

# The Mwendwa Protocol: A modification of the Bouyoucos method of soil texture analysis

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## Research

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# Abstract

## Background

This study aimed to modify the Bouyoucos method of soil texture analysis by proposing the best sample treatment and analysis protocol that would give the most accurate results. The Bouyoucos method lacks sample pre-treatment whereby samples are dispersed for only 2 minutes after being soaked in 5% sodium hexametaphosphate (calgon) for 15 to 20 hours. In this study, the Pipette method was used as the standard method due to its increased precision and reproducibility. Different treatments modified from the Bouyoucos procedure were compared with the Pipette method aiming to identify the treatment whose soil texture proportions were closest to those obtained through the Pipette method. The treatments involved variation in the concentration of the digesting material during sample pre-treatment, concentration of the dispersing material, time of hydrometer readings, method of agitation before taking the first hydrometer reading and method of dispersing. Obtained data was analyzed statistically using Genstat and SPSS.

## Results

Percent sand obtained through the Pipette method significantly correlated with that obtained through Shaking at the 0.01 level ( $r = 0.862$ ;  $P\text{-value} = 0.001$ ) and that obtained through Shaking + Stirring at the 0.05 level ( $r = 0.737$ ;  $P\text{-value} = 0.015$ ). The sand measurements made using the hydrometer variations overestimated the sand fraction in the soil samples. None of the Bouyoucos treatments estimated the percentage silt with sufficient accuracy. There was a positive correlation in the clay proportion between the Pipette method and Shaking + Stirring treatment ( $r = 0.644$ ,  $P\text{-value} = 0.044$ ) at the 0.01 level. There was a negative correlation between the clay proportion obtained using the Pipette method and no-digestion treatment ( $r = -0.234$ ). The 4%Calgon treatment was negatively correlated with the Pipette method ( $r = -0.712$ ;  $P\text{-value} = 0.021$ ) at the 0.05 level. Treatments involving shaking and increased concentrations of hydrogen peroxide and calgon estimated the clay proportion with sufficient accuracy.

## Conclusions

These observations are indicative of the need for soil pretreatment with hydrogen peroxide and also the use 10% calgon as the dispersing agent during soil texture analysis. A modification to the Bouyoucos method was proposed and dubbed The Mwendwa Protocol.

## Background

There is no conventionally agreed protocol for soil texture analysis in terms of concentrations of reagents, shaking method and the time of readings. This paper aims to guide in the analysis of soil texture through modification and simplification of the Bouyoucos (Hydrometer) method, which is the

most commonly used procedure for texture analysis [1, 2]. Soil is a basic component of the environment and its texture is a key aspect of environmental regulation as it determines the movement of pollutants from one area to another. Particle size distribution (soil texture) has become a fundamental parameter in pedological, agronomic and environmental studies therefore its analysis, calculation and interpretation is paramount. It can be determined qualitatively by the feel method and quantitatively using a myriad of methods. The feel method is basically an estimation of the proportions of the separates based on the feeling of a moist soil between fingers [3, 4]. In this method, organic matter is not removed therefore, this could be a source of error. The Pipette method is precise and reproducible [5] albeit being time consuming and therefore not suitable for routine analysis [6].

Agricultural applications of soil texture includes the determination of crop suitability and prediction of the response of soil to environmental and management conditions including drought or calcium carbonate requirements [7]. Particle size distribution is useful for the characterization of a land's suitability for various agricultural, geotechnical, landscaping and reclamation purposes. The chemical relationships within soils are influenced by the petrography, clay mineralogical makeup and the proportions of sand, silt, and clay which also have important influence on the soil structure [8]. Soil texture affects the soils' water and nutrients holding capacities whereby fine textured soils generally have a higher capacity to retain water, whilst sandy soils have more capacity to leach owing to its large pores. The texture is not readily subject to change due to farm practices therefore it is considered a permanent soil attribute [9]. There exists a direct relationship between the particle size distribution and other soil characteristics including the shear induced volume change, porosity, saturated hydraulic conductivity and nutrient retention [10]. The USDA Soil Taxonomy and the WRB systems of soil classification have 12 textural classes based on percent sand, silt, and clay in the soil [11]. The USDA soil textural triangle is used to assign a sample its texture class after calculations. A Soil Texture Wizard has been appended in R statistical functions to classify and to transform soil texture data.

The Bouyoucos method was first invented in 1927 [12] as a method for mechanical analysis of soils. Bouyoucos slightly modified it in 1928 and 1929 then compared it with the Pipette method in 1934. An improved protocol was published in 1936 [13] before recalibrating the method in 1951 [14]. His final publication [15] is the procedure used by most research organizations to date. The 1962 dispersing technique consisted of soaking the soils in 5% of calgon overnight or 15 to 20 hours, and dispersing them for 2 minutes only by the dispersing machine running at a speed of 16000 revolutions per minute.

