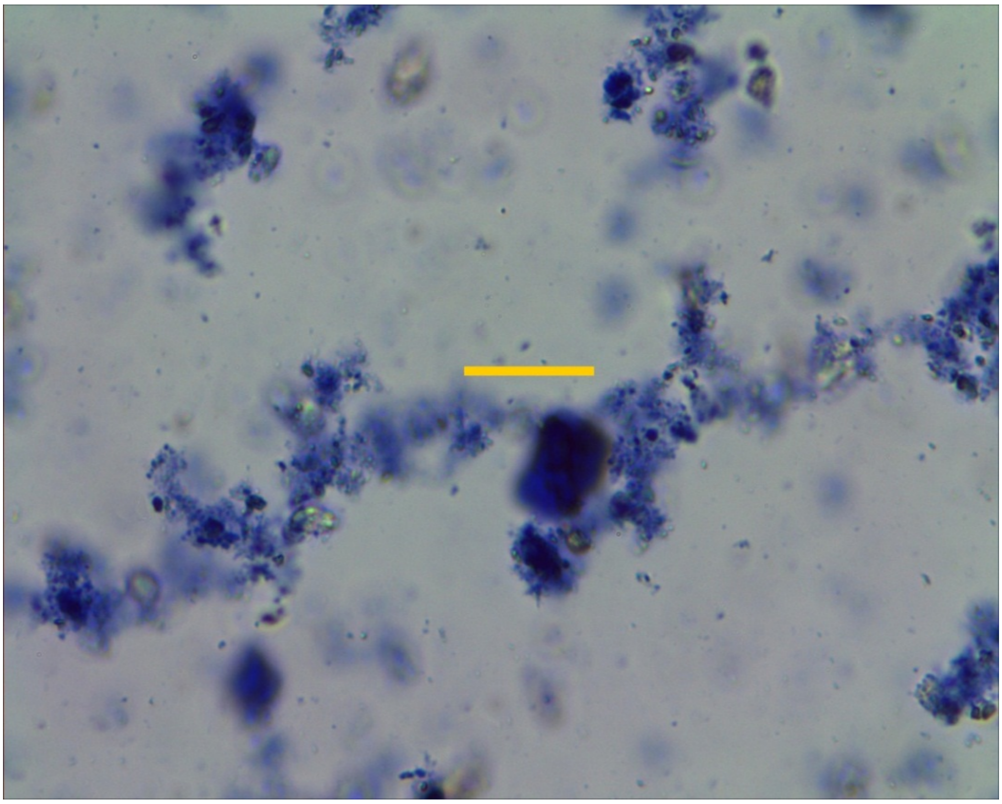
APPENDIX A - Figure 9: Sand layer from soil 5 – TSPP



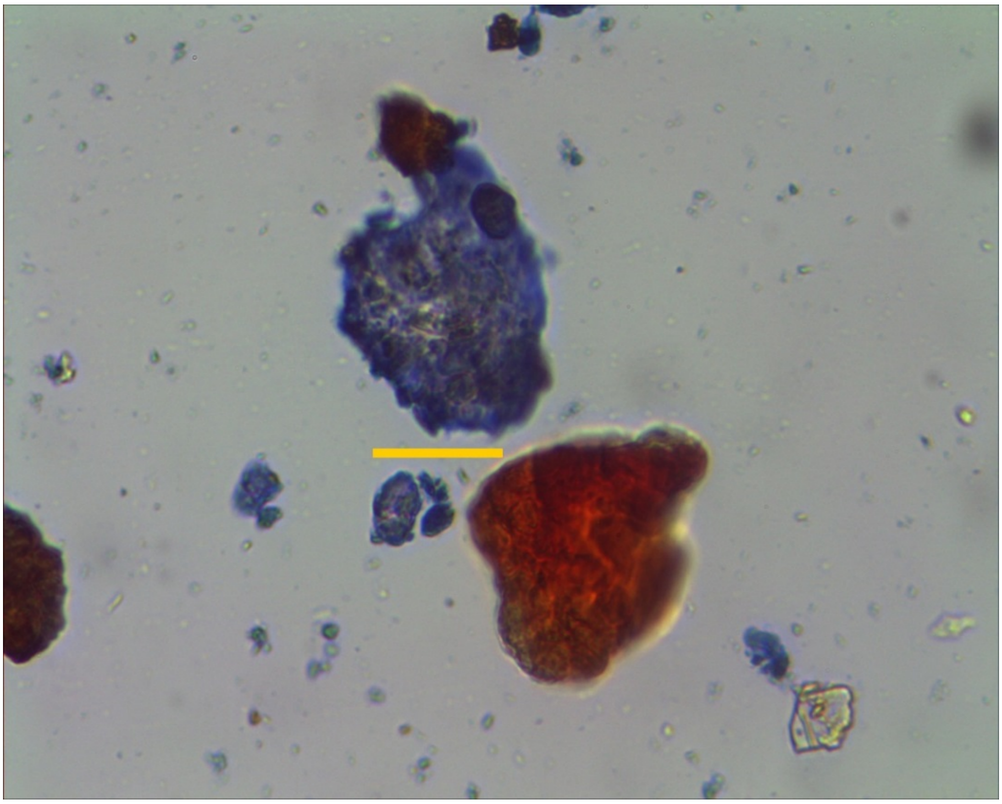
APPENDIX A - Figure 10: Fine sand layer from soil 5 – Calgon



