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**Microstructural changes of an undisturbed, reconstituted and compacted high
plasticity clay subjected to wetting and drying**

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Abstract

The behaviour of soil, and in particular compacted clay fill, can have significant implications on the safe and reliable operation of man-made infrastructure. The mechanical behaviour of soil (e.g. volume change and shear strength) is widely recognised as being associated with the microstructural arrangement (fabric/structure). In the case of high plasticity clays, despite the large amount of research carried out, soil microstructure and its evolution along mechanical and hydraulic paths is still not well understood. This makes incorporation of microstructural analysis difficult in engineering practice and highlights the need for further research. A comprehensive microstructural analysis of Maryland clay, a high plasticity residual soil, based on Mercury Intrusion Porosimetry tests, is presented in this paper. Experimental results obtained from undisturbed, reconstituted and compacted specimens subjected to different hydraulic and mechanical paths are described. As with mechanical investigations, the reconstituted state is proposed to be used routinely as a reference state for comparison of undisturbed and compacted soil. The microstructural evolution of the compacted clay, prepared on the wet side of standard Proctor optimum water content, with an initially high void ratio, is examined along the main drying path. Importantly, a monotonic suction increase from the as-compacted state is shown to have negligible effect on the distribution of macropores. However, a new insight is provided based on the evolution of the dominant micro-pore entrance diameter which is shown to reduce with increased suction. This micro-pore entrance diameter is shown to correspond with the theoretical suction back-calculated from a simple capillary tube model, up to a limit. It is observed that, under oedometric conditions, the as-compacted microstructure is erased during saturation (soaking) and resembles the reconstituted microstructure. For this particular material and preparation conditions, it is demonstrated that a bimodal microstructure is not recovered on drying from a saturated state.

Keywords: partial saturation, fabric/structure of soil, expansive soils.

1 INTRODUCTION

The microstructure of soil has previously been recognized to influence the mechanical behaviour of natural and compacted geomaterials. The pore size density functions (PSD), for both saturated and unsaturated soils, obtained from mercury intrusion porosimetry (MIP) have been shown to evolve as a result of hydraulic, mechanical and chemical changes (Delage & Lefebvre 1984; Delage et al. 1996; Romero et al. 1999; Simms & Yanful 2001; Koliji et al. 2006; Casini et al. 2012; Musso et al. 2013; Wang et al. 2013). Despite this, there is still limited understanding of how the soil microstructure evolves during combined hydraulic and mechanical paths. This paper focuses on the distribution of pore sizes and their separation into micro and macro components. Many additional factors contribute to soil microstructure (e.g. geometrical arrangement of pores, cementation/binding, pore-water chemistry), however these are not specifically investigated here.

The complexity of microstructural behaviour has led to compacted and reconstituted soils being considered as fundamentally different materials. However, the boundary between compacted and reconstituted soils is not always well defined in terms of microstructure (Tarantino 2011). Soils compacted dry of the standard Proctor optimum water content, are commonly reported to exhibit a bimodal PSD function. This characteristic is sometimes reported as an intrinsic characteristic of soils compacted dry of optimum. However, previous MIP studies have shown that on saturation the bimodal microstructure can either evolve towards a unimodal structure (Monroy et al. 2010) or remain unchanged (Thom et al. 2007). This conflicting experimental evidence makes difficult the development of unified frameworks and highlights the necessity for further experimental research.

The behaviour of materials compacted on the wet side of standard Proctor optimum water content are also relevant to engineering practice. For example, in the design of landfill

barriers and clay cores for embankment dams (Fell et al. 2005; Mitchell & Soga 2005). However, despite the practical relevance, microstructural investigations of soils prepared on the wet side of standard Proctor optimum water content are rather limited. It is widely believed that a compacted soil prepared on the wet side of optimum is homogeneous and has a unimodal pore size distribution (Tarantino 2011). However, Simms and Yanful (2001) showed that samples prepared on the wet side can also be bimodal, albeit for a material with a coarse shielding skeleton (Romero 2013).

Microstructural experimental evidence has been used for the development of constitutive models which attempt to describe the behaviour of unsaturated soils (e.g. Gens & Alonso 1992; Alonso et al. 1999; Alonso et al. 2010; Koliji et al. 2010; Della Vecchia et al. 2013; Alonso et al. 2013; Mašín 2013). In the model of Alonso et al. (1990), and models built upon this, a fundamental ingredient is the concept of a LC yield curve which relates the plastic compression of unsaturated soils to compression and suction variations. Alonso et al. (1987) and Sheng (2011) pointed out that the notion of a LC curve is inextricably linked to that of a bimodal PSD. For example, let us consider a compacted soil at point A, shown in Figure 1, which is assumed to have a bimodal structure. On saturation to point B, i.e. full saturation but not just zero suction, the size of macro-pores decreases with the possible combined effects of expansion of the clayey aggregates and collapse. This induces a predominant unimodal PSD. If the sample is then dried to a suction above the air-entry value and compressed to point C, the movement of the LC curve suggests that collapse can occur again which implies that at point C the sample should recover a bimodal PSD. On the other hand, for soils that contain a “shielding skeleton” of coarse particles it is possible to envisage that the shielding skeleton limits the shrinkage on drying of the bulk sample while the clayey fraction may continue to shrink and create macro-pores (Romero 2013). It is well known that collapse can occur in silty soils, similar to the loess described by Muñoz-Castelblanco et al. (2011) and the

volcanic ash described by Ferrari et al. (2013). This paper on the other hand is concerned with a high plasticity clay. Due to the soil-dependent evolution of the PSD additional microstructural experimental evidence is needed in order to understand the role played by the soil microstructure in the LC concept as well as its evolution with combined stress paths.

