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The Effect of Laboratory Test Methods on Soil Particle Size Distribution Results

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ABSTRACT

Knowledge of the particle size distribution of soils (including clay content) is required in many geotechnical engineering analyses and designs, including in geoenvironmental engineering. In such cases, the premise is made that that the laboratory test grading results are correct. Engineers have relied on grading results which subsequently turned out to be incorrect with unacceptable design consequences. This research assesses the influence of test methods on the particle size distribution of soils. This was done by sending identical samples of five different soils to nine accredited commercial testing laboratories, in South Africa. The results indicated that the largest variations occurred in the size ranges that are determined using the hydrometer analysis (percentages finer than 0.075 mm, 0.005 mm and 0.002 mm). Furthermore, when considering the clay contents, the range in values from the nine laboratories increased (to a maximum of 23 %) in the case of the sample with the highest average clay content (maximum of 42 %). A similar positive correlation was observed when considering the standard deviations (of the clay contents), which increased (to a maximum of 7.5) with an increase in the average clay content of a soil. Furthermore, in the case of each soil, the values determined by each laboratory for each percentage finer size did not differ significantly from the mean (of that percentage finer size) in 90 % of the tests conducted. In conclusion, the dispersing agent type, concentration and dosage influenced the results of the test methods considered, resulting in an up to 79 % increase in the clay content results.

Keywords: Soil testing, particle size analysis, dispersing agents, hydrometer.

1 INTRODUCTION

The particle size distribution of a soil is an important assessment that is used in determining the suitability of a soil for various purposes. This includes determining the suitability of a soil as a construction material for various purposes or as a founding material. The accuracy of soil grading results is particularly relevant in assessing hydraulic conductivity (Boadu, 2000) and other properties as well as for soil classification. The clay content (percentage finer than 0.002 mm) for example, is used in various analyses and designs. The consequences of designs based on incorrect particle size distributions have often been unacceptable and even led to failures. Hence, the accurate determination of the particle size distribution of a soil is imperative.

In South Africa, the accuracy of particle size distributions of soils determined by laboratory tests has often been questioned, based on the predominant soil separates anticipated from observing the soil sample. Day and Jacobsz (2008) formally investigated the accuracy of particle size distributions determined by four laboratories for two representative soil samples. The results received from the laboratories were unacceptably variable, resulting in the possible incorrect assessment of the anticipated soil behaviour.

The inaccurate determination of particle size distributions by laboratories may be attributed to various reasons including uncalibrated or ageing test equipment, non-compliance with test methods, inadequately trained laboratory technicians and poor or absent quality control. In addition, identical methods are not used by all laboratories.

Most or all methods use a sieve analysis for the determination of the distribution of the coarser fraction (> 0.075 mm) and a dispersion and sedimentation procedure, based on Stokes Law, (utilising a hydrometer) for the determination of the particle distribution of the fines fraction (< 0.075 mm). The sedimentation procedure employs a dispersing agent. In addition, the dispersing agent specified by the different test methods may differ in type, concentration and volume.

This research specifically assessed the influence of test methods on the particle size distribution of soils. This was done by sending identical samples of five different soils to nine accredited commercial testing laboratories, located in the Province of Gauteng in South Africa, for the determination of their grading characteristics and statistically analysing the results.

2 EXPERIMENTAL DETAILS

2.1 Soil samples

The five soils were sampled from a study area which is located approximately 150 km north of the city of Rustenburg in South Africa, to the south of the confluence of the Crocodile and Marico Rivers. This area is bounded by Longitude 26° 40’ and 27° 15’ South Latitude 24° 05’ and 24° 40’ East.

The engineering soil classification, according to the Unified Soil Classification System (USCS), (USAEWES, 1960) and the World Reference Base (WRB) Classification System (IUSS-WRB 2015) as well as the soil types of the five samples are included in Table 1.