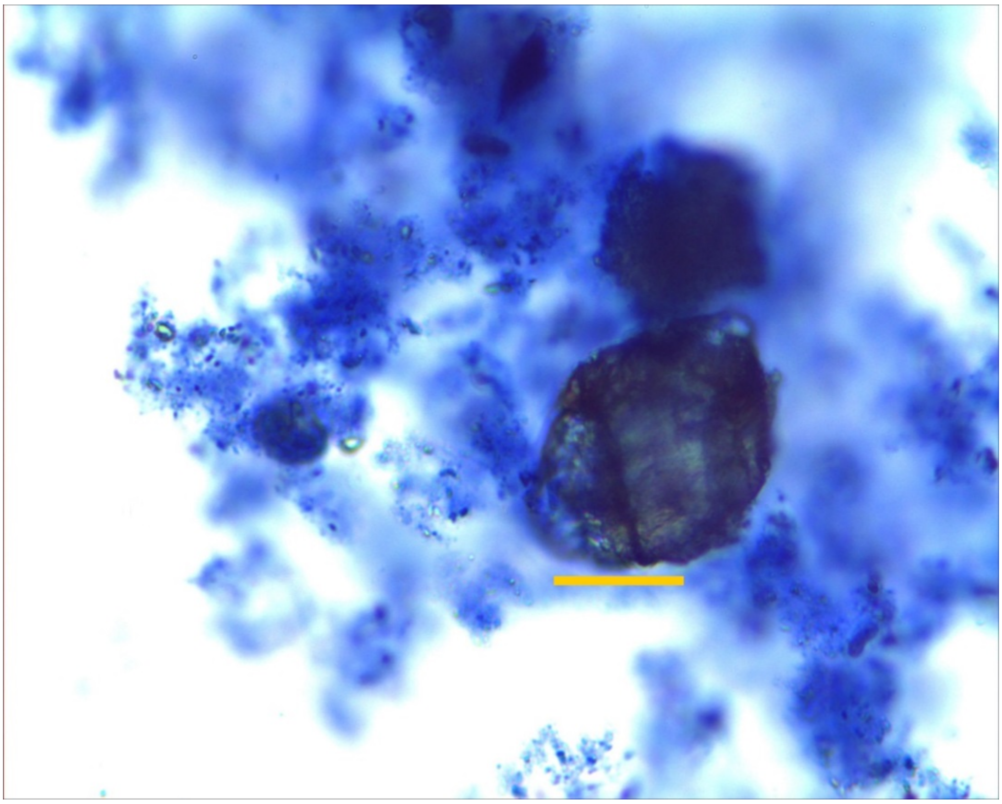
APPENDIX A - **Figure 11:** Fine sand layer from soil 5 - Sodium Silicate/Oxalate



