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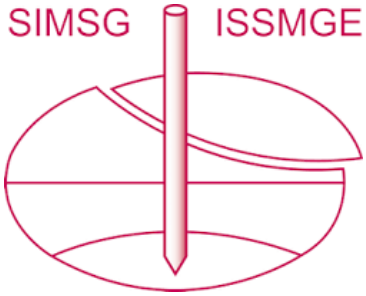

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Destroying the structure of halloysite clay through oven drying

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ABSTRACT: Halloysitic soils may develop in areas where volcanic ash is exposed to intense tropical weathering in high rainfall climates. It can exhibit unusual characteristics when tested using conventional geotechnical laboratory techniques due to the sensitivity of the clay minerals and soil structure to high temperatures and low humidity. The effect of low temperature oven drying (which conventional wisdom suggests may limit the physical alteration of soil samples) on Atterberg Limit classification testing is analysed through comparison of a range of drying and rehydration tests. Test results indicate that low temperature oven drying at 40ºC does not prevent the transition of halloysite to metahalloysite and may lead to the incorrect classification of halloysite soil.

1 THE GENESIS OF HALLOYSITE CLAY

Halloysite is formed when alternating kaolinite clay minerals and water molecules form delicate hollow cylindrical nanotubes. Typically derived from the weathering of volcanic ash soils and rock in tropical environments, halloysite can exist in two distinct wet states; as hydrated halloysite or dehydrated metahalloysite. Halloysite typically develops where intense weathering processes leach minerals such as silica and magnesium oxide from the soil profile, resulting in remnant soils rich in iron and aluminium oxides. In some areas with very high rainfall and no significant dry season, strong interparticle bonding resulting from the dehydration of aluminium and iron sequioxides to form aggregations (Fookes 1997) is prevented, allowing halloysite rich residual soils to remain in a natural hydrated form.

The structure of natural hydrated halloysite clay retains water in three distinct ways; as adsorbed water on the outside of the halloysite tubes, as interlayer water within the tubes as depicted in Figure 1 and as water molecules within the kaolinite crystalline lattice structure.

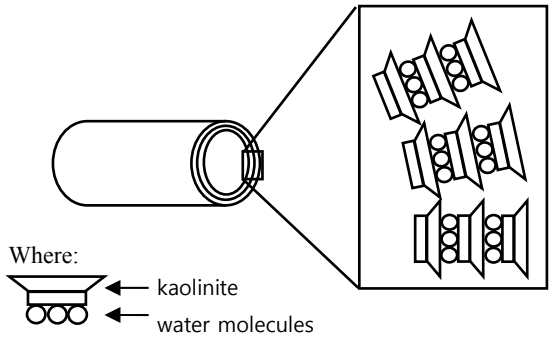

Figure 1. Interlayer water within a halloysite nanotube.

Some adsorbed water may be drawn out of the soil mass at room temperature, as for many soils, whereas the interlayer water may be partially expelled at temperatures over 50º C or relative humidity less the 50% (Fookes 1997). The mobilisation of the interlayer water (also referred to as "water of hydration") marks the transition from hydrated halloysite to metahalloysite. The dehydration of halloysite to metahalloysite is an irreversible process and may result in the splitting or unrolling of the tubes (Mitchell 1976). Ross and Kerr (1934) provide evidence that suggests the water retained in the kaolinite lattice of halloysite is expelled at a temperature of approximately 400ºC to 550ºC.

The specific conditions required for the formation and preservation of hydrated halloysitic residual soil are key to understanding the behaviour of the clay if conditions are changed. How changing conditions affect soils is a fundamental question of geomechanics, but it's the dramatic and irreversible nature of the changes that can occur in halloysite that can be misinterpreted if viewed under the microscope of conventional soil mechanics.

2 MISLEADING LAB TEST RESULTS

If Fookes' (1997) assertion that the water within the clay mineral structure of halloysite is "inert and has no influence on the mechanical behaviour" of the soil is correct, then a true account of the physical behaviour for a soil mass (reflected in Atterberg Limits) should exclude this water. In addition to recording a higher Liquid Limit than would be recorded for a sample where only the adsorbed water is removed via drying, the loss of the water of hydration in halloysite will change the structure of the soil from its in situ state. This has the effect of erroneously changing the USCS classification of the soil from that of a high plasticity clay to a high liquid limit silt. The misleading nature of the laboratory test results would be somewhat concealed through a tactile assessment of the tested soil, which would exhibit the characteristics of metahalloysite, not halloysite.

This results of testing presented in this paper support the notion that oven drying may lead to incorrect classification of soil in accordance with USCS guidelines. The author recommends that geotechnical investigations proceed with caution or eliminate Atterberg Limit classification testing at sites where structure-sensitive residual soils, such as halloysite, are present.

3 THE INFLUENCE OF OVEN DRYING

In this study, seven soil samples known to contain halloysite clay minerals as a result of x-ray diffraction analysis, oxalate analysis and electron microscopy (see Figure 2) were split and placed in either a 40ºC oven or 105ºC oven for a period of 103 hours. Over the 103 hour drying period the moisture content of all the samples was measured nine times and was seen to stabilise in the final 24 hours to within 0.4% (by mass) for the samples in the 40ºC oven and 0.1% (by mass) for the samples in the 105ºC oven. The total measured moisture loss in the samples dried in the 105ºC oven was used to infer the initial moisture content of the split bulk sample (i.e. the samples in the 105ºC oven are assumed to have reached a moisture content of zero after 103 hours for the purposes of this study).