Table 1. *Soil classifications and soil types of the samples selected*

Sample No.	USCS Class	General correlation with World Reference Base (WRB) Classification System	Soil type
1	SM	Ferralsols and Arenosols	Ferrallitic and Fersiallitic (not characterised by the dominance of smectitic clay minerals)
2	CL		
3	SC	Luvisols	Fersiallitic (characterised by the dominance of smectitic clay minerals)
4	CH	Vertisols	Margallitic
5	MH		

2.2 Test methods

For the purposes of this research, each method was randomly allocated a method number (M1 to M5) and the dispersing agent used in each case was allocated a symbol (A to G), as shown in Table 2. More specific details in the form of reference to the methods and composition of the dispersing are indicated in Tables 3 and 4, respectively.

Table 2. *General details of laboratory methods*

Laboratory No.	Method	Dispersing Agent
1	M1	E
2	M1	F
3	M1	E
4	M1	C
5	M1	G
6	M2	A
7	M3	D
8	M4	B
9	M5	C

Table 3. *Details of methods of particle size analysis*

Method	Reference to method used
M1	The methods that were adopted for sieving and sedimentation procedures were Methods A1 and A6, respectively. (According to TMH1, 1986). Method A6 was modified with respect to the dispersing agent used and the times at which the hydrometer readings were taken.
M2	The methods adopted for sieving and sedimentation procedures were Methods A1 and A6, respectively. (According to TMH1, 1986).
M3	The methods adopted for sieving and sedimentation procedures were Tests 7(B) and 7(D), respectively. (According to British Standards 1377: 1975).
M4	The methods adopted for sieving and sedimentation procedures were according to Lambe (1951).
M5	The methods adopted for sieving and sedimentation procedures were Method A1 (according to TMH1, 1986) and CSIR CA17, respectively.

Table 4. *List of dispersing agents used*

Symbol	Dispersing agent	Preparation of stock solution
A	Five millilitres of sodium silicate and five millilitres of sodium oxalate.	Sodium silicate: Dissolve sodium silicate, preferably the waterglass solution (Na_2Si0_3), in distilled water until the solution yields a reading of 36 at a temperature of 20 ° C on the standard soil hydrometer. Sodium Oxalate: This consists of a filtered saturated solution of sodium oxalate ($Na_2C_20_4$).
B	One-hundred and twenty-five millilitres of four percent sodium hexametaphosphate.	Four percent sodium hexametaphosphate: Dissolve 40 grams of sodium hexametaphos-phate ($NaP0_3$) in a sufficient quantity of distilled water to bring the volume of the solution to one litre.
C	One-hundred and twenty-five millilitres of Calgon 35:7 solution.	Calgon 35:7: Mix 35 grams of sodium hexametaphosphate ($NaP0_3$) with seven grams of sodium carbonate ($NaC0_3$) and

		add a sufficient quantity of distilled water to bring the volume of the solution to one litre.
D	One-hundred and twenty-five millilitres of Calgon 33:7 solution.	Calgon 33:7: Mix 33 grams of sodium hexametaphosphate ($NaP0_3$) with seven grams of sodium carbonate ($NaC0_3$) and add a sufficient quantity of distilled water to bring the volume of the solution to one litre.
E	Twenty millilitres of sodium pyrophosphate.	Sodium pyrophosphate: Mix 36 grams of sodium pyrophosphate ($Na_4P_20_710H_2O$) with a sufficient quantity of distilled water to bring the volume of the solution to one litre.
F	Twenty millilitres of sodium tetrapyrophosphate.	Sodium tetrapyrophosphate: Mix 36 grams of sodium tetrapyrophosphate with a sufficient quantity of distilled water to bring the volume of the solution to one litre.
G	Forty milliliters of sodium silicate and 40 millilitres of di-sodium di-hydrogen pyrophosphate.	Di-sodium di-hydrogen pyrophosphate: Mix 36 grams of di-sodium di-hydrogen pyrophosphate ($Na_2H_2P_20_7$) with a sufficient quantity of distilled water to bring the volume of the solution to one litre. Sodium silicate: Add sodium silicate syrup (Na_2SiO_2) to distilled water until the solution yields a reading of 36 at a temperature of 19.5 ° C on the standard soil hydrometer.