This paper examines the existence and evolution of a bimodal PSD in compacted clay prepared on the wet side of the standard Proctor optimum water content. Maryland clay, a high plasticity residual soil from New South Wales (Australia) has been used for this purpose. The PSD of the compacted soil is evaluated in relation to those of undisturbed and reconstituted specimens which are used as a reference framework for interpreting the experimental results. The PSD obtained after the application of hydraulic (wetting and/or drying) as well as combined hydro-mechanical (wetting at constant stress) paths are used to evaluate the evolution of microstructure. Starting from the as-compacted state the PSD evolution along a monotonic drying path is investigated, and the evolution of the dominant micro-pore diameter (i.e. the peak of the PSD in the micro range) monitored. Soil microstructure is also evaluated after monotonic wetting paths (by soaking) applied to compacted specimens under different vertical stress levels. The possibility for developing a bimodal PSD in compacted-saturated specimens when subjected to air drying is also investigated.

2 MATERIAL, EQUIPMENT AND METHODS

2.1. Soil tested

Maryland clay is a residual soil, from west of Newcastle (Australia), derived from the weathering of the parent mudrock material (Fityus & Smith 2004). The material has a liquid limit of $w_L = 69\%$, plasticity index $I_p = 43\%$ and specific gravity of $G_s = 2.65$. The

standard Proctor maximum dry density is 1.5 Mg/m^3 (void ratio 0.76) and the standard Proctor optimum water content is 24.8%.

Three different soil conditions were tested in this study: 1) natural or undisturbed, 2) reconstituted, and 3) compacted. Two undisturbed samples were collected from slightly different locations in a test-pit excavated at the Maryland test site to assess the in situ variability. The in situ water contents were 22.7% and 23.6%, respectively. Block samples were sealed with successive layers of plastic wrap, aluminium foil and wax. These block samples were then stored in a fog room for later testing. The structural arrangement of the undisturbed soil was qualitatively evaluated by means of scanning electron microscope (SEM) images as shown in Figure 2. Two structural levels can be clearly defined. Large macro-pores are evident in Figure 2a, which defines the macro porosity. At larger magnification (Figure 2b) a rather homogeneous structural arrangement, albeit displaying a complex geometrical arrangement, seems to define the micro porosity.

Reconstituted specimens were prepared by mixing dry powdered Maryland clay with deionized water at water content roughly 1.5 times the liquid limit. The slurry was then one dimensionally consolidated in a mould to a vertical stress of around 50 kPa. At the end of consolidation the samples had water contents between 50.6 % and 54.6 %.

To prepare the compacted samples, the powdered clay was mixed with water to a gravimetric water content of around 28% ($\approx 3\%$ higher than optimum). The blended material was then sealed in air tight vacuum storage bags and allowed to equilibrate. The samples were statically compacted to the target void ratio (1.27) in a single layer 10 mm high and 50 mm in diameter. The target void ratio was selected to provide an initial void ratio close to the void ratio attained by the reconstituted samples.

2.2. Mercury intrusion porosimetry (MIP) tests

The pore size distribution was determined using an AutoPore IV 9500 (Micrometrics) with a maximum injection pressure of 228 MPa. In the MIP technique, the theoretical or equivalent pore entrance size d is related to the applied pressure p through the Washburn (1921) equation:

$$1 \quad p = \frac{-4\sigma_{Hg} \cos \theta_{nw}}{d} \quad (1)$$

where σ_{Hg} is the surface tension of mercury (0.484 N/m) and θ_{nw} is the mercury-soil contact angle. A contact angle of 140° (Romero 1999) was adopted in this study. Values of void ratio are computed from the results obtained during the intrusion stage as follows:

$$2 \quad e_{MIP} = \frac{V_{mercury}}{V_s} \quad (2)$$

where V_s is the volume of the solids and $V_{mercury}$ is the volume of intruded mercury up to the current pressure.

The pore size density (PSD) function is obtained, at $\log x_m$ (the midpoint of the pore diameter class), from the derivative of the cumulative intrusion curve:

$$3 \quad f(\log x_m) = \frac{\delta(e_{MIP})}{\delta(\log d)} \quad (3)$$

Adopting Washburn's equation (Equation 1) assumes that all pores within the soil can be treated as continuous capillary tubes and that no deformation of the soil structure occurs during the intrusion of mercury under pressure. Although this is a gross over-simplification of the conditions likely to be encountered in natural porous materials (Diamond 2000), the MIP

data provide consistent results for analysing differences in PSDs between samples and the evolution of the PSD obtained in this study.