Different other modifications have been suggested by various researchers aiming to make the hydrometer method more accurate [16–19, 11]. These modifications revolve around how the soil sample is pre-treated before analysis, concentrations of the dispersing solution and the timing for the first and second hydrometer readings. The Bouyoucos method is based on Stoke's law of sedimentation [20] which relates the particle size and the rate of sedimentation in a water column. The Stokes' law assumes that fluid flow around a particle is in the creeping flow regime, that particles are rigid and spherical and that the hydrodynamic interactions among particles in the suspension are neglected. In the hydrometer method, the size of the solids in the suspension is estimated from the density of the solution using the

hydrometer. The Pipette method and the Bouyoucos methods differ in pretreatment of the samples before sedimentation whereby the Pipette method recommends destruction of organic matter using hydrogen peroxide whilst the Bouyoucos method does not recommend this pretreatment [15]. The clay fraction is read after two hours of sedimentation using the Bouyoucos method [6].

The Bouyoucos method uses calgon as a dispersant to separate the soil aggregates. The effectiveness of the calgon is improved by shaking on a mechanical reciprocal shaker or by stirring using a high speed electric stirrer. A blank having water and the dispersing agent is used to calibrate the hydrometer readings, to correct for variation in solution density and is subtracted from each hydrometer reading. The first reading which represents clay and silt is used to calculate percent sand that has already settled and is taken after 40 seconds of agitation. The second reading is taken after 2 hours of settling without agitation and used to calculate percent clay while the silt is calculated from percentages of sand and clay. The use of the ASTM 152H-Type hydrometer is based on a standard temperature of 68°F (20°C) and a particle density of  $2.65 \text{ g cm}^{-3}$  and units are expressed as grams of soil per liter. The sand fraction may be quantified by sieving the soil sample through a  $53 \mu$  sieve as suggested by [17]. The objective of this study was to compare the sand, silt and clay contents measured using different treatments revolving around the Bouyoucos technique and determine the treatment combination that correlates with fractions obtained through the accurate Pipette method. This acted as a basis to propose a modification to the rapid Bouyoucos technique.

## Materials And Methods

### Analytical Procedure

Experiments were done at the University of Nairobi, Upper Kabete Campus, in Soil Physics and Water Management Laboratory. Samples were air dried and passed through two millimeter sieve. The soil samples were analyzed under different treatments modified from the Bouyoucos method. Selected samples had been collected from the top, eluvial soil horizons of profile pits opened in Upper Kabete Campus field, University of Nairobi and classified as Mollic Nitisols (WRB); Very fine, mixed, isothermic Oxic Humiustalf (Soil Taxonomy) [21]. Fifty (50) grams of air dry samples was placed in beakers and successive aliquots of hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) slowly added to the 8<sup>th</sup> day when effervescence stopped, except for samples digested for 5 days or samples that did not undergo pre-treatment. The  $\text{H}_2\text{O}_2$  was added to remove organic matter from the soil samples. There was initially 10 large samples whose subsamples were used for the treatments. Unless otherwise specified, analytical procedure involved digesting the samples for 10 days using 35%  $\text{H}_2\text{O}_2$  until when no more effervescence could be observed, dispersed with 50 ml of 10% calgon, stirred on high speed electric stirrer for 3 minutes and inverted to agitate before taking the first hydrometer reading. The first hydrometer reading was taken after 40 seconds of agitation while the second hydrometer reading was taken after 2 hours except for one batch where the readings were taken again after 60 seconds and after 6 hours respectively. Temperature readings were taken alongside the hydrometer readings for correction of variation in temperature between

the samples and the hydrometer calibration temperature. A blank having water and the dispersing agent was used to calibrate the hydrometer readings. Three (3) drops of pentan-1-ol (amyl alcohol) solution was added to the suspension after agitation to knock off frothing. Digestion is used to connote removal of organic matter using H<sub>2</sub>O<sub>2</sub>.

### **Experimental Treatments**

Ten (10) subsamples were digested for 10 days (10Days Digestion) and other 10 samples for 5 days (5Days Digestion). Other 10 subsamples were digested using 40% H<sub>2</sub>O<sub>2</sub> (40%H<sub>2</sub>O<sub>2</sub>) and other 10 using 100% H<sub>2</sub>O<sub>2</sub> (100%H<sub>2</sub>O<sub>2</sub>). Other 10 subsamples were analyzed without pretreatment with H<sub>2</sub>O<sub>2</sub> (No Digestion). Other 10 subsamples were dispersed using 4% calgon (4%Calgon). Other 10 subsamples were shaken for 6 hours on reciprocating shaker (Shaking). Other 10 subsamples were shaken for 6 hours on reciprocating shaker and stirred for 3 minutes (Shaking+Stirring). The inversion method of agitation was compared with plunging (Plunger) the samples using one of the selected above treatments; the number of inversions and plunger strokes was maintained constant at 10. Forty (40) seconds versus 60 seconds for first hydrometer reading and 2 hours versus 6 hours (60sec6hrs) for the second hydrometer reading were compared using one of the selected above treatments. The feel method was used to determine the texture class of the samples by observing the nature of the soil when wet. One batch of samples was analyzed for particle size distribution using the Pipette method.