At the time, one of the laboratories (No. 6) strictly adopted the South African test methods (TMH 1 Methods A1 for the sieve analysis and A6 for the hydrometer analysis). Five laboratories (No.1 to No. 5) employed the TMH 1 method with modifications to the dispersing agent prescribed by Method A6. The reasons for deviations from the dispersing agent prescribed by a method were not established.

Laboratory No. 4 also deviated from the method (M1) by not determining the clay content (percentage finer than 0.002 mm). Furthermore, Laboratory No. 7 used the BS 1377:1975, Laboratory No. 8 employed the method from Lambe (1951) and Laboratory No. 9 used a combination of Method A1 of TMH 1 with CSIR CA17.

It should be noted that, at the time, in South Africa, the officially adopted methods for sieving and sedimentation procedures were Methods A1 and A6, respectively. (According to TMH1, 1986). The TMH1 (1986) methods were subsequently replaced by SANS 3001 (2011) which has not been adopted by most laboratories. Almost all the national laboratories follow the superseded TMH1 (1986) method, with a deviation in the dispersing agent.

Differences between TMH1 (1986) and SANS 3001 (2011) include sample preparation procedures and the amending of the dispersing agent from five millilitres of sodium silicate and five millilitres of sodium oxalate to 125 millilitres of Calgon 35:7 and an adjustment in the hydrometer readings by subtracting the hydrometer readings obtained on a “blank” companion specimens to account for the effect of the dispersing agent. Incidentally, SANS 3001 (2011) was still found to be unsatisfactory for certain applications (Stott and Theron, 2015).

Furthermore, British Standards (BS) 1377: 1975 was replaced BS 1377 (1990). The later standard still prescribes Calgon 33:7 as a dispersing agent.

Internationally, there are at least four methods which recommend the use of Calgon as a dispersing agent. These are SANS 3001 (2011), BS 1377 (1990), IS 2720 (IS 1985), International Soil Reference and Information Centre (ISRIC, 2002).

3 RESULTS AND DISCUSSION

3.1 Particle size distribution curves

Figures 1 to 5 show the particle size distributions of the five samples as determined by the nine laboratories.

As the samples of each soil type were quartered and riffled, to obtain the representative samples that were sent to the laboratories, it was assumed that differences in results were not attributed to natural composition variability. Furthermore, the samples were not characterised by organic matter.

