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Rapid shrinkage curve generation using small samples and highly volatile non-polar liquids

Generation rapide de courbes de retrait a l'aide de petits echantillons et de liquids non polaires hautement volatils

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ABSTRACT: One of the essential tools for assessing swelling potential of expansive soils is the shrinkage curve. A number of methods of measuring shrinkage curves have become available but they generally suffer from two disadvantages. Firstly several days are required to generate a shrinkage curve, and secondly recent developments in geotechnical practice call for estimates of soil parameter values which meet statistical criteria for reliability. Currently popular procedures are not well suited to give an indication of the probable range of variability of shrinkage characteristics needed to meet any statistical criterion. A method is presented which can give accurate shrinkage curves and an estimate of their variability within one or two days. It is shown that for some soils the intrinsic variability in shrinkage curve characteristics can be large.

RÉSUMÉ : L'un des outiles essentiels pour evaluer le potentiel de gonflement des sols expansifs est la courbe de retrait. Un certain nombre de methods de mesure des courbes de retrait sont disponibles mais elles souffrent generalement de deux inconvenients. Premierement, plusieurs jours sont necessaires pour generer une courbe de retrait, et deuxiemement, les developpements recents de la pratique geotetechnique exigent des valeurs de parametres du sol qui repondent aux criteres statistiques de fiabilite. Les procedures actuellement populaires ne sont pas bien adaptees pour donner une indication de la plage probable de variabilite des caracteristiques de retrait necessaire pour repondre a un critere statistique. Une methode est presentee qui peut donner des courbes de retrait precises et une estimation de leur variabilite en un ou deux jours. On montre que pour certaines sols, la variabilite intrinseque des caracteristiques de la courbe de retrait peut etre importante.

KEYWORDS: Rapid soil shrinkage testing, soil shrinkage curves, unsaturated soil mechanics.

1 INTRODUCTION.

The shrinkage curve is one of the fundamental properties of a soil and has long been recognised by soil scientists and engineers as essential for the understanding of several problems including water retention, water movement, and heave potential. Several methods have been devised to generate shrinkage curves. Such tests typically take about one week to complete. A method is here presented which allows for numerous samples to be tested in one or two days with apparently good accuracy and allows some materials not amenable to other testing methods to be assessed.

2 THE SOIL SHRINKAGE CURVE

Soils which shrink and swell do not have a "rigid" structure, i.e. they do not have stable relations between their solid and pore volumes and experience significant bulk density variations during water content variation (Taboada 2003).

Peng and Horn (2005) noted that understanding and prediction of water transport processes in non-rigid soils requires a knowledge of the dynamics of soil shrinkage. Chertkov (2003) noted that the shrinkage curve is one of the physical characteristics of soils and it is necessary for the prediction of soil behaviour when a soil interacts with water. The shrinkage curve is then, an important tool for dealing with shrinking/swelling soils. In principle, it demonstrates a relationship between volume and water content. A typical representation of the shrinkage curve from Fredlund (1999) is shown in Figure 1.

Unfortunately, there is no universally accepted way to illustrate this dependence. Among soil scientists, two popular ways to show this dependence are a) specific volume (volume per unit mass) versus water content and b) void ratio (volume of voids : volume of solids) versus moisture ratio (volume of water : volume of solids). Among engineers, common ways to show this dependence are void ratio versus water content and specific volume versus water content. Even more unfortunately, there is no consistency in the use of the term "water content"; sometimes it refers to volumetric, sometimes gravimetric measure. In this paper, "water content" is always gravimetric and the pattern of Figure1 is followed. This has the advantage that specific volume has a definite base line value (the inverse of density of the soil solids) and a definite reference line (the zero air voids (or saturation) line which allows immediate and convenient comparisons between different specimens if required.