The preparation procedure of soil samples for MIP testing has been found to affect the results (e.g. Diamond 1970). For this reason specimens were dehydrated by sublimation (Delage & Pellerin 1984). Small ($\approx 1 \text{ cm}^3$) sub-samples were trimmed from the bulk samples using a wire saw. The sample was then immersed in liquid nitrogen followed by vacuum freeze drying at -56°C and vacuum of 0.3 Pa for around 48 hours.

3 EXPERIMENTAL PROGRAM

Table 1 and Figure 1 summarize the experimental program carried out in this study. The microstructure of the undisturbed material was examined on two samples (UD1 & UD2) tested immediately following sampling (i.e. at the in situ water content and void ratio). A third sample (UD1/1) was allowed to air dry at laboratory conditions. During this process, suction measurements were taken using a chilled-mirror dew-point psychrometer (WP4 Dewpoint Potentiometer, Decagon Devices) calibrated against partially saturated salt (KCl and NaCl) solutions. Following completion of the main drying measurements an MIP test was performed (with the final vestiges of water removed by drying in an oven). The measured suctions, water contents and void ratios of the undisturbed specimens are shown in Table 1.

The microstructure of the reconstituted soil was examined on specimens previously consolidated to a vertical stress of 50 kPa. Two samples for the MIP test were trimmed from separate reconstituted cakes to determine the uniformity. However, only one sample (R1) is shown here for clarity. A reconstituted sample (R2) was also allowed to air dry, under laboratory conditions, and prior to MIP the final water was removed by oven drying. In addition, one reconstituted sample (R3) was isotropically compressed to a mean net stress of

50 kPa and dried (in steps) to a suction of 400 kPa. In this case suction was measured/controlled using the axis-translation technique (0.5 MPa air-entry ceramic) in a triaxial apparatus with internal volume change measurement based on the volume of displaced water (Toyota et al. 2001; Ng et al. 2002). From sample R3, a sub-sample (R4) was taken and allowed to air dry in the laboratory followed by oven drying prior to MIP testing. A fifth sample (R5) was allowed to dry until a suction of 800 kPa using a suction-controlled oedometer cell.

To examine the existence of a bimodal PSD within the compacted clay, a high initial void ratio of 1.27 was selected for the compacted material, which also closely matched the initial void ratio of the reconstituted samples. The compaction water content was around 28% (3% wet of standard Proctor optimum). Following compaction a sample (C1) was taken for MIP testing. This sample is referred to as the “as-compacted” state in this paper.

In a series of experiments on the compacted material a monotonic increase in suction was imposed (stress path O→D in Figure 1). Eight samples (C2 to C9) were air dried from the as-compacted state to target water contents along the main drying curve (see Table 1). When dried to the desired water content samples were freeze dried for MIP testing immediately following suction measurement with the WP4.

To examine the microstructural evolution during saturation, five additional compacted samples, previously compressed to vertical stresses of 2, 20, 100, 200 and 600 kPa (C10 to C14, respectively) from the as-compacted state were subjected to a monotonic decrease in suction (by soaking). The stress path applied in this case is shown in Figure 1 as O→A→B. Samples were compressed at constant water content to the desired vertical stress in a lever arm oedometer. The suction reduction was achieved by filling the oedometer cell with water while maintaining the vertical stress. Samples were soaked until they came to volumetric

equilibrium based on a plot of square root time vs. displacement. Following rapid (undrained) unloading from the oedometer the samples were taken and freeze dried for MIP testing. The samples were rapidly unloaded in an attempt to minimise expansion that may have occurred during slow unloading – however it is inevitable that some expansion did indeed occur during this process. Following the same stress path as C10, a sample (C15) was soaked at a vertical stress of 2 kPa and removed from the lever arm oedometer. The sample was then mounted in the axis-translation oedometer and dried to a suction of 1200 kPa (stress path $O \rightarrow A \rightarrow B \rightarrow E \rightarrow D$, Figure 1).

4 EXPERIMENTAL RESULTS

The main drying branch of the water retention curve (WRC) for the undisturbed, reconstituted and compacted Maryland clay is shown in Figure 3 in terms of gravimetric water content vs. suction. Drying of the undisturbed material commenced from the in situ water content ($w=22.7-23.6\%$), while the reconstituted started directly from the reconstituted state ($w=50.6-56.6\%$) and the compacted from the as-compacted water content ($w=28\%$), respectively. The low suction range for the reconstituted specimens ($s<800\text{kPa}$ in R1, R5) and the compacted-saturated sample ($s<1200\text{ kPa}$ in C15) were measured using the axis-translation technique (i.e. matric suctions); for higher suctions ($s>1200\text{ kPa}$) the WP4 device (i.e. total suction) was used. An osmotic suction of 30 kPa was estimated based on electrical conductivity measurements of extracted pore-water from the reconstituted material. The monotonic drying process for the compacted specimens is indicated in Figure 1 as path $O \rightarrow D$, where O refers to the as-compacted state and D represents the suction for each specimen prior to MIP. For suctions less than 1 MPa there is some scatter to the results, related to limitations of the WP4 (reported accuracy in this range of $\pm 100\text{ kPa}$) and due to sensitivity of suction measurement with changes in void ratio. At high suctions, where the