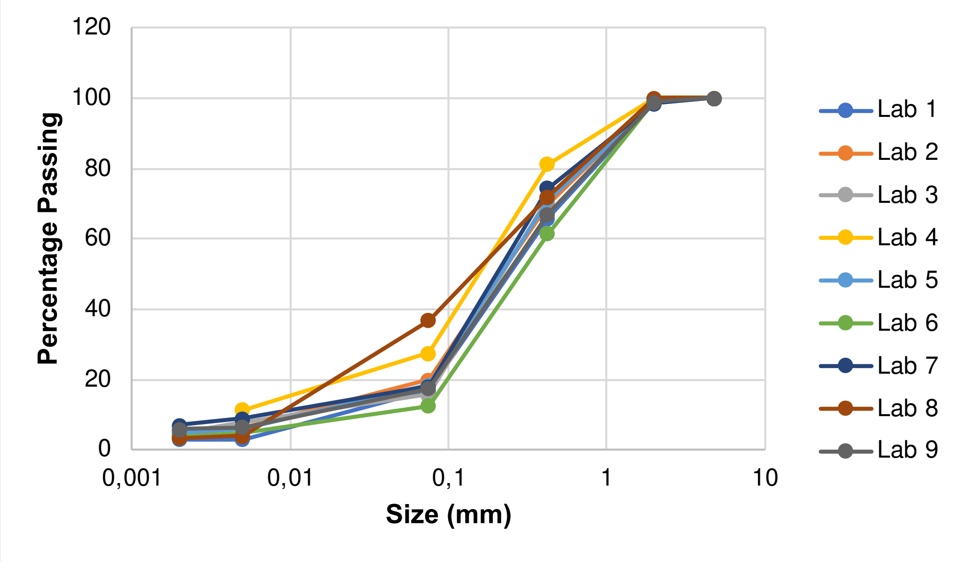

Figure 1. *Particle size distribution of sample 1*

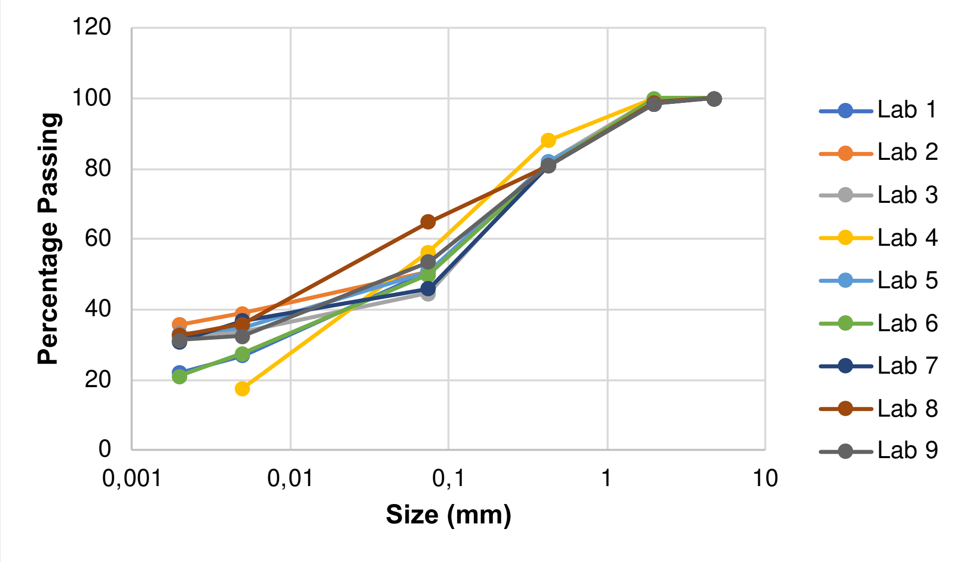

Figure 2. *Particle size distribution of sample 2*

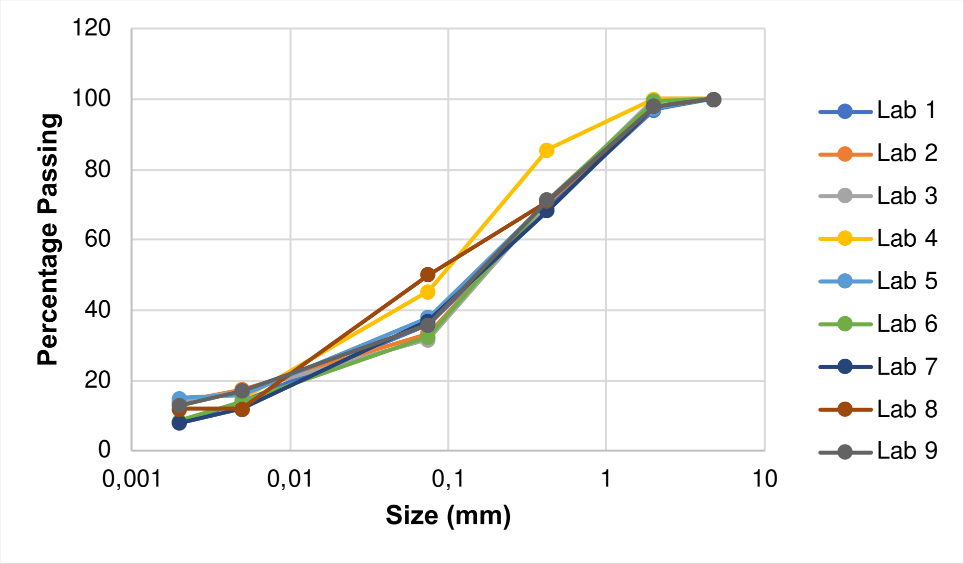

Figure 3. *Particle size distribution of sample 3*