### **Temperature corrections**

Temperature correction was done in all samples whose temperatures deviated from 20° Celsius (68° Fahrenheit). This is the temperature at which the used ASTM (E100) 152H soil hydrometer has been calibrated at. Temperature corrections for hydrometer readings are given in the proposed protocol.

### **Calculations for the hydrometer method**

The percentage proportions of sand, silt and clay in each sample were calculated as follows: *Percent Sand = (50-(H1-Br)/50\*100..... (Equation 1)*

*Percent Clay = H2-Br/50\*100..... (Equation 2)*

*Percent Silt = 100% - (%Sand + %Clay)..... (Equation 3)*

Where: 50 = Amount of soil sample weighed in grams; H1 = Temperature corrected first hydrometer reading after 40 seconds of agitation and H2 = Temperature corrected second hydrometer reading after 2 hours of settling, Br = Temperature corrected blank reading.

### **The Pipette method**

Ten (10) grams of air dry soil samples that had passed through 2 mm sieve was placed in a beaker and digested using H<sub>2</sub>O<sub>2</sub> to burn the organic matter. Fifty (50) ml of 10% calgon solution was added into the

sample and shaken for 6 hours to facilitate the dispersion of individual particles. The suspension was then sieved through a 53 $\mu$  sieve whereby the retained proportion was washed into a beaker, decanted and oven dried at 105°C for 24 hours then gravimetrically quantified as sand. Suspension that passed through the sieve and consisted of particles of silt, clay and the dispersant was transferred in a 500 ml measuring cylinder. The sample was agitated by inverting 10 times. A pipette was used to extract 10 ml of the suspension in the upper 10 cm from the surface after 4.5 minutes, 50 minutes and 7 hours 36 minutes of settling at suspension temperature of 21°C. The samples taken by the pipette were transferred to beakers of known weights and oven dried for at 105° C for 48 hours. The samples taken after 7 hours and 36 minutes were containing clay and the dispersing agent only and was calculated according to the following equation:

..... (Equation 4)

Where: Dry weight = Oven dry weight of the sample; Pipette volume = 10 ml; Weight of dispersing agent in 500 ml suspension = 0.01 g; Volume of suspension = 500 ml; Weight of soil = 10 g.

### Data analysis

Data analysis was done using Genstat 14<sup>th</sup> Edition and IBM SPSS. Means were separated using the Least Significant Difference (LSD) through Analysis of Variance (ANOVA) in Genstat. Mean differences greater than the LSD was indicative of significant difference between the treatments. The more accurate Pipette method was used as a basis for comparison of the percent sand, silt and clay by the various Bouyoucos treatments through bivariate correlation in SPSS. This was aimed to identify the treatment combination that was nearest to the Pipette technique and for basis of recommendation of more accurate protocol. The error in measurements of percent sand, silt and clay was considered to be the differences in the measurements obtained using the hydrometer treatments and the Pipette method.

## Results And Discussions

### The clay samples

All samples done by the Pipette method were classified as clay with little proportion of sand. In the 100% $H_2O_2$  treatment, 100% (10) of the samples was also classified as clay. In the 60sec6hrs treatment, in which 60 seconds and 6 hours represents the time of first and second hydrometer reading respectively, 90% of the samples (9) was classified as clay whilst 10% was clay loam. In the 40% $H_2O_2$  treatment, 60% (6) of the samples was classified as clay, with 30% (3) being sandy clay and 10% (1) being sandy clay loam. In the 10Days Digestion treatment with 35%  $H_2O_2$ , only 40% (4 samples) was classified as clay, with the rest being coarser, having 10% clay loam, 20 % sandy clay and 30% sandy clay loam. In 5Days Digestion treatment, none of the samples was clay, with 100% (10) of the samples classified as sandy clay loam. In the 4%Calgon treatment, none of the samples was clay, with 40% being loam and 60% sandy clay loam. In samples where organic matter was not removed, none of the samples qualified as clay, with 50% being sandy clay loam and 50% qualifying as sandy clay. These observations are

indicative of the importance of using of hydrogen peroxide to remove organic matter before soil texture analysis. This suggestion can be attributed to the fact that organic matter is a coagulating agent having cementing properties that entangles clay particles. Using  $H_2O_2$  is therefore recommended as sample pre-treatment to avoid underestimation of the clay proportion. It also lucidly exposes the need to use 10% instead of 5% calgon proposed in the Bouyoucos procedure. The importance of removal of organic matter was also supported by [22].

### **Removal of Organic Matter**

Results of this study show the importance of removing organic matter as an important pre-treatment before soil texture analysis. It should be noted, however, that the digestion should be complete as incomplete destruction of organic matter led to underestimation of the clay proportion. This is shown by the values of 5 days' treatment with hydrogen peroxide (Table 3). The concentration of hydrogen peroxide seems to affect the proportions of sand, silt and clay with samples digested using 100%  $H_2O_2$  having the highest proportion of clay content. Compared to values obtained from the Pipette method, 100% hydrogen peroxide is recommended except when shaking instead of stirring is done, when 35% hydrogen peroxide can be used for digestion. [14] and [15] suggested that particle size distribution may for practical purposes, be characterized by analysis done on whole soil and that, if desired, lime and organic matter can be quantified separately. The time of digestion of the sample is also contributing to differences in the proportions of the texture components as shown by the results of 10Days versus 5Days Digestion. Samples digested for longer period of time showed a higher clay content (Table 3) which can be attributed to complete destruction of the clay-cementing organic material leading to release of entangled clay. This observation leads to the conclusion that, in the interest of accuracy, samples should be subjected to complete digestion by increasing days of digestion or increasing the concentration of hydrogen peroxide. This finding is consistent with the observations of [22] who reported that failure to remove organic matter underestimated the clay proportion.