water retention capacity is mainly controlled by the specific surface of the clayey fraction (Romero & Vaunat 2000), all three sample types converge to a unique linear semi logarithmic relationship between water content and suction. The compacted-saturated sample (C15) appears to approach a similar water retention relationship to that of the reconstituted material, even in the low suction range. A wide range of water content (suction) has been examined for the compacted material, indicated by the circles in Figure 3. The compacted samples which were soaked (C10-C14) and the initially saturated reconstituted sample (R1) are shown at a nominal suction of 1 kPa for the purpose of presentation in Figure 3. The vertical effective stress after soaking is shown in Figure 3 and projected on the suction axis (dashed lines). The water content following soaking at a vertical stress of 20 kPa closely aligns with that of the reconstituted sample on drying at a suction of 25 kPa. Likewise, the same relationship is observed for the higher vertical stresses and suctions.

The MIP results of two undisturbed samples (UD1 & UD2), and one undisturbed sample following air drying (UD1/1), are shown in Figure 4. The undisturbed material displays essentially a unimodal PSD with a dominant peak around a pore entrance diameter of 0.07 μm despite some evidence of larger pores greater than about 5 μm in diameter. Using the approach proposed by Delage and Lefebvre (1984) a diameter of approximately 0.07 μm separates micro and macro pores as shown in Figure 4. Qualitative analysis of the undisturbed microstructure in Figure 2, however, suggests that macro pores are significantly larger than this diameter and are better described as having pore sizes greater than 5 μm . A reduction in the dominant pore entrance diameter to 0.04 μm is observed for the UD1/1 subjected to drying. Minor reduction in the intrusion for pore sizes between 5 μm and 100 μm is also observed. This suggests that drying of the undisturbed material is mainly associated with the shrinkage of the micro voids.

Figure 5 shows the PSD of five reconstituted specimens (R1-R5). The PSD of specimen R1 shows an overwhelmingly dominant pore domain with a peak around 3 μm in diameter. There is also some evidence of a smaller domain around 0.1 μm in diameter, similar in size to that of the undisturbed sample (see Figure 4). Virtually no intrusion of mercury was recorded for pore entrance diameters in excess of 20 μm with the first significant intrusion occurring at a diameter of 10 μm . As discussed further below, 10 μm is used to separate micro and macro components. From the same reconstituted sample that was allowed to dry (R2) a large reduction in the total void ratio (see Table 1) and shrinkage of the dominant pore entrance diameter to 0.08 μm is observed. A reconstituted specimen (R3) was dried to a matric suction of 400 kPa under a mean net stress of 50 kPa (path E→F→G in Figure 1) and a specimen (R5) was dried to a suction of 800 kPa under a vertical net stress of 30 kPa. The dominant pore entrance diameter in this case shows a slight reduction from 3 μm to 1.5 μm for R3, while for R5 the dominant pore entrance diameter reduced to 0.7 μm . However, similarity in the shape of the PSD with R1 can still be seen for the two specimens. The cumulative intrusion (Figure 5a) for R5 is very similar to R3 where it was expected that some reduction in the total intruded void ratio would have been observed due to the higher applied suction. In addition, at the completion of test R3, a specimen, R4, was air dried (stress path E→F→G→D in Figure 1) prior to oven drying and MIP testing. In both cases following air drying (R2 & R4 in Figure 5) a single peak was observed in the PSD around a diameter of 0.07-0.08 μm with virtually no evidence of pore diameters greater than 0.2 μm . These results demonstrate the large reduction in pore size of the reconstituted samples that occurs on drying resulting in a pore entrance diameter similar to that of the undisturbed material.

The evolution of the PSD for compacted samples, subjected to a monotonic increase of suction, from the as-compacted state, was also investigated. The as-compacted suction was inferred to be around 200 kPa based on WP4 and axis-translation oedometer tests (Burton et