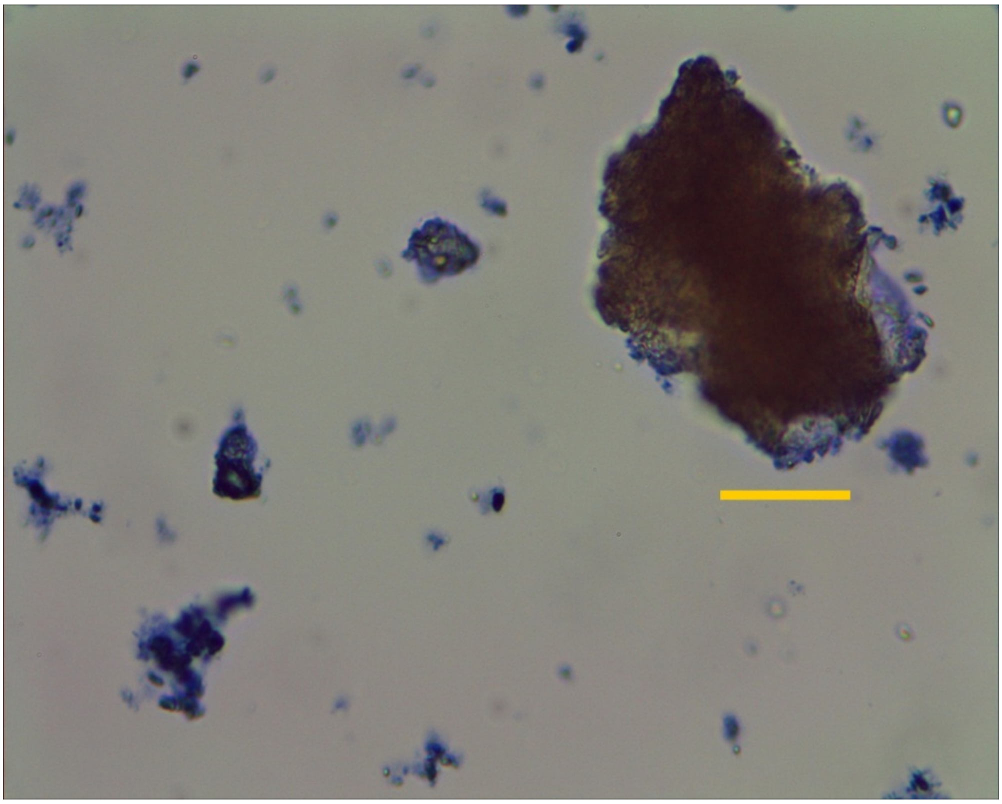
APPENDIX A - Figure 12: Coarse silt layer from soil 5 - Calgon



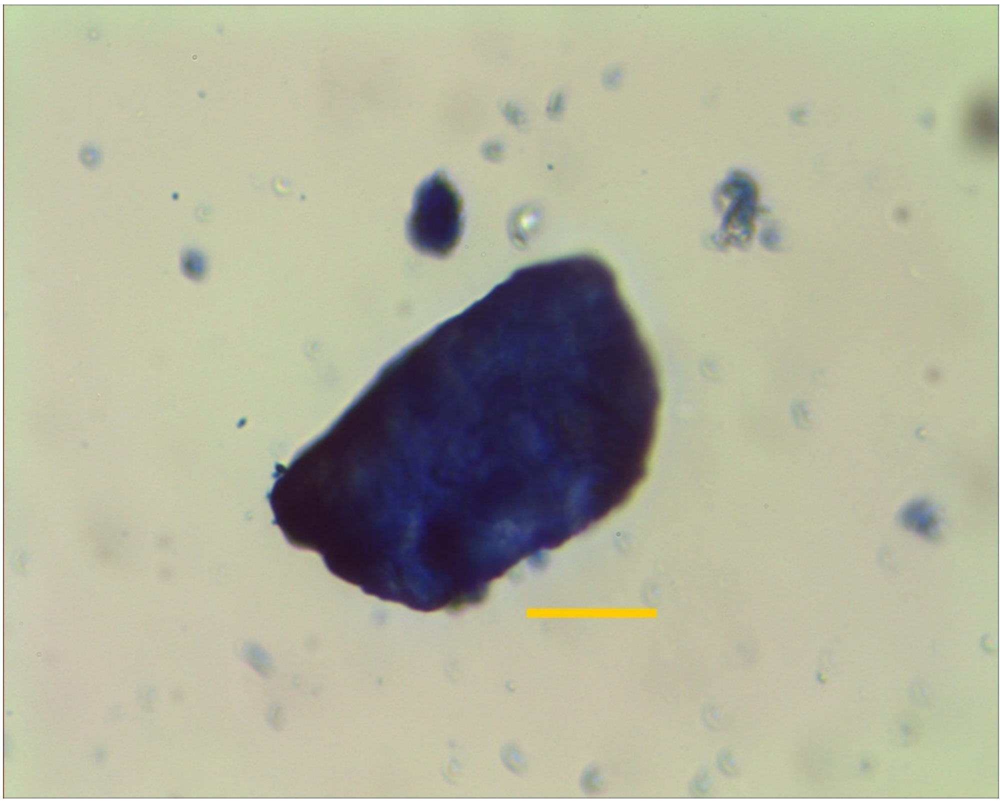
APPENDIX A - Figure 13: Coarse silt layer from soil 5 - Sodium Silicate/Oxalate