### **Correcting the Raw Hydrometer Readings**

The actual blank reading ( $Br$ ) was made in the dispersing solution (with no soil) at the same temperature as that of the soil suspensions according to [23] and [17]. A blank sample with water only measured 1g/L, with 10% calgon measured 5g/L while with 4% calgon measured 2.5g/L. These densities are indicative of the importance of taking away blank readings from temperature corrected hydrometer readings to increase the accuracy of particle estimates. This observation supports the view that blank-adjusted readings indicate with sufficient accuracy the density  $D$  (in  $gL^{-1}$ ) of suspended solids according to the equation:

$$D = Hr - Br \dots\dots\dots (Equation 5)$$

Where:  $D$  = Density in  $gL^{-1}$ ,  $Hr$  = Temperature corrected hydrometer reading,  $Br$  = Blank reading at the same temperature as the sample.

Temperature corrections were done for hydrometer readings whose respective temperatures deviated from 20°C. The practice of subtracting an actual  $Br$  from  $Hr$  was aimed to offset any discrepancies in environmental conditions between the lab and the hydrometer calibration.

### **Sand Reading**

There was a linear relationship for the batch of 10 samples selected for the Pipette method and samples under Shaking treatment (Table 1). Percent sand in the Pipette method was determined gravimetrically using the particles retained on a 53 $\mu$  sieve whereas in Shaking treatment, the sand fraction was calculated from blank-adjusted 40 seconds hydrometer readings ( $Hr_{40s}$ ) in 1000 ml suspensions (Equation 1). The near 1:1 relationship obtained ( $r=0.862$ ) supports the empirical choice of 40 seconds for the sand reading time (Table 1).

### **Time of Clay Reading**

Clay content obtained after 6 hours (6sec6hrs) was lesser than that taken after 2 hours (100%  $H_2O_2$ ) as shown in Table 3 but exhibited a significant linear correlation ( $r = 0.957$ ; P-value = 0.000). Using a 6-hour reading could have removed any bias in percentage clay associated with a 2 hour reading as noted by [24]. However, taking the second hydrometer reading after 6 hours may be practical for researchers having relatively few samples but impractical for students' demonstration in a lab whereby practical lectures run for at most 3 hours using already pre-treated samples or in the case whereby a researcher has many samples. In a real laboratory environment, whereby clients need soil analysis results at close intervals, a 2-hour reading is recommended.

### **Comparison of the Pipette method with Bouyoucos treatments**

Percent sand obtained through the Pipette method significantly correlated with that obtained through Shaking treatment at the 0.01 level ( $r = 0.862$ ; P-value = 0.001) and that obtained through Shaking+Stirring at the 0.05 level ( $r = 0.737$ ; P-value = 0.015) as shown in Table 1. This observation can be attributed to the extended time of vigorous shaking on a reciprocal shaker that could have completely dispersed the sample into individual constituent aggregates. It is indicative of the importance of careful consideration of the duration and the magnitude of shaking of the soil samples to enhance the action of sodium hexametaphosphate. The sand measurements made using the hydrometer treatments overestimated the sand fraction in the soil samples. This finding is consistent with the observations of [25] and [19]. Differences in the procedures for destroying soil organic matter and the dispersion of the samples between the Pipette method and the Bouyoucos treatments could have significantly affected the quantity of sand. Therefore, the observed differences can be attributed to variation in analytical procedures. [6] also reported a similar finding of overestimated sand by the Bouyoucos method compared to the Pipette method. In this study, Bouyoucos treatments overestimated the sand proportion by between 25.63 to 47.23%. This observation is consistent with findings of [25] who reported a 9.69% sand overestimation when estimating the sand proportion in 29 samples from the Andean region using the Bouyoucos method.

Table 1: Means of percent sand and correlation with the Pipette method

Treatment	Mean		Std. Deviation	Coe. Correl.
Pipette	7.57	a	2.229	1.000
Shaking+Stirring	33.2	b	2.86	0.737*
Shaking	34.2	b	2.741	0.862**
100%H <sub>2</sub> O <sub>2</sub>	36.8	c	1.033	0.127
60sec6hrs	38.8	c	1.033	0.127
Plunger	42.8	d	1.932	-0.397
40%H <sub>2</sub> O <sub>2</sub>	43.6	de	3.098	0.267
10Days Digestion	44	de	2.667	-0.366
4%Calgon	45.6	e	0.843	0.401
No Digestion	48.8	f	1.033	0.053
5Days Digestion	54.8	g	4.638	-0.122
Overall p-value = <0.001				
Least Significant Difference (LSD) = 2.126				
**Correlation is significant at the 0.01 level (2-tailed).				
*Correlation is significant at the 0.05 level (2-tailed).				