al. 2014a). Suction measurements were taken with the WP4 immediately prior to freeze drying as shown in Figure 3 (stress path O→D, Figure 1). The measured suction, water content, void ratio and degree of saturation for these samples is summarised in Table 1. Two pore domains can be discerned from the PSD function shown in Figure 6. Firstly, a large pore domain is observed with a dominant entrance diameter of 80 μm and, secondly, a smaller pore domain with a peak around 0.6 μm in diameter. The smaller peak is almost one order of magnitude larger than for the reconstituted and undisturbed material (0.07 μm). Following an initial shrinkage on drying from the as-compacted state C1 ($s \approx 200$ kPa) to sample C2/C3 ($s \approx 2160\text{--}2190$ kPa), the larger pore domain around 80 μm remains essentially unaltered in its initial shape. There is some variability (shrinkage followed by expansion between tests, for example in C3→C4, C6→C7, C8→C9) in the large pore domain range which may suggest some increase in the larger pore domain, or alternatively sample variability. However focusing on the progression of samples C1→C3→C6→C8, the overall inclination is for slight macro-pore shrinkage. Conversely, in the micro-pore domain the response is much easier to discern. The peak in the PSD for diameters less than 1 μm is referred to as the dominant micro-pore diameter in the following. Monotonic contraction of the dominant micro-pore diameter is observed with increasing suction. To highlight this phenomenon, the micro-pore domain is enlarged in Figure 7. In this figure, the progression of the dominant micro-pore diameter is tracked (indicated by arrows) for a number of the samples. These results are summarized in Table 1. The largest contraction in the dominant micro-pore diameter is observed from sample C1 ($s \approx 200$ kPa) at 0.4 μm to sample C2 ($s \approx 2160$ kPa) at 0.1 μm . Also illustrated in Figure 7 is the reduction in water content (shown as the grey fill beneath the PSD) as drying proceeds. The distribution of pore-water is estimated assuming that the pore-water is preferentially held within the micro-pores. As suction is increased from 8170 kPa to 11910 kPa (sample C5→C6, Figure 7c→d) shrinkage

of the dominant micro-pore diameter is largely completed. Drying to higher suctions, the dominant pore diameter remains relatively stable around $0.045\ \mu\text{m}$. This is comparable with the diameter reported for both the undisturbed and reconstituted material following air drying discussed previously. Below a pore entrance diameter of $0.045\ \mu\text{m}$ the PSD function of the compacted material on drying is not significantly altered and is very similar to the PSD obtained for the undisturbed and reconstituted samples.

In contrast, monotonic decrease of suction (soaking) was applied to another set of compacted samples (C10-C14). Figure 8 shows the MIP results obtained in each case following unloading from the oedometer. Schematically the stress path followed by these tests is shown in Figure 1 as $O \rightarrow A \rightarrow B$. In the net stress compression plane ($e : \log \bar{\sigma}_v$), shown also in the inset of Figure 8b, the void ratio after soaking approximates that of the normal compression line (NCL) for the reconstituted material at stresses of 100, 200 and 600 kPa. At 2 and 20 kPa the samples remain below the NCL. The PSD of C10 (2 kPa) in Figure 8b recovers a PSD function comparable to that of the reconstituted (R1). Soaking at higher vertical stresses also appears to recover a PSD similar to the reconstituted soil. Naturally however, samples soaked at higher stress exhibit a lower void ratio which is represented as a reduction in the cumulative intrusion (e_{MIP}) and a reduction in the peak of the PSD. The diameter of the peak in the PSD also appears to show some stress level dependency with a reduction in diameter for higher vertical stress (arrows shown in the figure). From the sample C13, a small sub-sample was oven dried to determine the water content and then tested with the MIP (C13/1). The stress path of this sample is given by path $O \rightarrow A \rightarrow B \rightarrow D$ in Figure 1. On oven drying pore entrance diameters larger than $0.2\ \mu\text{m}$ are erased whereas the dominant pore size is located around $0.04\ \mu\text{m}$, comparable with the pore size of the reconstituted and undisturbed following drying.

5 DISCUSSION

The experimental results described above have demonstrated that the PSD of samples subjected to monotonic drying display a distinctly different behaviour to that of samples brought to near saturation. Moreover, the microstructure of the compacted material after saturation and drying is vastly different to that of the material subjected only to monotonic drying. A common feature on the other hand, is the similarity of the small pore size domain located between 0.02 and 0.1 μm following drying, irrespective of the material preparation, hydraulic path or stress level applied.

The MIP results demonstrate the overwhelming influence of the constituent material (i.e. soil particles) on the water retention behaviour in the high suction range, rather than unimodal vs. bimodal or compacted vs. reconstituted. For example, the undisturbed material shows a distinctly different microstructural arrangement than that of compacted material. However, in the high suction range (see Figure 3) the water retention behaviour is almost identical on drying, as is the behaviour of the reconstituted soil. This behaviour suggests that the microstructure tends to be over ridden by the material characteristics, such as specific surface area as previously pointed out by (Romero & Vaunat 2000).

The reconstituted material provides a framework in which the PSD of the compacted and undisturbed material can be assessed. Comparison of the reconstituted and compacted specimens has revealed that the compacted PSD is simply a state of the soil (but not intrinsic), and that a bimodal PSD can exist for material compacted on the wet side of standard Proctor optimum water content. The initial bimodal distribution of the compacted PSD depends heavily on whether or not the compacted soil reaches a state of saturation or near saturation during compaction (Toll 2000; Delage et al. 2006; Merchán 2011).