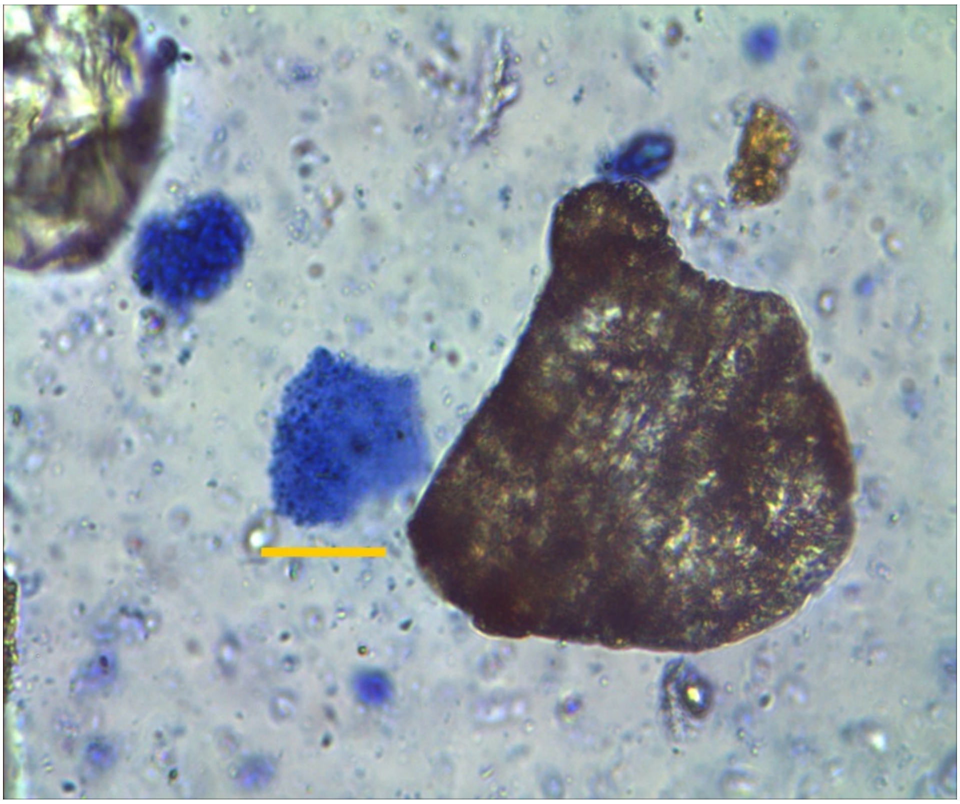
APPENDIX A - Figure 14: Fine silt layer from soil 5 – Calgon



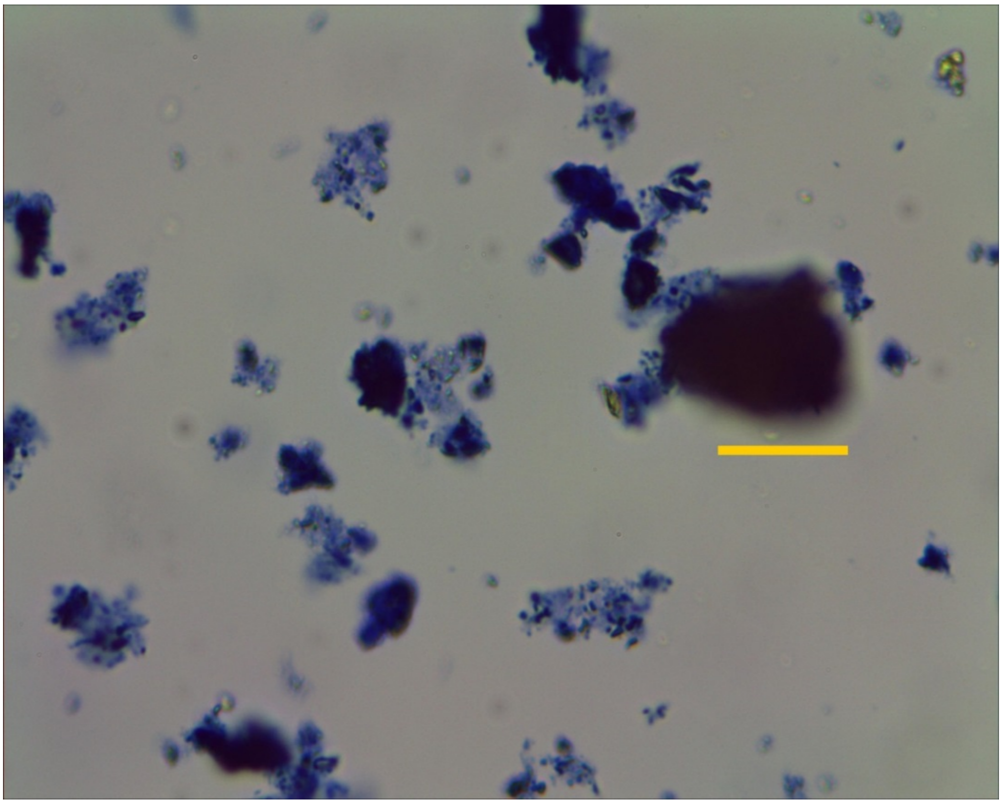
APPENDIX A - Figure 15: Coarse sand layer from soil 6 – Calgon