Where Correl. Coe. = Correlation coefficient (r) with the Pipette method.

In terms of percentage silt, none of the treatments correlated significantly with the pipette method with the highest correlation observed in the 10Days Digestion treatment ( $r = 0.512$ ;  $p\text{-value} = 0.130$ ). This finding indicative that none of the Bouyoucos treatments estimated the percentage silt with sufficient accuracy. The mean of percent silt obtained through the Pipette method is statistically significant ( $P\text{-value} = <0.001$ ) compared to the Bouyoucos treatments (Table 2). The silt fraction is, however, determined through calculation using percent clay and sand therefore more emphasis should be put on accurate determination of sand and clay. Since all the Bouyoucos treatments evaluated in this study estimated the silt concentration as the difference between 100 percent and the percent sand and clay (Equation 3), any analytical errors would impact the estimation of the silt content when determining these two fractions. This finding is consistent with the observations of [22] who reported that the silt fraction was systematically overestimated when soil organic matter was not removed. The average silt content determined using the Bouyoucos methods was 6.89% ( $P = <0.001$ ) lesser than that determined using the Pipette method. This observation can mathematically be attributed to overestimation of sand fraction

accentuate in the hydrometer method. It is consistent with the findings of [26] who observed a 9.58% silt underestimation when comparing the Bouyoucos method with the Pipette method.

Table 2: Means of percent silt and correlation with the Pipette method

Treatment	Mean		Std. Deviation	Correl. Coe.
Shaking	9.2	a	2.86	0.279
Shaking+Stirring	9.2	a	1.687	-0.005
40%H <sub>2</sub> O <sub>2</sub>	14.6	b	2.503	0.489
No Digestion	14.8	b	4.237	-0.221
100%H <sub>2</sub> O <sub>2</sub>	16	b	2.981	-0.358
60sec6hrs	18.8	c	2.86	-0.229
10Days Digestion	19.4	cd	3.134	0.512
Plunger	20.6	cd	3.273	0.511
5Days Digestion	22	de	3.651	0.026
Pipette	23.93	ef	3.751	1.000
4%Calgon	25.8	f	3.19	-0.435
Overall p-value = <0.001				
Least Significant Difference (LSD) = 2.665				

Where Correl. Coe. = Correlation coefficient (r) with the Pipette method.

There was a positive correlation in the clay proportion between the Pipette method and Shaking+Stirring treatment ( $r = 0.644$ ,  $P\text{-value} = 0.044$ ) at the 0.01 level. Shaking treatment was not significantly correlated with the Pipette method ( $r = 0.577$ ). Treatments involving shaking estimated the clay proportion with sufficient accuracy, which can be attributed to enhanced dispersion and conversion of the relatively resistant, moderately coarse and coarse material to finer proportions on complete dispersion. This calls for increase in the amount of time if the samples are stirred instead of shaking and also consideration of the stirrer revolutions per minute (r.p.m) which should be at least 16000. [15] suggested the use of a mixer running at a speed of about 16000 r.p.m for 2 minutes. There was a negative correlation between the clay proportion obtained using the Pipette method and No Digestion treatment ( $r = -0.234$ ). This finding can be attributed to entanglement of the clay particles by organic matter that could have cemented some clay particles owing to its coagulating properties, preventing its breakdown and resulting to clay underestimation in undigested samples. This necessitates the pre-treatment of the soil samples to remove organic matter before texture analysis. This observation is consistent with the findings of [22], who reported underestimation of the clay content in samples where organic matter was not removed. The

4%Calgon treatment was negatively correlated with the Pipette method ( $r = -0.712$ ; P-value = 0.021) at the 0.05 level which can be attributed to the low concentration of the dispersant that could have led to incomplete dispersion. It is therefore recommended to use 10% sodium hexametaphosphate as the dispersing agent during soil texture analysis. This finding is consistent with the observations of [17] and [22] who also reported clay underestimation by the Bouyoucos method.

[25] reported that the Bouyoucos method did not differ from the sieve, even without the destruction of the Soil Organic Matter (SOM) in the samples. Those results can, however, be attributed to very low SOM concentration in the samples analyzed by the researchers. [26] determined a minor difference in clay content obtained using the Hydrometer method in comparison to that obtained using the Pipette method when the soil samples were pre-treated to destroy the soil organic matter. SOM acts as a cementing agent and could have bound clay particles together into groups that would precipitate more rapidly than individual particles and could thus be quantified as silt. This finding is consistent with the observations of [6] who recommended the need for complete digestion of soil samples. This finding can also explain why samples digested for 5 days underestimated the clay fraction (Table 3). The underestimation of clay can be attributed to incomplete dispersion of soil aggregates. Silt-sized micro-aggregates composed of organic matter-clay complexes could have settled faster and quantified as silt instead of clay. This suggestion is consistent with that of [27] and [22] who suggested a possibility of flocculation after dispersion of soil samples which would classify them as silt.