3 REVIEW OF CURRENTLY POPULAR METHODS

Shrinkage curves have been produced by soil scientists since before engineers appear to have appreciated their value. A major concern of soil scientists is agriculture and hence with soils containing enough moisture for crop production. Soil scientists have, for many years, been producing shrinkage curves for the wetter part of the range by immersion in paraffin (kerosene), toluene or other non-polar liquids. A container of non-polar liquid is placed on a balance and a sample is immersed by hanging from a support (Taboada, 2003). When a soil is saturated it does not absorb non-polar liquids and the method gives a shrinkage curve very conveniently up to the air entry point. When a soil is drier than the air-entry point, however, it does absorb non-polar liquids. Unsaturated soil mechanics deals with soils drier than the air-entry point and to overcome this problem the technique of coating soil samples with Saran resin dissolved in methyl ethyl ketone was developed (Brasher et al., 1966), and became widely accepted as the standard method for shrinkage curve generation. The essential feature of the Saran resin coating is impermeability to liquid water (allowing volume determination by immersion), combined with permeability to water vapor (allowing the sample to undergo a drying or wetting regime). While this method appears to be straightforward and reasonably accurate it suffers from the disadvantages that about six weeks are needed to produce a shrinkage curve and methyl ethyl ketone is hazardous to human health and living environment. The test should be performed under a fume hood exhausting above the level of the local buildings. Alternative, less hazardous encasement materials such as Elmers craft glue have been proposed (Krosley et al., 2003).

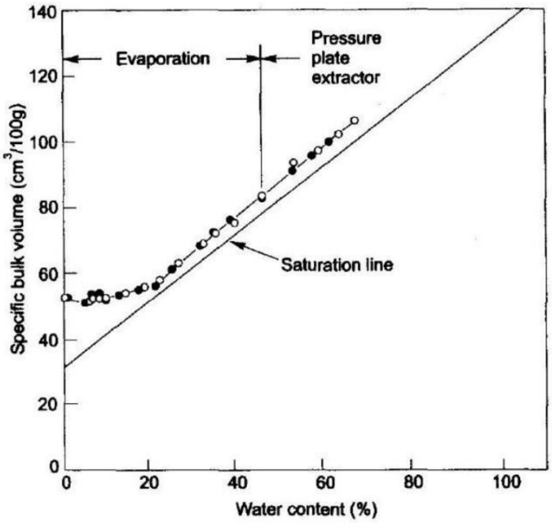

Figure 1 Shrinkage curve for Regina Clay (Fredlund, 1999)

It is also possible to determine the volume of a soil clod by coating with a totally impermeable material before immersing in liquid. Paraffin-wax is the standard coating material, but it suffers from a number of disadvantages. The temperature of melting, and the high latent heat of fusion cause considerable heat input to the sample being coated. There is usually a significant expulsion of air and/or water vapour from any sample which is not desiccated. This may lead to small blow- holes in the coating. These may be so small that they are not easily seen and are only detected when the sample is found to gain weight on immersion. Paraffin-wax also contracts significantly on solidification, and tension cracking of the coating may occur, particularly in cool conditions. Cornelis, *et al.* (2006) noted an advantage of this method in that samples for testing can be broken from clods along lines of cracking for specimens in the dryer range of the shrinkage curve, eliminating the inclusion of voids from the test sample. This is not normally considered for clod-test specimens, and the results of these tests may be significantly affected by included voids.

Another approach to the production of shrinkage curves is the direct measurement of dimension changes in a sample during drying. This requires a large enough sample to allow acceptable accuracy in dimension measurements. This in turn requires a considerable time for evaporation, particularly for clays, which have low permeability both to water and to water vapour. Bensallam *et al.* (2012) determined shrinkage curves using such a method. Their apparatus allowed shrinkage curve readings to be taken at 6 hourly intervals, the final stage requiring three days of oven drying. The total time required to produce a shrinkage curve was not stated. Fredlund (2019) advocated a less complex variant of this method where the dimensions are measured by micrometre callipers each time the sample is weighed.

A rather simple method of shrinkage determination was devised by Tariq and Durnford, (1993). A soil sample is enclosed in a normal rubber balloon. The balloon provides a waterproof coating; its flexibility allows volume determination. Drying of the soil sample is achieved by pumping air into the balloon between mass and volume readings. Cornelis *et al.* (2006) reported favourably on this method. They noted that using an air pump delivering 50 to 100 L per hour they were able to take measurements at 12h intervals. Shrinkage curve production took two weeks. They noted that it should be possible to reduce the time by using a more powerful pump.

Another simple and inexpensive method has been proposed by Cerato and Lutenegger (2006). A shrinkage trough is filled as for a normal linear shrinkage determination. The sample is dried progressively, and measurements of linear dimensions taken at suitable intervals. The moisture content is determined by weighing the trough and its contents. The procedure has been found to give good results for determining the shrinkage limit, and gives a fair indication of the shape of the shrinkage curve. Two problems may be encountered with this method. Firstly, expansive clay samples arch and shatter to an unpredictable extent even when dried slowly; this can make linear measurement uncertain. Secondly it is difficult to exclude all voids when filling the shrinkage trough; this may lead to uncertainties in the estimation of volume. A shrinkage curve can be obtained in about one week by this method.