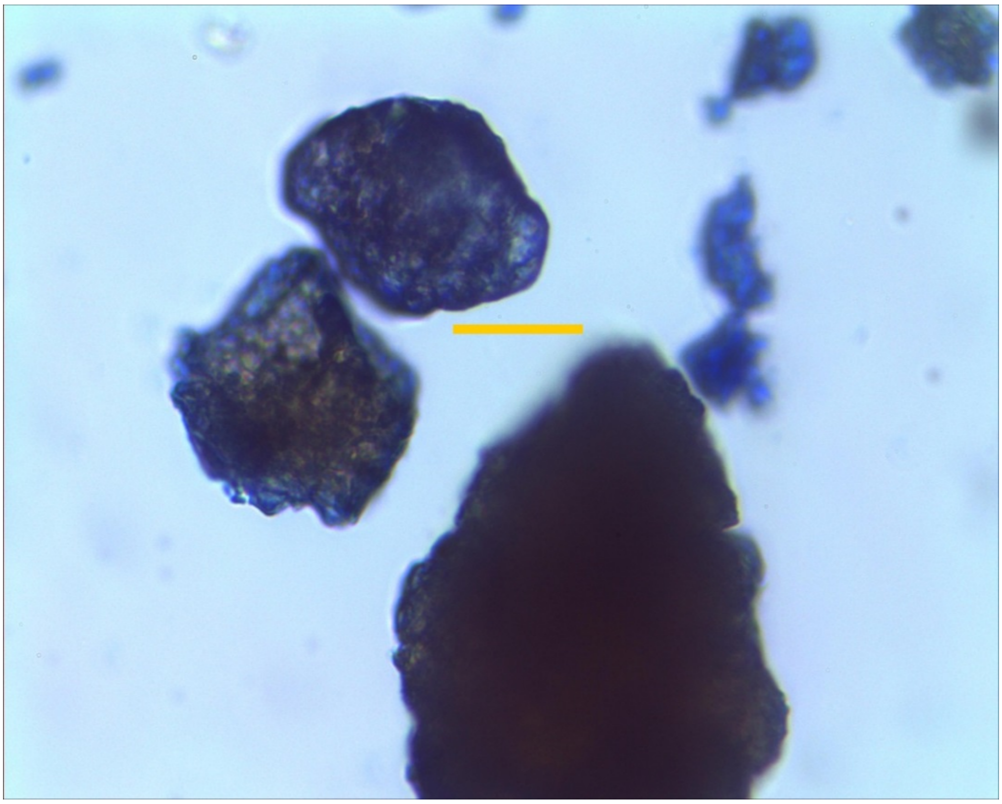
APPENDIX A - Figure 16: Coarse sand layer from soil 6 - Sodium Silicate/Oxalate