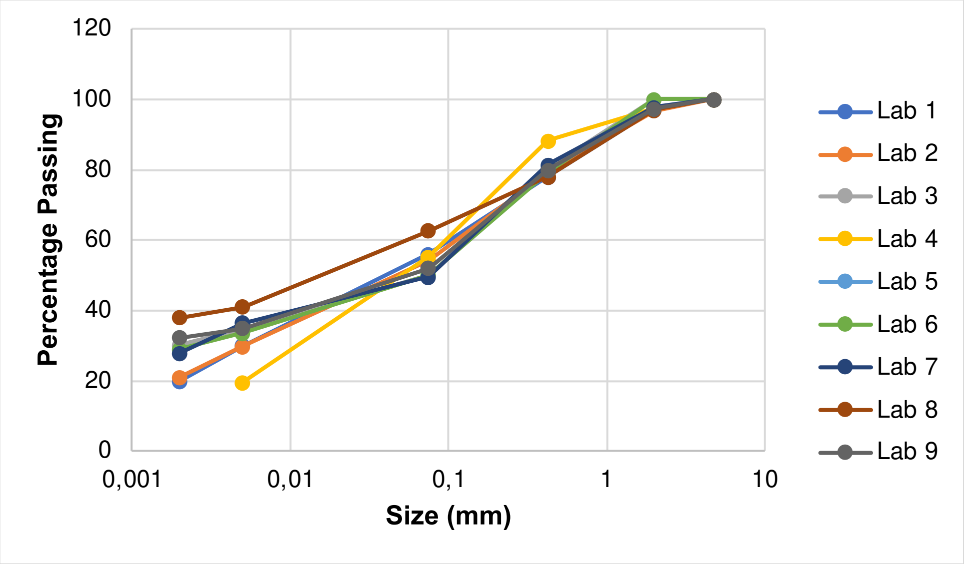

Figure 4. *Particle size distribution of sample 4*

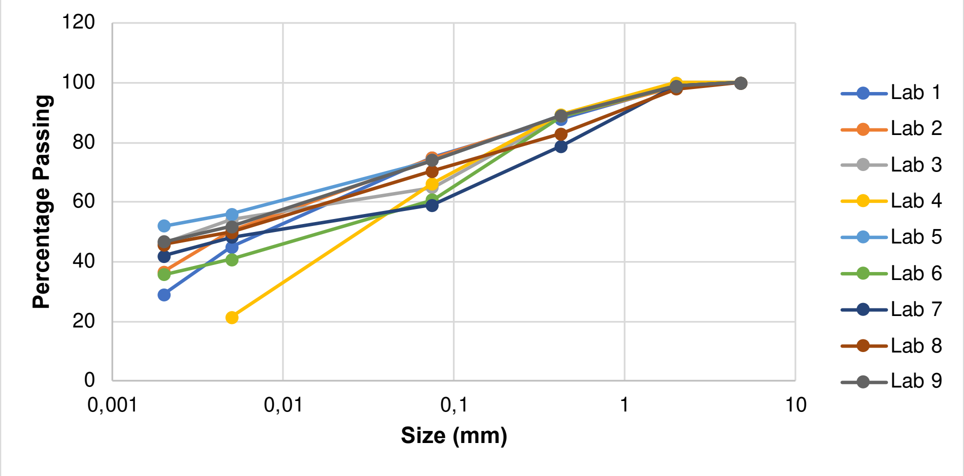

Figure 5. *Particle size distribution of sample 5*

With reference to the results shown in Figures 1 to 5, the following is evident.