3 GENERAL CONSIDERATIONS

Several alternative coatings were tried following the methods of Krosley et al. (2003). It was soon realised that any such coating, which can give impermeability to water, but permeability to water vapour, cannot give a shrinkage curve in a short period of time. The limiting factor for a rapid shrinkage curve is rapid drying. This implies a small sample and all-round exposure to the air. To maintain acceptable accuracy this in turn demands a sensitive balance. Sensitive balances have a relatively small measuring capacity, which necessitates modification of the normal approach (Taboada 2003) to volume determination by Archimedes principle. Dispensing with a water-tight coating requires that volume determination be done with a non-polar liquid. Immersion at moisture contents below the air entry point results in liquid entering the sample. Volume and mass determination must therefore account for absorbed liquid.

4 MATERIALS AND METHODS

An analytical balance capable of reading to 0.0001g was used. To utilize this accuracy a balance requires a stable operating environment free from air currents and vibration as well as temperature and humidity fluctuations. The balance was located in a shielded area where it was mounted on a small table closed on three sides to give protection from air currents. A hole in the centre of the tabletop allowed a hook to hang from an attachment point in the balance to the shielded area below. Cradles made from 1mm galvanized wire were used to carry the soil samples. The cradles and their samples were suspended from the hook attached to the balance. A manual lifting device was made to raise a container of liquid to immerse the suspended sample. To minimize disturbance of the fluid and allow rapid stabilization of the balance reading, the lifting must be smooth and vertical. It should also always be to the same height and the fluid should be maintained at the same level in the container. The apparatus is shown in Figure 2.

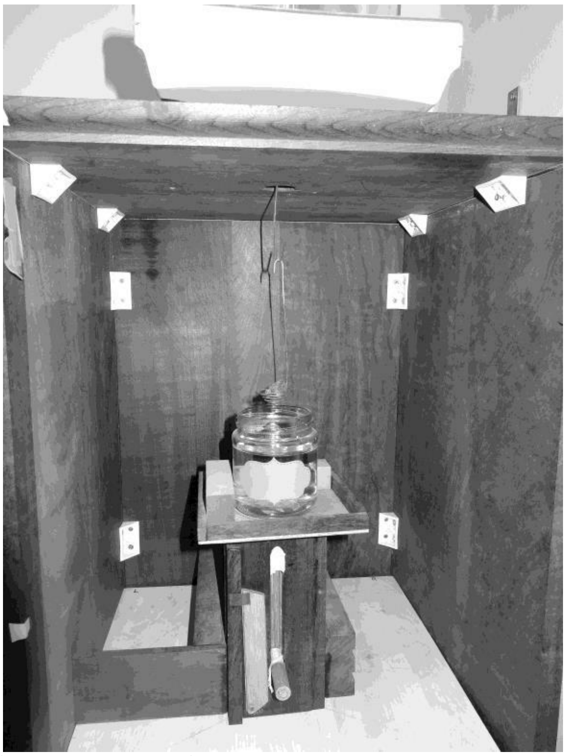

Figure 2 Apparatus used.

Some turbulence is inevitable however smooth the immersion of the sample, and stable readings can only be obtained when the turbulence has considerably dissipated. A time of about 5 seconds is usually sufficient for readings to become stable for saturated samples. Figure 3 shows graphs of balance reading (g) against time (s) for a saturated sample and for the same sample after air drying, during submergence in Chloroform. The reading for the saturated case stabilises to a constant value of 2.4448g about 5 seconds after immersion. It is usual for the balance reading to initially dip below the equilibrium value and rise to the equilibrium value as turbulence subsides. The unsaturated sample absorbs some of the non-polar fluid. After the initial turbulence, absorption of solvent is indicated by a smooth curve which can be fitted by a trend line, in this case a power-series with coefficient of determination $R^2 = 0.997$ which is quite typical. The vertical lines mark immersion and extraction, and the best estimate corresponding to start of immersion is 1.971. Before testing, the cradle is weighed in air and in the solvent. The sample remains in the cradle throughout the shrinkage curve procedure.