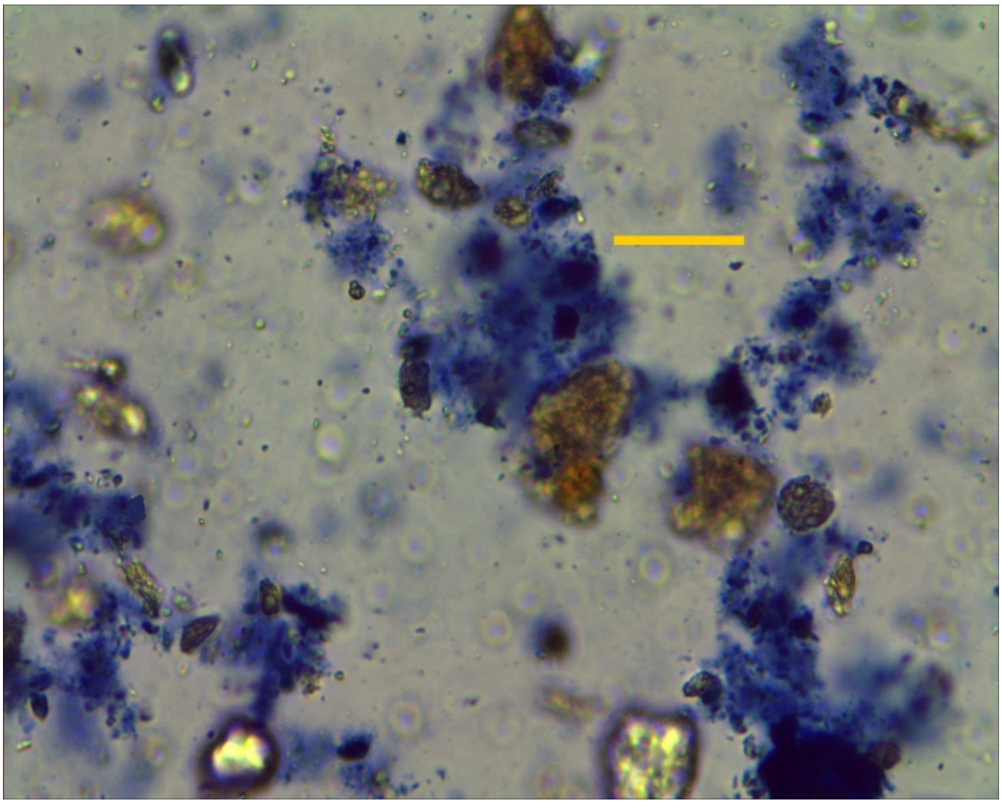
APPENDIX A - Figure 17: Coarse sand layer from soil 6 – TSPP



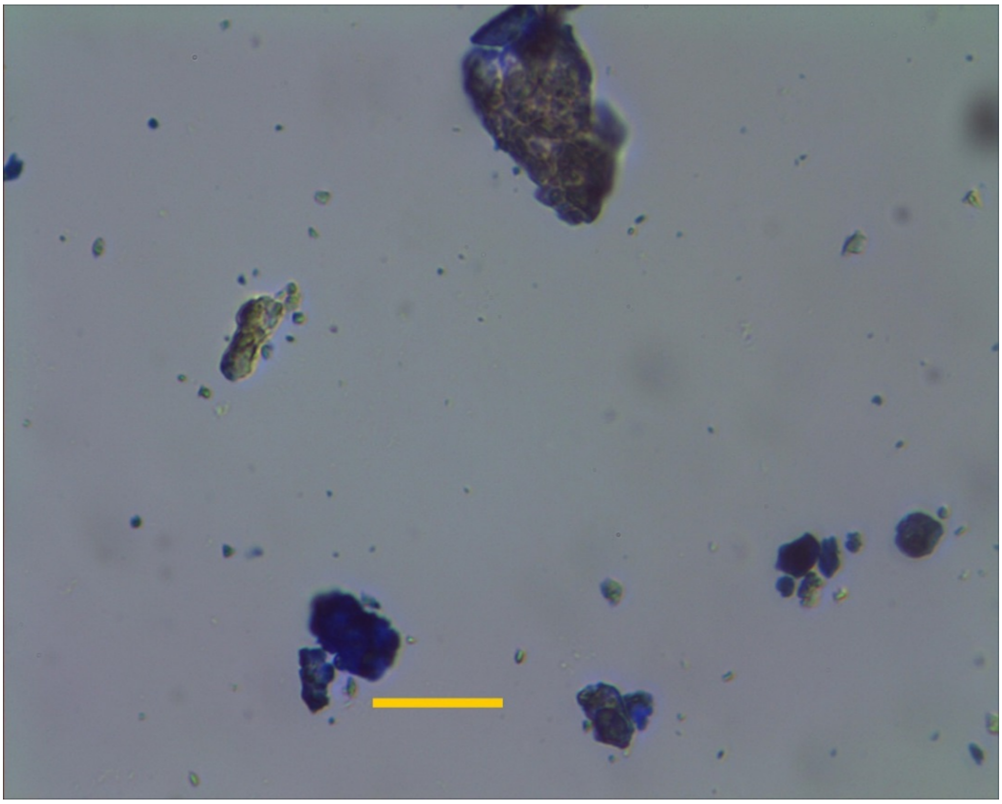
APPENDIX A - Figure 18: Fine sand layer from soil 6 – Calgon