Pore refinement, defined as the progressive reduction in pore sizes, is clearly evident in the undisturbed, reconstituted and compacted samples that are dried from near saturation. Pore refinement was also shown during compression of the reconstituted and compacted material by (Burton et al. 2014b). The initial threshold pore entrance diameter, defined as the diameter where the first significant intrusion of mercury occurs (obtained from a plot of cumulative intrusion versus pore size, e.g. Figure 4a), of the undisturbed material is approximately 1 μm (excluding the larger macro pores) and following air drying, the threshold reduces to 0.1 μm . However, as noted by Diamond (2000), the volume of mercury intruded at low diameters may in fact be a representation of larger pores which were not easily accessible. Pore refinement on drying is also observed for the reconstituted material. Initially the threshold diameter is approximately 10 μm , but following drying the threshold diameter evolved to 5 μm ($s=400\text{kPa}$), 3 μm ($s=800\text{kPa}$), and finally to a diameter of 0.2 μm on air drying. In comparison, compression of the reconstituted material presented by Burton et al. (2014b) showed that the threshold diameter reduced to 3-4 μm for a maximum vertical stress of 2000 kPa. These results indicate that drying paths, from high degree of saturation, can produce significant pore refinement.

In the compacted material on the other hand, two different types of pore refinement behaviour are observed depending on the degree of saturation prior to drying. From the as-compacted state, which begins with a low initial degree of saturation of 0.58, pore refinement is largely limited to the micro-pore region. Drying from a low degree of saturation was also shown by Diamond (1970) to produce negligible effect on the microstructure of natural samples. The distinctly bimodal structure of the as-compacted material on the other hand was erased as a result of soaking (saturation), in similar manner to that reported by Monroy et al. (2010). The microstructure post soaking was similar to the microstructure of the reconstituted material and related to the current stress state (e.g. the vertical stress at soaking as shown in

Figure 8). In contrast to samples dried from a low degree of saturation (i.e. C1-C9), the drying of samples with an initially high degree of saturation (UD1/1, R2, R4, C13/1 and C15) results in a different microstructural evolution. Drying from an initially high degree of saturation, results in a predominantly unimodal PSD as shown in Figure 9. Samples C13/1 and C15, following drying after saturation, were not able to recover the distinctly bimodal PSD of the as-compacted material (e.g. C1). Sample C15 ($S_r=1.0$) does however show some pore sizes between 10 and 45 μm , which would appear to be erased on further drying (e.g. to C13/1). It seems clear, at least for Maryland clay, that the bimodal PSD developed during compaction should be regarded as an initial condition (material state), that once lost cannot be recovered, at least when subjected to the stress paths described in this paper. This may be the case for other clayey soils with the absence of coarser material and should be validated with further experimental results. Similar results were also found for compacted MX80 bentonite by Seiphoori et al. (2014), for example.

From the experimental results in Figures 6 and 7, it appears that the dominant micro-pore entrance diameter of the compacted material (which is subjected to monotonic drying from the as compacted state) can be reasonably well tracked based on the measured suction, and the theoretical suction based on a capillary tube model. This suggests a relationship between micro-pore (entrance) diameter and the suction experienced by the soil. The Washburn equation (Eq. 1) can be used to estimate the suction (Romero 1999), based on the mercury injection pressure:

$$s = u_a - u_w = -\frac{\sigma_{H_2O} \cos \theta_w}{\sigma_{Hg} \cos \theta_{nw}} p \approx 0.196 p \quad (4)$$

where u_a is the pore-air pressure, u_w is the pore-water pressure, σ_{H_2O} is the water surface tension with $\cos \theta_w = 1$.

The interpreted dominant of the micro-pore diameter and measured suction are reported in Table 1 and plotted in Figure 10a. The suction calculated using Equation 4 is also shown in Figure 10a. A reasonable trend of the results up to suctions of 4150-8170 kPa is obtained. The contribution of osmotic potential is included, evaluated from electrical conductivity measurements on the pore water using the van-Hoff equation (see Mitchell & Soga 2005). Based on the measured water content, and assuming that pore-water is preferentially held within the micro-pores, a suction of 4150-8170 kPa corresponds closely to what might be defined as air-entry of the micro-pores as shown by the solid fill in Figure 7c & d. To the authors knowledge, the tracking of the micro-pore size behaviour has not previously been reported despite providing relevant information about the evolution of the soil microstructure. Drying from a low degree of saturation was also shown by Diamond (1970) to produce negligible effect on the microstructure of natural samples. Although the capillary tube model is a simplification of the true microstructure of soil it appears reasonable for characterising the size of the saturated (dominant) micro-pores within the soil.