Table 3: Means of percent clay and correlation with the Pipette method

Treatment	Mean		Std. Deviation	Correl. Coe.
5Days Digestion	23.2	a	1.687	0.015
4%Calgon	28.6	b	2.675	-0.712*
No Digestion	36.4	c	4.3	-0.234
10Days Digestion	36.6	c	4.006	0.482
Plunger	36.6	c	4.006	0.482
40%H <sub>2</sub> O <sub>2</sub>	41.8	d	3.458	0.238
60sec6hrs	42.4	d	3.502	-0.081
100%H <sub>2</sub> O <sub>2</sub>	47.2	e	3.553	-0.21
Shaking	56.6	f	3.658	0.577
Shaking+Stirring	57.6	f	3.239	0.644*
Pipette	68.5	g	4.314	1.000
Overall p-value = <0.001				
Least Significant Difference (LSD) = 2.707				
*Correlation is significant at the 0.05 level (2-tailed).				

Where Correl. Coe. = Correlation coefficient (r) with the Pipette method.

Inter-treatment correlations were also done and significant relationships discussed, aiming to identify treatments that can be substituted for others and still give the same accuracy and rapidity. In terms of percent sand, 100% H<sub>2</sub>O<sub>2</sub> treatment correlated perfectly with 60sec6hrs (r = 1; P-value = 0.000) at the 0.01 level. This can be attributed to the fact that the samples treated with 100% hydrogen peroxide and readings taken after 40 seconds and 2 hours respectively are the same samples used for the 60 seconds and 6-hour readings. Percent sand from 10Days Digestion had a positive correlation with the Plunger (r = 0.949; P-value = 0.000) at the 0.01 level. This finding demonstrated that before inserting the hydrometer, agitation can be done through inversion or through plunging as long as the latter does not induce circular movements within the column as this may affect the settling velocity of the sand particles. Compared to the pipette method, the magnitude of error for the batch agitated using the plunger (40%H<sub>2</sub>O<sub>2</sub>) and the same batch by inversion was 35.23% and 36.03% respectively. The lesser sand when using the plunger can be attributed to the circular movements in the suspension that could have been induced by the plunger therefore slowing the settling velocity of the sand particles. Shaking and Shaking+Stirring also correlated at 0.01 level (r = 0.817; P-value = 0.004). This can be attributed to the similar process the two treatments underwent. Shaking+Stirring had lesser sand fraction indicating that the more the dispersion, the lesser the proportion of the coarse fraction in the sample.

The silt fraction also demonstrated significant correlations among the treatments. The proportion obtained from 100% $H_2O_2$  treatment correlated with that obtained in 60sec6hrs treatment ( $r = 0.938$ ; P-value = 0.000) and that obtained with No Digestion treatment ( $r = 0.844$ ; P-value = 0.002), all at the 0.01 level of significance. The silt percentage obtained through 60sec6hrs also correlated with that obtained through No Digestion ( $r=0.895$ ; P-value=0.000). These observations indicate that pre-treatment does not influence the silt proportion, despite the silt being affected by the proportions of sand and clay, with the latter being directly influenced by pretreatment of the sample with hydrogen peroxide. Silt from 10Days Digestion that used 10% calgon negatively correlated with the fraction obtained through 4%Calgon treatment, with the latter having more silt ( $r = -0.769$ ; P-value = 0.009) at the 0.01 level. This can be attributed to decreased dispersion in 4%Calgon, resulting to higher proportion of fractions coarser than clay. This observation is indicative that the lesser the concentration of the dispersant, the more the overestimation of the moderately coarse fraction. The silt fraction obtained from 10Days Digestion also correlated with that obtained through use of plunger ( $r = 0.949$ ; P-value = 0.000) at the 0.01 level which could be due to similar treatments in digestion and dispersion. Silt from 4%Calgon negatively correlated with that from use of Plunger ( $r = -0.839$ ; P-value = 0.002) at the 0.01 level of significance. This can be attributed to differences in the concentration of the dispersing reagent.

The clay proportion obtained through 100% $H_2O_2$  significantly correlated with that obtained through 60sec6hrs ( $r = 0.957$ ; P-value = 0.000) and that obtained through No Digestion ( $r = 0.925$ ; P-value = 0.000) at the 0.01 level. It should, however, be noted that the No Digestion treatment has the least mean among the three treatments therefore necessitating pretreatment so as not to underestimate the clay. The observation also demonstrated that the clay fraction obtained after 2 hours of settling is sufficiently reflecting the amount of clay in the sample and there is no need of waiting for 6 hours. Clay obtained through 60sec6hrs also significantly correlates with the fraction obtained via No Digestion ( $r = 0.933$ ; P-value = 0.000) at the 0.01 level. This can be attributed to the fact that No Digestion treatment could have underestimated the clay fraction and its correlation with 60sec6hrs is not counterintuitive given the functionality of the ASTM soil hydrometer. Since the hydrometer reading is a reflection of what is suspended, there is a possibility that after 6 hours of settling, some clay particles could have settled and therefore not reflected by the hydrometer as would be the case with a 2 hour reading. This scenario could have led to underestimation of the clay to some extent.