A problem with the absorption of fluid by the sample is its removal before the next mass and volume determination can be made. This necessitates a very volatile liquid if rapid determination is to be achieved. A number of volatile liquids were considered. Some were discounted because of health hazard; others were found to have damaging effects on soil samples. Two were chosen as suitable for the purpose: chloroform and benzene (petroleum ether). Both have the advantages of reasonable price and ready availability. Both are widely used as solvents. Chloroform has the advantage of posing no fire hazard – it is non-inflammable, but it has the disadvantage that it will produce toxic gasses when exposed to fire. Benzene is highly inflammable and poses a significant fire risk. Chloroform has the disadvantage that it has anaesthetic properties and could cause lack of concentration or worse if inhaled at high enough concentration. No such problems were noted in many months of use for shrinkage curve production. Chloroform appears to have two major advantages over benzene. It is more volatile, and its density is far greater (more than double). This means that for any sample size the time between data readings can be shorter, and for any required degree of accuracy of volume determination the sample size can be smaller.

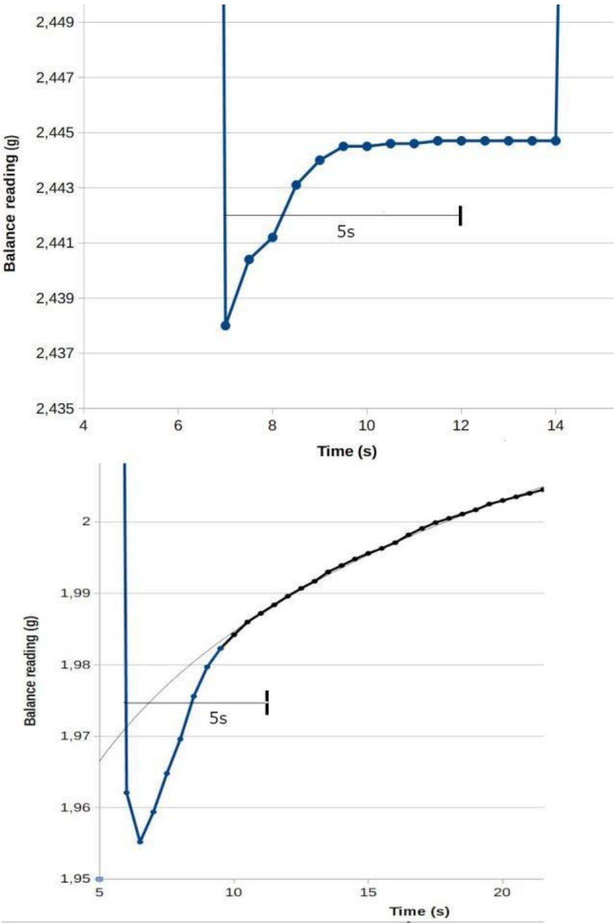

Figure 3 Graphs of weight (g) against time (s) for (upper) saturated sample during immersion and (lower) the same sample after air drying with a trend-line for estimating the weight immediately after submersion.

To assess time advantages of various volatile solvents control filter papers were saturated with non-polar solvents and allowed to reach equilibrium with prevailing air conditions. Chloroform reached its residual value in about 60% of the time required for benzene and in a very small fraction of that required by solvents traditionally used by soil scientists. A small residual (of the order 1-2% of the initially absorbed mass for both liquids) remained held by the filter paper. This residual can be expelled by oven drying. The filter paper used (Whatman’s 42) held an equilibrium water content of approximately 7.4% by mass at the prevailing temperature and humidity at the time of the experiment. The retained content of chloroform and benzene at the same conditions were approximately 1.0% and 1.5% by mass respectively. Evaporation tests were performed on several clayey soils, with PI ranging from 7 to 42, in order to assess evaporation times and residual retention.

Figure 4 shows drying curves for three samples of a saturated clay soil. The weight of each of the three samples was approximately 5g. Weights have been normalised to 1g and superimposed for meaningful comparison. One sample was simply air-dried, the other two were immersed for 20 seconds commencing 120 seconds after the start of the test. The sample immersed in chloroform took approximately 3 minutes to reach the air-drying curve, the sample immersed in benzene took approximately 5 minutes. Water content remained above air entry level throughout the tests. The fact that the immersed samples reach the air-drying curve appears to confirm that no solvent was retained by the samples.