The differentiation of the types of porosity, between micro and macro, can be somewhat of a subjective process. The reconstituted microstructural results demonstrate that two domains can be observed, indicating that a unimodal (or uniform) pore size may not accurately describe the pores within saturated material. The approach followed to separate micro and macro porosity involves the examination of the PSD to determine a pore entrance size that is bounded by the dominant pore domains (i.e. selecting the intermediate low point of a bimodal PSD as the delimiting pore size). In this case a delimiting pore size of 0.3 μm might be adopted for the reconstituted material. Alternatively, where the intrusion and extrusion portions of the MIP results are available, Delage and Lefebvre (1984) proposed that the micro-pores are represented by the reversible intrusion/extrusion of mercury. If this method is used to differentiate micro and macro pore entrance diameters as shown in Figure 4, a

limiting pore entrance size of $0.07\ \mu\text{m}$ is found for the undisturbed material. In both cases it would appear that these methods provide an inconsistent representation of micro and macro pores based on the interpretation of the SEM images in Figure 2 of the undisturbed material. Romero et al. (2011) recently proposed an alternative method which involves saturating compacted samples, either under constant volume or by free swelling. This produced a single (unique) dominant peak of the PSD function obtained. Pores sizes less than the peak were then assigned to micro, and larger than to the macro. If this method is adopted, the delimiting pore size is $6\ \mu\text{m}$ for the sample soaked at a vertical stress of $2\ \text{kPa}$ (C10), or around $3\ \mu\text{m}$ would appear appropriate based on the reconstituted (R1, with a vertical stress of $50\ \text{kPa}$). The data presented on the reconstituted soil, and the evolution of the peak diameter following soaking at increasing stress highlights some stress level dependency in the diameter of the peak in the PSD.

However, as mentioned by Romero et al. (2011), from the single peak observed after saturation it is not possible to distinguish between micro and macro pores. Romero et al. (2011) then proposed that pore sizes less than the peak could be assigned to micro, and those above assigned to macro. Selection of the peak of the PSD, although it appears unique, may not be the only possibility. Given the logarithmic scale used in the presentation of the results, the selection of a delimiting pore size can significantly affect the decomposition of void ratio into micro and macro components, $e = e_m + e_M$, where e_m is the micro void ratio and e_M is the macro void ratio.

In order to evaluate the influence of the method used for differentiating micro and macro pore levels two delimiting pore sizes have been used in this paper. Firstly, $3\ \mu\text{m}$ based on the peak in the PSD of the reconstituted material and secondly $10\ \mu\text{m}$ based on the threshold diameter of the reconstituted, discussed above. It is worth noting that the adoption of $10\ \mu\text{m}$ for

differentiating micro and macro pores is also consistent with the size of the macro pores observed in SEM images for undisturbed soil (see Figure 2a). The resulting comparison is shown in Figure 11 and Figure 12 in terms of void ratio vs. suction and void ratio vs. water ratio ($e_w = wG_s$). Specimens C10-C14, subjected to soaking at different stress levels, are plotted using a nominal suction of 1 kPa whereas the vertical stress applied during soaking is highlighted by dashed lines. As observed in Figure 11b and Figure 12b the void ratio attained at the end of soaking locates close to the saturation line which is also followed by specimen R3 during drying.

Adopting 3 μm as a delimiting pore size limited values of e_m to less than about 1.0 (see Figure 11). The comparison based on a 3 μm pore size would indeed suggest the need for a parameter, β , as proposed by Romero et al. (2011) to capture the evolution of the micro void ratio. However, if the analysis is based on 10 μm , higher values of e_m are allowed which in this case display 1:1 relationship for water ratios higher than 0.5 (Figure 12). It is worth noting that the deviation of e_m from the 1:1 line at low water ratio indicates commencement of micro-pore desaturation. Based on the results shown in Figure 11 and Figure 12, the suction corresponding to this is around 4000 kPa, in agreement with the analysis described previously in Figure 7 in terms of the dominant micro pore size. It seems that the alternative approach to separating micro and macro, based on the threshold diameter, reduces the need for introduction of an additional parameter. The main aspect to highlight from the inspection of Figure 11 and Figure 12 is that the evolution of e_m for the undisturbed, reconstituted and compacted material appears to be independent of the initial state.

To complement the analysis on Maryland clay above, additional experimental results where both reconstituted and compacted MIP test results were presented, have been analysed. MIP test results on London clay, speswhite kaolin and Boom clay were presented by Monroy

(2006), Tarantino and De Col (2008) and Merchán (2011), respectively. Material characteristics are summarised in Table 2. Monroy (2006) tested both compacted and reconstituted material with the delimiting pore size of $3\text{ }\mu\text{m}$ being selected based on the reconstituted sample. Compacted speswhite kaolin results were presented by Tarantino and De Col (2008) while results on reconstituted material following some initial drying were presented by Tarantino (2011). Based on the reconstituted results the delimiting pore size was estimated to be approximately $0.3\text{ }\mu\text{m}$. Compacted and remoulded samples of Boom clay were tested by Merchán (2011) and the delimiting pore size was selected based on samples remoulded to be around $1\text{ }\mu\text{m}$. The resulting separation into micro and macro components for each of these three materials is shown in Figure 13. Similar to the results on Maryland clay, selecting the delimiting pore size based on the reconstituted/remoulded samples suggests that the micro void ratio closely follows the 1:1 line as shown in Figure 13 until close to the shrinkage limit, at which further reduction in the micro void ratio ceases. Although the calculated e_m closely follow the 1:1 relation, they tend to be slightly below the line (i.e. $e_w > e_m$). This is consistent with a small percentage of the water being held within the macro-pores as required for the formation of menisci (“liquid bridges”) between the aggregates and connectivity of the pore-water phase.