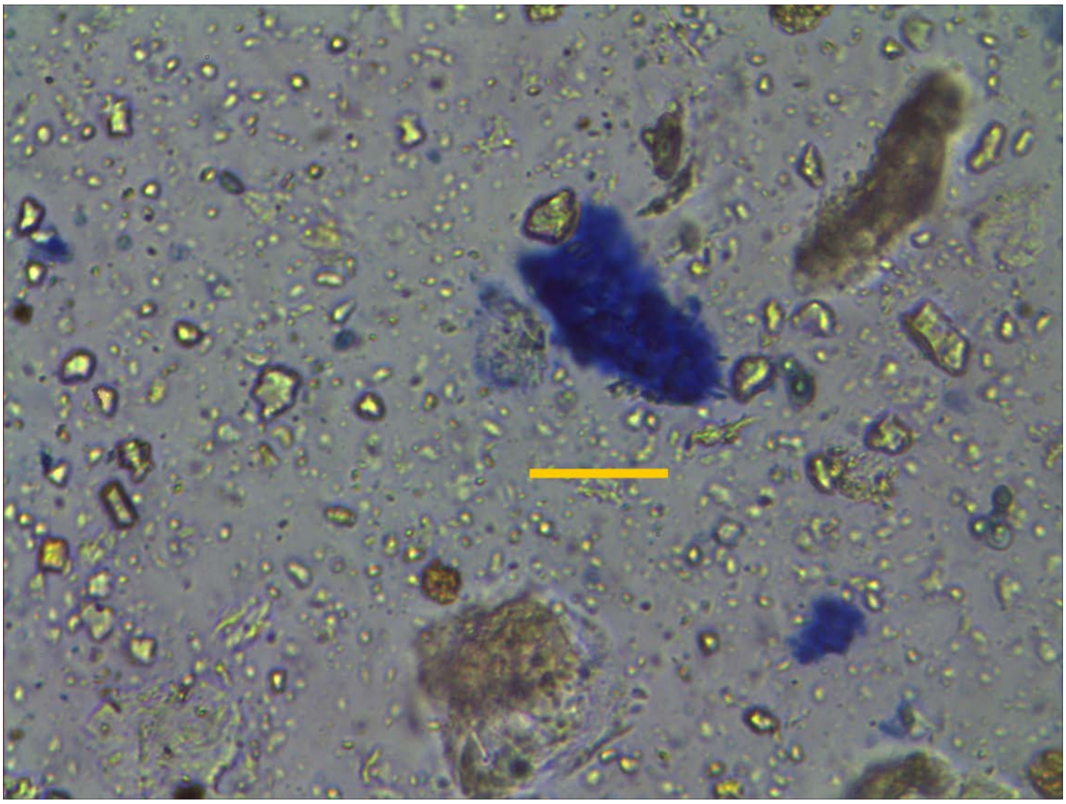
APPENDIX A - Figure 19: Fine sand layer from soil 6 - Sodium Silicate/Oxalate