Percentage clay obtained through 10Days Digestion correlates with that obtained through 4%Calgon ( $r = -0.701$ ; P-value = 0.024) at the 0.05 level, that obtained through Shaking ( $r=0.883$ ; P-value=0.001), that obtained through Shaking +Stirring ( $r = 0.774$ ; P-value = 0.009) and that obtained through Plunger ( $r = 1$ ; P-value = 0.000) at the 0.01 level of significance. This observation is indicative that for rapidity purposes especially for a practical lecture, samples digested for 10 days can be used to offer sufficient clay percentage with stirring for 3 minutes instead of the lengthy 6-hour shaking process. The negative correlation with 4%Calgon indicates that 10% calgon should be adopted as the conventional concentration of the soil dispersant to avoid underestimation of the clay. The clay fraction obtained by 4%Calgon significantly correlates with that obtained through Shaking ( $r = -0.768$ ; P-value = -0.010) and

that obtained through Shaking+Stirring ( $r = -0.841$ ;  $P\text{-value} = 0.002$ ) at 0.01 level. This can be attributed to the reduced concentration of sodium hexametaphosphate. It also correlates with that obtained through Plunger ( $r=-0.701$ ;  $P\text{-value}=0.024$ ) at 0.05 level. The values of clay obtained through Shaking correlated significantly with those obtained through Shaking+Stirring ( $r = 0.961$ ;  $P\text{-value} = 0.000$ ). This finding indicates that the values of clay obtained through shaking alone can give satisfactory clay percentages without necessarily stirring the sample after shaking. The use of plunger may not be of direct importance for the clay fraction because there is no agitation prior to second hydrometer reading that reflects clay.

When considering the magnitude of error in the proportions of sand and clay obtained from the hydrometer treatments and those from the standard Pipette method, Shaking+Stirring, Shaking and 100% $H_2O_2$  treatments have the least magnitude of error (Table 1 and Table 3). These three treatments would therefore present the smallest analytical error when compared to the particle size distribution obtained through the Pipette method. These observations are consistent with the findings of [19] who suggested that the hydrometer can be used instead of the pipette method only in cases where the pre-treatment of the sample completely destroys the SOM and a total dispersion of the sample is achieved.

## Conclusions

Soil properties including particle size distribution determines the workability of the soil which is in turn used for assessment of the suitability of the land for agricultural purposes [28]. Its precise determination is therefore critical. The validity of equations 1, 2 and 3 is unlikely to be affected by minor differences in design of measuring cylinders provided that corrections for density and temperatures are done. However, errors in estimates of particle fractions of sample weight can be caused by variation in the amount or type of dispersing chemical used, using suspension volumes other than 1000 ml, using inappropriate reading times, or applying a temperature correction to the hydrometer reading in addition to an actual blank adjustment. Temperature readings should be undertaken alongside blank correction for viscosity to avoid discrepancies that may be caused by differences in settling due to temperatures different from hydrometer calibration league of 68°F (20°C). This is because based on the kinetic theory, higher temperatures would favor faster settling while lower temperatures would lower the settling velocity of particles. Digestion can take lesser days if the concentration of hydrogen peroxide is increased or if the samples are coarser than clay by the feel method. Hydrometer readings should be done as accurately as possible because for example when estimating the clay content, having used 50 g of soil, an error of  $\pm 1$  g/L hydrometer reading would result to an error of  $\pm 2\%$  clay (Eq. 1). The feel method characterized the soils commensurate to soil classification of the area. It should, however, be done with caveats especially in highly aggregated, stable clay soils as they behave like coarse sands in terms of water infiltration. They may therefore be identified in the field as sands or coarse loams and should be classified in a much finer category than they appear insitu. The choice of either glass or plastic cylinders is at the discretion of the researcher. However, glass cylinders are suggested due to their increased visibility while taking hydrometer and temperature readings.

Further research on this line may focus on evaluating magnitude of error in soil fractions when the meniscus and temperature are not corrected, when using different types of pestle to break the soil agglomerates for example by comparing rubber coated and ceramic pestle. A study may also compare the effectiveness of sodium hexametaphosphate against other dispersants, the effect of using larger sample and using more dispersing solution. In the following proposed protocol, the samples are digested with 100% H<sub>2</sub>O<sub>2</sub> until effervescence stops, dispersed with 10% sodium hexametaphosphate, stirred with a high speed electric stirrer for 3 minutes or shaken in a reciprocal shaker for 6 hours and inverted for agitation.

## The Mwendwa Protocol

### A Modified and Simplified Protocol for Soil Texture Analysis

#### Definition

Soil texture analysis – Also known as particle size distribution, is the determination of the percentage proportions of sand, silt and clay in a soil sample.