Following the 103 hours of oven drying, all samples were stored on a shelf and allowed to rehydrate in room temperature conditions for a further 180 hours, during which time the mass of the samples was measured seven times. The mass of all samples was measured to increase by between 2.4 % and 5.7 %. The increase in mass is inferred to be the result of water adsorbing to the clay from the humidity in the atmosphere. After 190 hours the mass of soil samples, having adsorbed

water from the atmosphere was seen to stabilise, as shown on Figure 3.

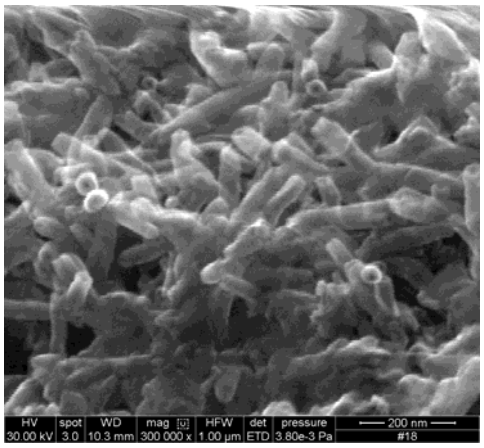

Figure 2. Electron microscope image of soil samples tested in this study showing hollow tubes approx. 35 - 50 nanometres in diameter and 150 - 190 nanometres in length.

To quantify the effect of oven drying the samples, a third subset of the original seven samples was allowed to air dry from the as-sampled field moisture content. After 220 hours, the samples allowed to air dry had reached a constant mass. The results for air dried samples are also plotted on Figure 3. If it is assumed that the percentage of adsorbed water (as a percentage of the soil mass) was similar for all samples in equilibrium conditions (stored in the same location), then the difference in final moisture content of the samples dried in the 40ºC and 105ºC ovens (then allowed to rehydrate) and the air dried samples is inferred to represent the water of hydration in the samples tested.

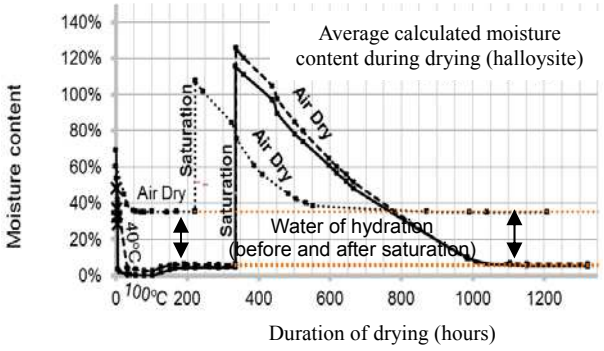

Figure 3: Equilibrium MC of samples in ambient air conditions.

The measured difference in the equilibrium moisture content of the air dried samples and the samples dried in the 105ºC oven was 29.9 % ± 0.1%. This suggests the water of hydration in the samples analysed is approximately 29.9%. This is higher than some previously published moisture content results, such as that of Fookes (1997) who asserts that "the loss of water of hydration in a pure halloysite is equal to 14% of the dry soil weight." In noting this difference, the author acknowledges that the samples tested were not a pure halloysite and contained multiple other clay mineral types.

By extension, this suggests that the low temperature oven was able to expel 27.2% of the 29.9% water of hydration in the samples.

The inference that the difference in moisture content after 220 hours reflects the water of hydration was further tested by saturating all sample sets then allowing them to air dry for a further 1000 hours (approximate). The moisture contents of the samples after 1200 hours were measured to be the same as they were prior to saturation (see Figure 3). This confirms the permanent nature of the loss of water of hydration.

For comparison, three samples of residual basaltic clay (inferred to be predominantly montmorillonite) were dried for 270 hours then allowed to rehydrate at room temperature. The moisture contents of the samples were seen to converge during rehydration, following drying (see Figure 4), suggesting the moisture loss is temporary in montmorillonite.

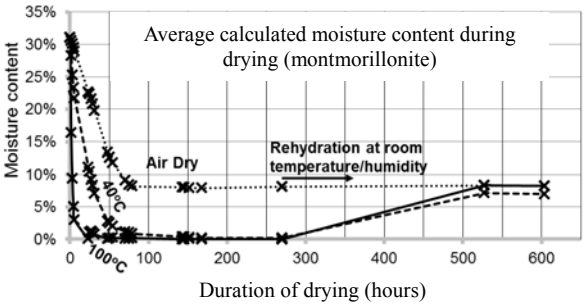

Figure 4. Equilibrium MC of montmorillonite samples in ambient air conditions (3 samples).

4 CONCLUSION

From the results of the drying test it can be inferred that, in industry standard conditions, a low temperature (40ºC) non-humidity-controlled oven can expel the water of hydration from halloysite clay samples. If the high temperature oven is assumed to remove 100% of the water of hydration from the samples and the water of hydration is assumed to represent 29.9% of the soil mass, the low temperature oven may be inferred to expel up to 91% of the water of hydration, if the relative humidity is allowed to approach zero. This suggests that an industry standard laboratory oven at 40ºC (in which the humidity in the oven is not controlled), will mostly convert hydrated halloysite to metahalloysite. This is consistent with the findings of Brindley and Goodyear (1948), that suggest a relative humidity of 15% (even at low temperatures) may expel the majority of interlayer water from halloysite.

This result suggests that samples of halloysitic clay prepared for Atterberg Limit classification testing by oven drying will exhibit altered mechanical behaviour from that exhibited in situ. This suggests that when halloysite clay is identified at a site, conventional wisdom requiring the use of low temperature ovens, may also lead to Atterberg Limit classification results that do not reflect the in situ mechanical behaviour of the soil.

5 REFERENCES

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