- The largest variations in results occurred in the size ranges that are determined using the hydrometer analysis (percentages finer than 0.075 mm, 0.005 mm and 0.002 mm). This is expected to be due to the different dispersing agents used.
- In the samples with the relatively lower average clay contents (Sample 1 with 4.7 % clay and Sample 3 with 11.5 % clay) the percentage finer than 0.075 mm values exhibited the highest standard deviations. These were 7.4 and 6.1 for Samples 1 and 3, respectively.
- In the other three samples (2, 4 and 5), which respectively had clay contents of 29.9 %, 28.4 % and 41.8 %, the highest standard deviation occurred in the percentage finer than 0.005 mm. In samples 4 and 5, the standard deviations pertaining to the clay contents determined were the second highest.
- When considering the clay contents, the range in values from the nine laboratories increased (to a maximum of 23 %) in the case of the sample (No. 5) with the highest average clay content (maximum of 41.8 %). In addition, a similar positive correlation was observed when considering the standard deviations (of the clay contents), which increased (to a maximum of 7.5) with an increase in the average clay content. This trend is attributed to the effectiveness of the various dispersing agents.
- The five laboratories (1,2,3,5 and 6) using the South African Method and a dispersing agent of the (prescribed) sodium silicate combined with sodium oxalate (A) or Sodium pyrophosphate (E) or sodium tetrapyrophosphate (F) or sodium silicate combined with di-sodium di-hydrogen pyrophosphate (G), generally yielded similar grading results.
- Three laboratories (4,7 9) used different methods and a dispersing agent of Calgon (C or D). Two of these laboratories (7,9) yielded similar grading results to the five abovementioned laboratories (1,2,3,5 and 6) that all used the TMH1 (1986) method. However, the other (Laboratory 4) generally yielded relatively high percentages passing 0.425 and 0.075 and the lowest percentages finer than 0.005 mm. The slight difference in sodium hexametaphosphate ($NaP0_3$) content (in the Calgon) used by Laboratory 7 appeared to have no obvious effect. The differing of the results of Laboratory 4 from those of Laboratories 7 and 9 is probably attributable to factors that influence the testing other than the dispersing agent.
- Finally, Laboratory 8, which used sodium hexametaphosphate as a dispersing agent (B), yielded relatively high percentages passing 0.075 and generally in finer sizes in the samples with a higher clay content. This was the most effective dispersing agent.
- The dispersing is the most important factor influencing the particle size analysis.

The significant influence of the dispersing agent was confirmed in an investigation, by Kaur and Fanourakis (2016a) into the effect of type, concentration and volume of five dispersing agents on the magnitude of clay content (determined by the hydrometer analysis). Their investigation indicated that, in one sample analysed, the determined clay fraction varied from 1 % to 32 %, depending on the dispersing agent used. Furthermore, Calgon 33:7 was found to be the most effective dispersing agent. In addition, Sridharan et al (1991) who also conducted a similar study concluded that the clay-sized fraction can vary from 4% – 45% for marine clays, depending on the dispersing agent used, strictly following the IS (1985) method. It was further seen that 100 ml – 125 ml of Calgon 33:7 was found to be the most effective dispersing agent. Other researchers who found Calgon to be the most effective dispersing agent include Bindu and Ramabhadran (2010) as well as Emeka (2015). In a detailed investigation using various concentrations of Calgon, Kaur and Fanourakis (2018) established that a 4.2 % Calgon (35:7) concentration was the most effective. This was in agreement with Emeka (2015).

Detailed investigations into the effectiveness of different dispersing agents, in varying volumes and concentrations are discussed in the works of Kaur and Fanourakis (2016a,b,c) and Kaur and Fanourakis (2018).