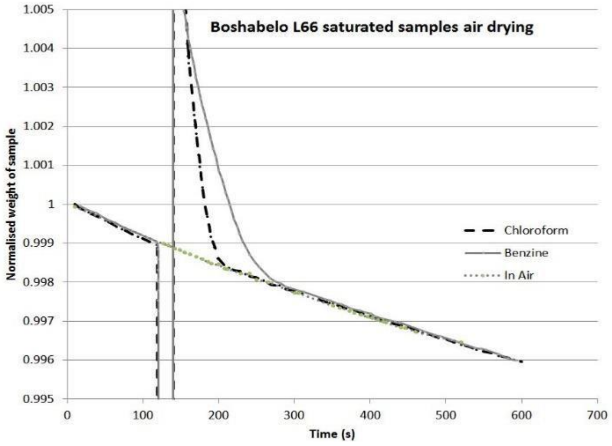

Figure 4. Normalized drying curves of 3 samples of a saturated clay

For unsaturated samples, liquid is absorbed into the soil on immersion. Evaporation from the fabric of the soil takes longer than from the surface only, and some of the liquid is retained in the sample by capillary suction.

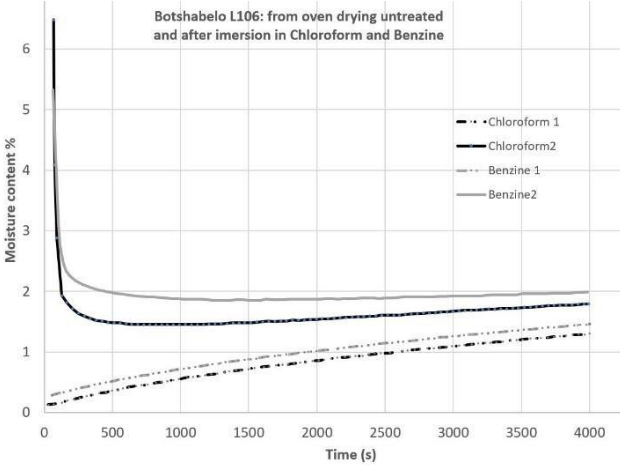

Figure 5. Moisture content (%) : Time (s) with and without immersion.

Figure 5 shows curves of gravimetric moisture content against time for samples of a clay soil on exposure to the air after oven drying. Four samples with mass of approximately 2g each were oven dried. Two were then exposed to the air. One of this pair was immersed in chloroform for 30 seconds, 40 seconds after the start of the test. The other two samples were treated in the same way but with immersion of one sample in benzene. Soil samples absorb moisture from the air when removed from the oven; the times taken for the samples to cool and be transferred to the scale were a little different for each pair of samples, so the starting moisture contents are not identical.

The form of the curves is similar and similar conclusions can be drawn. The curves for immersed and non-immersed samples do not meet but do tend to become parallel. The gap between the final parallel curves is the retained non-polar liquid content. The chloroform-dipped curve becomes parallel to the un-dipped curve after about 50 minutes. The curve for benzene is not quite parallel after 70 minutes. Both curves tend to a retained liquid content of approximately 0.5% of the dry weight of the sample. 1.0% retention was reached after about 12 minutes following immersion in chloroform and after about 23 minutes following immersion in benzene. Chloroform has more than twice the density of benzene, so a similar mass retained implies that more than twice the volume of benzene is retained compared to that of chloroform.

5 APPLICATION OF THE METHOD

Shrinkage curves of four samples of an active clay (PI 36) are shown in Figure 6. The samples were prepared using standard procedures for Liquid Limit determination and were well mixed and assumed to give representative values of re-moulded material for the whole sample. Air drying at approximately 23°C and 35% relative humidity required 8 hours for completion of the curve. Figure 6 shows plotted values for all 4 samples. Plotted values for all samples are all close to the best fit curve. Values were then adjusted by a correction for absorbed solvent for the readings after full saturation and before oven drying. The best fit involved the measured 0.5% correction for absorption just before oven drying, reducing to 0.05% just after air-entry. The corrected and uncorrected curves are so close that the difference is very small and barely discernible in the Figure. The correction could be neglected.

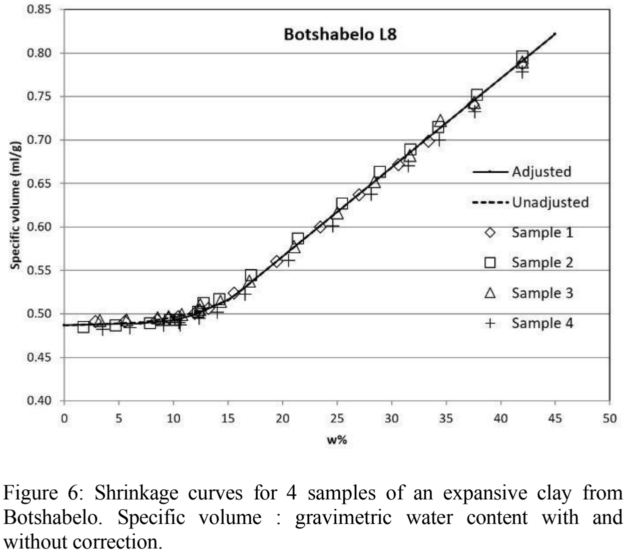

Figure 6: Shrinkage curves for 4 samples of an expansive clay from Botshabelo. Specific volume : gravimetric water content with and without correction.