The decomposition of void ratio into micro and macro components, based on the procedure proposed in this paper, can then be simplified as it only requires knowledge of the reconstituted void ratio behaviour (from a shrinkage test, for example). The reconstituted void ratio represents the micro void ratio while the macro void ratio is the void ratio in excess of the reconstituted. A similar type of analysis is used by some researchers for structured soils, where the void ratio is decomposed into the reconstituted and that void ratio in excess of the reconstituted (e.g. Liu & Carter 2002). . Using the reconstituted material to separate micro and macro components provides some efficiency (i.e. not requiring extensive MIP test

results) for the application of advanced (double structure) constitutive models in engineering practice.

6 CONCLUSIONS

The microstructure of undisturbed, reconstituted and compacted Maryland clay has been examined. The reconstituted soil (both saturated and after air drying) was found to provide a useful reference with which to interpret the results of MIP tests on compacted samples as was suggested by Griffiths and Joshi (1989). Good agreement between undisturbed (dried), reconstituted (dried) and compacted-saturated (dried) samples is observed in the micro-pore range. For compacted samples that are initially unsaturated on drying, the PSD function does not match that of samples which are dried from saturation, indicating a path dependency on the evolution of the PSD.

The dominant micro-pore entrance diameter of the compacted material is shown to be related to the imposed suction (effective stress) up to air entry. After saturation, the dominant pore size of the compacted material is shown to coincide closely with that of the reconstituted material taking into account some stress level dependency. As the suction level is increased, pore refinement occurs, and the peak pore entrance diameter is reduced. The as-compacted structure is erased on saturation.

The evolution of the micro void ratio with water ratio and suction has been examined in conjunction with discussion of methods to separate micro and macro. An alternative approach is proposed, where the threshold pore entrance diameter of the reconstituted soil is used as the delimiting diameter. Using this approach, the similarities between undisturbed, reconstituted and compacted material were highlighted.

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Fig. 1

Fig. 2

Fig. 3

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Fig. 6

Fig. 7

Fig. 8

Fig. 9

Fig. 10

Fig. 11

Fig. 12

Fig. 13

Table 1: Measured suction, water content, void ratio, degree of saturation and dominant micro-pore diameter for MIP test samples.

Sample	Suction (kPa)	Water Content (%)	Void ratio	Degree of saturation	Dominant micro-pore entrance diameter (μm)
UD1	1180	23.65	0.594	1.0	0.075
UD1/1 (UD1 oven dried)	-	-	0.484	-	0.04
UD2	1300	22.74	0.595	1.0	0.075
R1 “Reconstituted”	30 ¹	54.6	1.448	1.0	3
R2 (R1 oven dried)	-	-	0.421	-	0.08
R3	400	31.82	0.856	0.985	1.5
R4 (R3 oven dried)	-	-	0.414	-	0.06
R5	800	25.9	0.828	0.95	0.7
C1 “As-compacted”	200	28	1.27	0.58	0.4
C2	2160	19.4	0.986	0.52	0.1
C3	2190	19.5	0.98	0.53	
C4	4150	17.5	0.919	0.5	0.08
C5	8170	15	0.878	0.45	0.055
C6	11910	12.9	0.89	0.38	0.045
C7	18050	11.1	0.94	0.31	0.045
C8	27470	9.1	0.83	0.29	0.045
C9	44370	7.4	0.97	0.2	0.045
	Vertical stress at soaking (kPa)	Water Content (%)	Void ratio	Degree of saturation	Dominant macro-pore entrance diameter (μm)
C10	2	46.26	1.369	0.895	6
C11	20	44.26	1.253	0.938	3.5
C12	100	37.02	0.994	0.986	2.6
C13	200	34.23	0.911	0.995	1.6
C13/1	200	-	0.459	-	0.04
C14	600	28.48	0.764	0.987	1.2
C15	2 (s=1200)	23.6	0.625	1.0	0.3

1. An osmotic suction of 30 kPa was measured based on electrical conductivity measurements.

Table 2: Material properties

Soil	Liquid limit	Plastic limit	Specific gravity	Delimiting pore size [μm]
Boom clay	55% $\pm$ 2%	28% $\pm$ 2%	2.70	1
London clay	83%	29%	2.70	3
Speswhite kaolin	64%	32%	2.60	0.3

Highlights

Microstructural changes of an undisturbed, reconstituted and compacted high plasticity clay subjected to wetting and drying

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Manuscript ENGEO6603 Highlights

- MIP results on undisturbed, reconstituted and compacted Maryland clay have been presented.
- During monotonic drying, the dominant micro-pore diameter is tracked with suction.
- As-compacted microstructure is shown to be erased after saturation.
- A procedure for separating micro and macro components of void ratio has been proposed.