It should be borne in mind that the TMH1 (1986) method does not incorporate a companion "blank" solution, comprising distilled water and dispersing agent (no soil), in a second bouyoucos cylinder, in the same proportions as the solutions prepared with the soil. Readings taken in the "blank" solution, at the relevant time period, would be subtracted from the hydrometer reading taken in the cylinder containing soil and the same dispersing agent, to account for the effect of the dispersing agent on the hydrometer readings. This correction makes a difference to the calculation of the percentages passing by inflating the fines (including the clay) contents (Kaur and Fanourakis, 2018). Hence, ideally all the national laboratories, which follow the TMH1(1986) method with a deviation in the dispersing agent type, should consider correcting the hydrometer test readings if this is not being done. SANS 3001 (2011) makes provision for this procedure.

3.2 Student's t-tests

Paired t-Tests were conducted on the percentages passing of the mean of the laboratory values and the actual value determined by each laboratory, for each sample (considering the percentages passing 4.75 mm, 2 mm, 0.425 mm, 0.075 mm, 0.005 mm and 0.002 mm) to determine the level of significance. The null hypothesis was assumed. The five percent significance level was decided upon.

The significance levels (P %) which are the probabilities of differences of such magnitudes arising by chance, as determined by each of the t-Tests, are given in Table 5.

T-test probabilities less than five per cent indicate that the difference between the determined and the mean values is not due to chance, and hence significant.

Table 5. *Student's t-test results (P %)*

Laboratory			**Sample Number**		
No.	**1**	**2**	**3**	**4**	**5**
1	3.2	11.5	2.1	22.2	65.9
2	8.9	28.9	84.7	15.1	53.5
3	81.1	83.7	81.7	69.1	30.4
4	7.9	88.2	17.0	82.5	39.3
5	30.9	36.2	53.8		6.4
6	11.4	13.9	18.0	83.8	13.0
7	39.0	87.9	3.8	99.7	24.2
8	42.7	19.8	56.2	15.3	61.3
9	26.8	47.4	81.1	54.5	4.1

With reference to Table 5, it is evident that the difference between the two paired sets was significant (P< 0.005 %) in only four test results, which have been highlighted. Note that Laboratory 4 did not test sample 4. However, it should be borne in mind that the result of "not significant" is not so much a complete acceptance of the null hypothesis but rather an outcome of "significance of difference not established".

In view of the above, the intended purpose of the soil must be considered to ascertain whether the difference bears significant consequences. For example, if the potential expansiveness of the soil is of primary concern, in the case of Sample 1, the significant result (P = 3.2 %) is expected to be of no consequence as this soil was non-plastic and had a clay content of 3 % compared to the average of all the laboratories which was 4,7 %. Hence the potential expansiveness, based on Laboratory 1 was "Low", as determined by all the other laboratories. Furthermore, in the two significant results of Sample 3, the Activity of soil (Equivalent PI/clay content) was higher due to the lower clay content. However, this did not affect the associated potential expansiveness, which remained "Low". In the case of Sample 5, the potential expansiveness of the soil, as determined by Laboratory 9, was the same as seven of the other eight laboratories. However, depending on the intended purpose of the soil, if the potential expansiveness is not the primary concern, significant differences in grading may be of relevance.

4 CONCLUSIONS

The results indicated that the largest variations in the results of the laboratories occurred in the size ranges that are determined using the hydrometer analysis (percentages finer than 0.075 mm, 0.005 mm and 0.002 mm). Hence, it was established that the dispersing agent type, concentration, and dosage solely influenced the results of the test methods considered. The most effective of the dispersive agents considered was 125 millilitres of four percent sodium hexametaphosphate.

When considering the clay contents of the five samples, the range in values from the nine laboratories increased (to a maximum of 23 %) in the case of the sample with the highest average clay content (maximum of 42 %). A similar positive correlation was observed when considering the standard deviations (of the clay contents), which increased (to a maximum of 7.5) with an increase in the average clay content of a soil.

To improve the accuracy of hydrometer analyses, it is recommended that laboratories that are not including companion “blank” solution testing, to account for the effect of the dispersing agent on hydrometer readings, should introduce this procedure.