This procedure gives a rapid method of getting a representative shrinkage curve as suitable for the working load design method but gives no indication of the variability needed for reliability based design. Swelling clays give more problems when they have marked variability (Stott & Theron 2017). Ten individual samples were tested from one distinct layer of expansive clay under a building which suffered severe heave damage in a few restricted locations. Drying the samples for a few minutes in an incubator at 40^{0}C after immersion allowed the ten curves to be produced in just a few hours followed by oven drying. Figure 7 shows these shrinkage curves as second order polynomial trend lines fitting the uncorrected shrinkage data points.

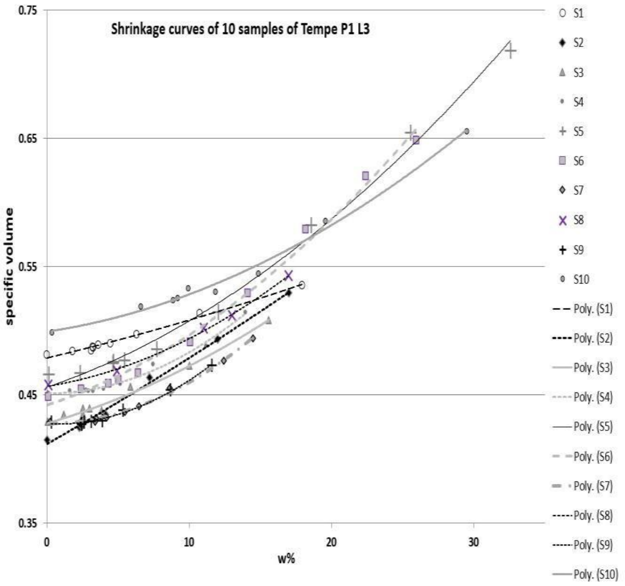

Figure 7. Shrinkage curves (specific volume : gravimetric water content) for ten individual samples from one soil layer below the foundation of a building which suffered localised heave damage.

Correction for retained solvent made a more noticeable difference than for the more highly expansive clay of Figure 6, but the adjustments were still small and were not applied to Figure 7. Figure 8 gives change in specific volume and water content for all ten samples. The large variability gives a strong indication that this soil could be problematic since heave damage depends on differential heave, and not just on average heave.

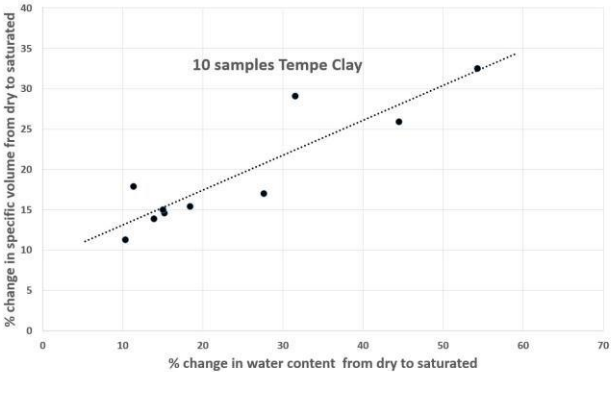

Figure 8. Change in Specific Volume and water content for 10 samples of Tempe clay plus linear trend-line

If only one sample were taken there appears to be a greater probability (60%) of finding change in specific volume less than 20 (suggesting low expansiveness) than the probability (20%) of finding a value above 40 (suggesting significant expansive potential). More tests would probably yield both lower and higher values, but even these ten indicate large differential heave, which is likely to be more troublesome than high heave alone. This suggests a reason for the severe damage in limited areas and negligible damage in other parts of the structure.

5 CONCLUSIONS

Shrinkage curves can be produced quickly and easily for small samples using volatile non-polar fluids. Chloroform appears to be an ideal liquid for this purpose, requiring little time for removal from samples between mass and volume determinations and requiring little correction for retention by desiccated samples. Drying at slightly elevated temperature in an incubator can reduce the total time required for at least twenty full shrinkage curves to below 8 hours even for highly active clays.

6 ACKNOWLEDGEMENTS

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