#### Principle

This method is based on Stoke's law of sedimentation of particles suspended in water. The law states that the differential settling velocity (difference in speed of settling) of a particle in a water column is directly proportional the square of its radius, gravitational acceleration, the difference between the density of the particle and the density of the fluid, but it is inversely proportional to the viscosity (resistance to flow) of the fluid. Increasing temperatures would therefore reduce the fluid viscosity and consequently increase the settling velocity of the particles. Decreasing temperatures would increase the fluid viscosity and reduce the settling velocity of the particles. Temperature corrections of hydrometer readings is therefore of essence.

$$V=d/t=..... \text{.(Equation 6)}$$

Where:  $V$  = settling velocity;  $d$  = distance;  $t$  = time;  $r$  = radius of the particle;  $g$  = gravitational force ( $9.81\text{Nkg}^{-1}$ );  $d_1$  = density of the particle ( $2650\text{kgm}^{-3}$ );  $d_2$  = density of the fluid ( $1000\text{kgm}^{-3}$ );  $n$  = viscosity of the fluid = ( $0.001\text{Nsm}^{-2}$ ).

#### Apparatus and Materials

- (a). 1000 ml capacity glass measuring cylinders with corks. The palm can be used as cork.
- (b). Thermometer.
- (c). Standard soil hydrometer, ASTM (E100) 152H-Type with Bouyoucos scale in  $\text{gL}^{-1}$ .
- (d). High speed electric stirrer with a dispersing cup.

(e). Analytical weighing balance sensitive to  $\pm 0.01\text{g}$ .

(f). 500 ml plastic shaking bottles with caps.

(g). Reciprocating shaker.

## Reagents

1. 100% Hydrogen Peroxide ( $\text{H}_2\text{O}_2$ ).
2. 10% dispersing solution (calgon); Dissolve 100 g of Sodium hexametaphosphate ( $\text{Na}_6(\text{PO}_3)_6$ ) plus 21.3 g of Sodium carbonate in 1 liter using distilled water. The sodium carbonate buffers the acidity of calgon that would otherwise lead to clay destruction.
3. Distilled water.
4. Amyl alcohol (pentan-1-ol).

## Procedure

1. Weigh 50 g of soil that has passed through 2 mm sieve to an accuracy of  $\pm 0.01\text{g}$ .
2. Transfer to a beaker and add hydrogen peroxide until effervescence stops.
3. Add 50 ml of 10% calgon and transfer sample to a dispersing cup. Include a blank sample.
4. Stir for 3 minutes in the dispersing cup using high speed electric mixer.
5. Alternatively, for step 3 and 4, the sample can be transferred to a shaking bottle, tightly corked and shaken for 6 hours in a reciprocal shaker.
6. Transfer the suspension quantitatively from the dispersing cup or shaking bottle to a 1000 ml measuring cylinder.
7. Fill to the 1000 ml mark with water.
8. Agitate the sample through inverting 10 times after vigorous shaking then insert the hydrometer immediately.
9. Read the lower meniscus of the blank sample so as to correct the sample readings.
10. Add 2 to 3 drops of amyl alcohol in case of frothing to knock off the bubbles.
11. Take first hydrometer reading ( $\text{Hr}_1$ ) after 40 seconds. Take the temperature of the suspension.
12. Take the second hydrometer reading ( $\text{Hr}_2$ ) after 2 hours of settling (no agitation). Take the temperature of the suspension too.
13. Record the readings of hydrometer and temperature.
14. Correct hydrometer readings based on the temperature. Temperature corrections for hydrometer readings are calculated according to Equation 7. This equation is adapted from [29]. The value is subtracted from or added to the hydrometer readings (Table 4).

*Temperature correction = (Temperature of sample -20)\*0.2 ..... (Equation 7).*

Where 20 = Hydrometer calibration temperature in degree Celsius.

Table 4: Temperature corrections for hydrometer readings

Temperature (°C)	Hydrometer reading correction factor (g/L)
15	-1.0
16	-0.8
17	-0.6
18	-0.4
19	-0.2
20	0.0
21	0.2
22	0.4
23	0.6
24	0.8
25	1.0

*Adapted from [29]*

### Calculations

$$\text{Percent Sand} = (50 - (H1 - Br)) / 50 * 100 \dots\dots\dots (\text{Equation 8})$$

$$\text{Percent Clay} = H2 - Br / 50 * 100 \dots\dots\dots (\text{Equation 9})$$

$$\text{Percent Silt} = 100\% - (\% \text{Sand} + \% \text{Clay}) \dots\dots\dots (\text{Equation 10})$$

Where: 50 = Amount of soil sample weighed in grams; *H1* = Temperature corrected first hydrometer reading and *H2* = Temperature corrected second hydrometer reading, *Br* = Blank reading at the same temperature as the sample suspension.

### Textural classification

Particle size distribution is reported as percentages of the mineral fraction namely the percent sand, silt and clay. Soil texture is based on the USDA textural triangle, physical or online.

## Declarations

### Ethics approval and consent to participate

This study meets all the ethical guidelines and adheres to legal requirements of my country.

### **Consent for publication**

Not applicable.

### **Availability of data and materials**

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

### **Competing interests**

Not applicable.

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### **Authors contributions**

S. M. M designed this study, did soil sampling and lab analysis, prepared, read and approved the final manuscript.

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