Finally, it is strongly recommended that laboratories send identical soil samples to other laboratories to be tested, on a regular basis, for quality control purposes. The results of this investigation were anonymously shared with all the participating laboratories.

REFERENCES

Bindu, J., & Ramabhadran, A. (2010). Effect of concentration of dispersing agent on the grain size distribution of fine grained soil. Proceedings of Indian Geotechnical Conference, (pp. 275–278). Mumbai, India.

Boadu, F. K. (2000). Hydraulic conductivity of soils from grain size distribution: New models. Journal of Geotechnical & Geoenvironmental Engineering. (126)8, 739-146.

BS (British Standard) (1975). Grain Size Distribution Determination. Methods of Test for Soils for Civil Engineering Purposes, British Standards Institution, London.

BS (British Standard) (1990). BS 1377-2:1990. Methods of Test for Soils for Civil Engineering Purposes, Part 2, Classification Tests, British Standards Institution, London.

Emeka, A. E. (2015). Effects of different dispersing solution on the properties of soil particle during hydrometer test. International Journal of Scientific & Engineering Research, 6(6), 1765–1776.

IS (Indian Standard) 1985. IS 2720: 1985 Methods of Tests for Soils, IS Designation 2720 Part IV. New Delhi: Bureau of Indian Standards (BIS).

ISRIC (2002). International Soil Reference and Information Centre. Procedures for Soil Analysis. Technical paper 9. Wageningen, Netherlands: ISRIC.

IUSS-WRB 2015. World Reference Base for Soil Resources 2014. Update 2015. International Soil Classification System for Naming Soils and Creating Legends for Soil Maps. World Soil Resources Report No 106. Rome: FAO.

Jacobsz, S. W., & Day, P. (2008). Are we getting what we pay for from geotechnical laboratories? Civil Engineering (South African Institution of Civil Engineering), 16(4), 8-11.

Kaur, A., & Fanourakis, G. C. (2016a). The effect of type, concentration and volume of dispersing agent on the magnitude of the clay content determined by the hydrometer analysis. Journal of the South African Institution of Civil Engineering, 58(4), 48-54.

Kaur, A., & Fanourakis, G. C. (2016b). Influence of phosphate dispersing agents on particle size distribution of soil fines. Acosta-Martínez & Kelly (Eds), Geotechnical and Geophysical Site Characterisation 5 – Lehane, Australian Geomechanics Society, Sydney, Australia.

Kaur, A., & Fanourakis, G. C. (2016c). Effect of hydrometer type on particle size distribution of fine grained soil. Proceedings of First Southern African Geotechnical Conference. (pp. 275–278). Sun City, South Africa.

Kaur, A., & Fanourakis, G. C. (2018). Effect of sodium carbonate concentration in calgon on hydrometer analysis results. Periodica Polytechnica Civil Engineering, 62(4), 866–872.

Lambe, T. W. (1951). Soil Testing for Engineers, New York: John Wiley & Sons, Inc.

SANS 3001 (2011). SANS 3001: Civil Engineering Test Methods. SABS Standard Division Designation 3001 Part GR3, South African Bureau of Standards, Pretoria, South Africa.

Sridharan, A., Jose, B. T., & Abraham, B. M. (1991). Determination of clay size fraction of marine clays. Geotechnical Testing Journal, 14(1), 103–107.

Stott, P R., & Theron, E. (2015). Some shortcomings in the standard South African testing procedures for assessing heaving clay. Journal of the South African Institution of Civil Engineering, 57(2), 36-44.

TMH1 (1986). Standard Methods of Testing Road Construction Materials. Pretoria: CSIR (Council for Scientific and Industrial Research), National Institute for Transport and Road Research.

USAEWES (United States Army Engineer Waterways Experiment Station) 1960. The Unified Soil Classification System. Technical Memorandum No 3-357. Appendix A, Characteristics of soil groups pertaining to embankments and foundations, 1953; Appendix B, Characteristics of soil groups pertaining to roads and airfields. Vicksburg, MS: USAEWES.

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