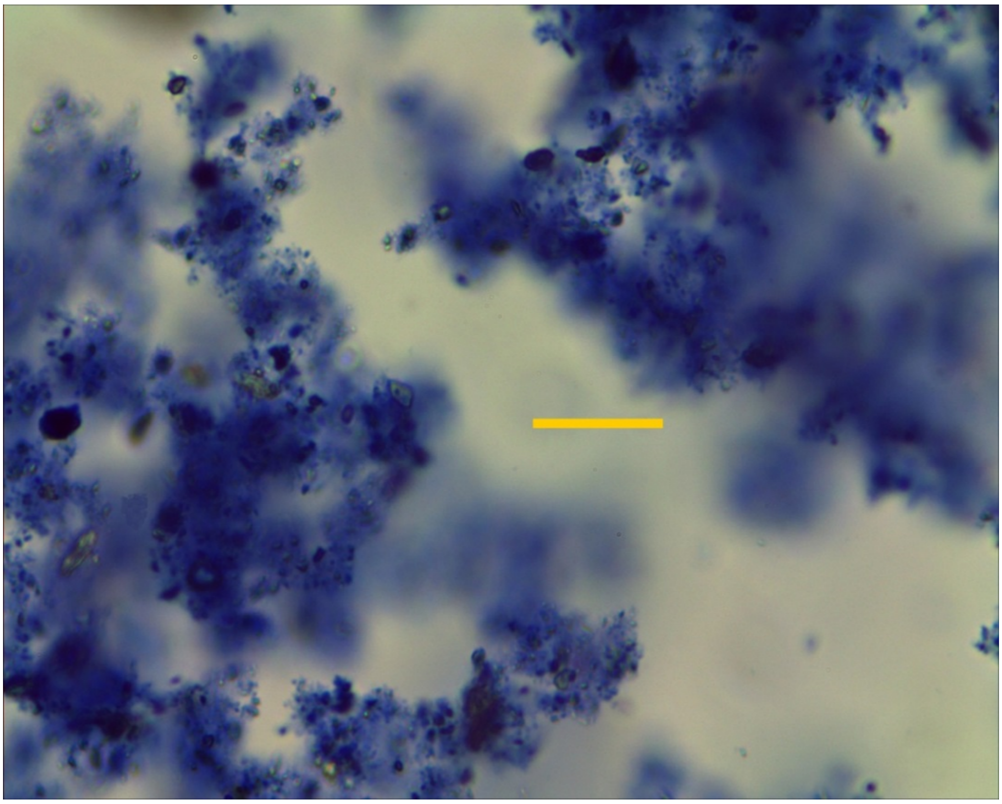
APPENDIX A - Figure 20: Coarse silt layer from soil 6 – Calgon



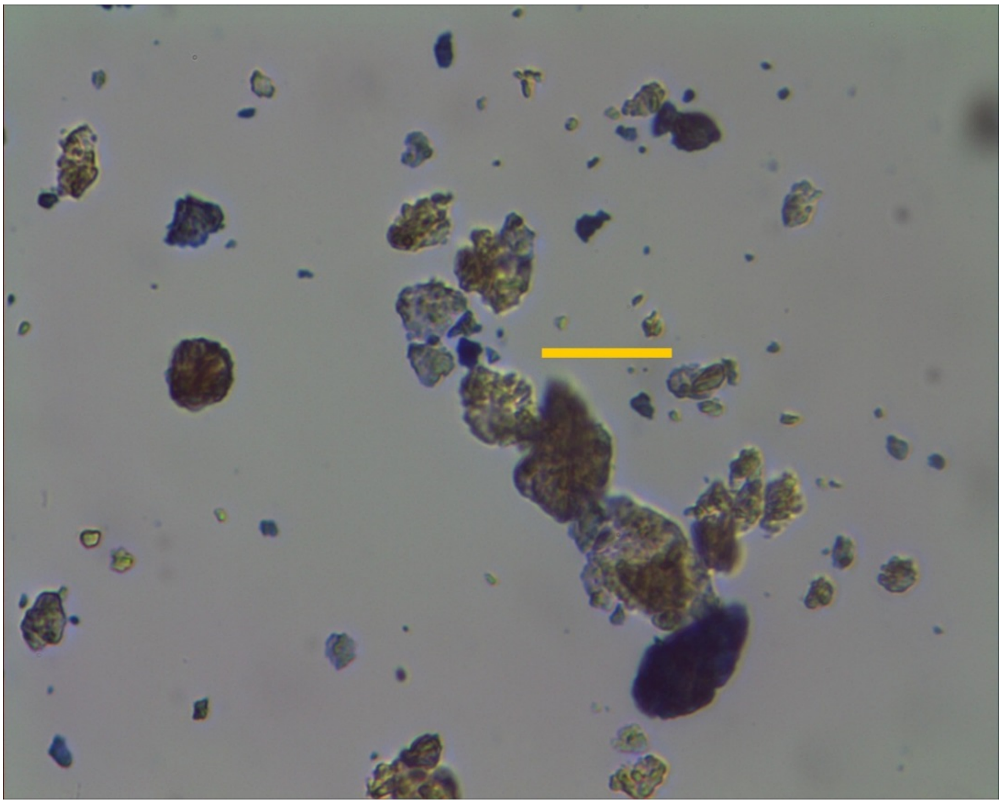
APPENDIX A - Figure 21: Coarse silt layer from soil 6 - Sodium Silicate/Oxalate



APPENDIX A - Figure 22: Silt layer from soil 6 – TSPP



APPENDIX A - Figure 23: Fine silt layer from soil 6 – Calgon



APPENDIX A - Figure 24: Fine silt layer from soil 6 - Sodium Silicate/Oxalate