

DISCUSSION

Compaction behaviour of clay

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The authors have to be congratulated for their significant contribution to the investigation of the behaviour of compacted soils: a geotechnical problem of great importance, compacted soil being the building material that is most used in terms of volume in civil engineering. The authors fruitfully made use of relatively recent advances made in the mechanics of unsaturated soils in terms of both experimental investigation and constitutive behaviour, also including microstructure aspects. The following comments, related to microstructure, aim at completing and expanding on some of the interesting features underlined in the paper and related to the combined effect of water content and density on the microstructure of compacted samples.

Considering sample dehydration prior to mercury intrusion porosimetry (MIP), an interesting alternative to the use of isopentane quickly to freeze the sample with liquid nitrogen is, as suggested by Tessier (personal communication, 2002), to submit nitrogen to vacuum for a number of minutes to decrease its temperature from the boiling point (-193°C) down to its freezing point (-210°C) as done, for instance, in Delage *et al.* (2006). Once vacuum is applied, the appearance of frozen nitrogen is similar to that of isopentane, the nitrogen surface progressively becoming white with increased consistency. Plunging small pieces of samples in frozen nitrogen occurs without boiling, optimising the freezing speed thanks to a lower temperature than with frozen isopentane. Indeed, this improvement appeared to simplify the freeze-drying procedure.

In Fig. 23, the authors compare the pore size distribution (PSD) curves of two samples of same water content ($w = 25.9\%$) compacted at two different void ratios ($e = 1.136$ and 0.993) corresponding to vertical stresses equal to 600 kPa and 1200 kPa respectively. It seems worth recalling that some interesting data concerning the changes in PSD of samples of kaolinite of same water content ($w = 21\%$) compacted at various void ratios were provided by Sridharan *et al.* (1971). The kaolinite used had a plastic limit $w_p = 26$ and a liquid limit $w_l = 62$, close to the values of the kaolinite used by the authors ($w_p = 32$ and $w_l = 64$). Sridharan *et al.* performed MIP tests on statically compacted samples at five values of void ratio smaller than that of the authors, that is between 0.85 and 0.56 (0.85, 0.74, 0.69, 0.59 and 0.56 with corresponding degrees of saturation S_r equal to 64, 74, 79.7, 93 and 100% respectively). Unfortunately, Sridharan *et al.* neither gave the corresponding vertical compaction stresses nor presented any MIP pressure release curve illustrating the non-constricted porosity as suggested by Delage & Lefebvre (1984).

Like the authors, Sridharan *et al.* observed that the decrease in void ratio owing to increased static compression only affected the coarser pores with no effect on the PSD of finer pores. Although the microstructure of soils compacted dry of optimum (including a kaolinite) had been described by Diamond (1970) as ‘a domain structure with adjacent domains largely separated by micrometer-sized interdomain voids’ (domain being another term for aggregate), Sridharan

et al. did not refer to any aggregate microstructure in their interpretation of the nature of the pores. The results of Sridharan *et al.* can be redrawn as shown in Fig. 26 in which cumulative PSD curves are presented together with the pore volume expressed in cm^3/g , a unit that permits direct comparison with water content. In Fig. 26, curves are superimposed in the zone of smallest porosity, an area in which PSD curves are very similar. Also represented is the total porosity of each sample that shows that, as observed by the authors, mercury filled almost all the voids at maximum applied pressure. This is not the case in compacted smectites in which a significant amount of intra-aggregate porosity too small to be detected by MIP is observed (Marcial, 2003; Lloret *et al.*, 2003; Delage *et al.*, 2006). Unlike smectites, kaolinite particles, made up of stacks of elementary kaolinite layers, are stable and do not change with changes in water content (Mitchell & Soga, 2005). The size of the smallest pores inside the aggregates is hence linked to the thickness of the particles that are in the range of the smallest pores intruded by mercury. In Fig. 26, the water content of 21% is plotted as a horizontal line, supposing that, as the authors did in Fig. 24, water saturates the smallest pores owing to strongest capillary actions.

The PSD curves at higher void ratios (0.69, 0.74 and 0.85) clearly exhibit bimodal pore distributions typically defining both intra-aggregate and inter-aggregate pore populations. Interestingly, the 21% water content seems to correspond reasonably with the intra-aggregate porosity. Inter-aggregate pores are apparently not affected by the decrease in total pore volume, with no significant change observed in the PSD curves below $0.40\ \mu\text{m}$. Fig. 26 clearly shows a phenomenon similar to that described by Delage & Lefebvre (1984) on a saturated sensitive clay, with all MIP cumulative curves superimposed along a wide range of pore sizes (including both intra- and inter-aggregate porosities) with the decrease in porosity only concerning the largest pores full of air. In other words, compression only affects the section of the PSD cumulative curve that corresponds to

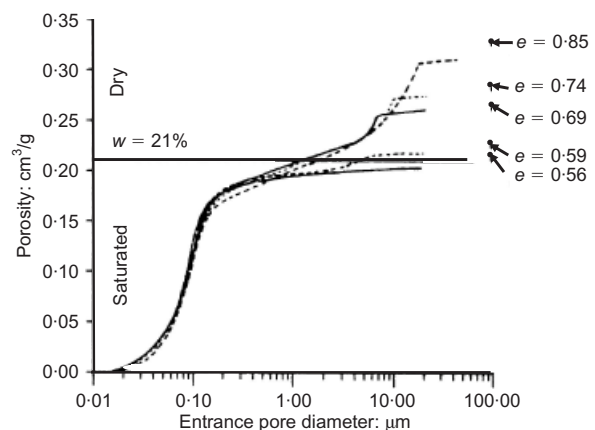


Fig. 26. PSD curves of samples compacted at various density with same water content (after Sridharan *et al.* (1971))

the largest pores. According to Fig. 26, compression between 0.85 and 0.69 only affects the pores with entrance diameters larger than $5\text{ }\mu\text{m}$. As shown by the position of the 21% water content line, compression progressively increases the degree of saturation until reaching almost full saturation at $e = 0.56$. At smaller void ratios and higher degrees of saturation ($e = 0.59$ and 0.56 corresponding to $S_r = 0.93$ and 1.00% respectively) in a zone where air continuity probably no longer exist, the shape of the PSD curves is different, with a unimodal curve observed at $e = 0.56$ and $S_r \approx 1.00$.

The same observation can be made by presenting the data of Fig. 23 in the same way, as shown in Fig. 27. This figure shows that compression from 600 kPa to 1200 kPa of a sample with $w = 25.9\%$ only erases the pores larger than $0.8\text{ }\mu\text{m}$, with no effect on smaller pores, neither inter-aggregates nor intra-aggregates. The collapse of the largest existing pores with no effect on smaller pores is also observed. This is typical of the behaviour of a rigid porous matrix formed by the assembly of aggregates that only collapse at the weakest points, that is the largest pores. This feature has been numerically modelled by Cerrolaza & Delage (1997) who, based on the changes in PSD curves and this micro-mechanism, obtained a satisfactory modelling of the macroscopic compression curve. This mechanism provides complementary information with respect to the decrease of the modal size from 0.8 to $0.7\text{ }\mu\text{m}$ noted by the authors.

Another important result presented by the authors comes from the description of the various PSD curves of compacted samples (Fig. 24) with various water contents submitted to the same compaction stress (1200 kPa). By plotting the PSD curves as done previously, the graph presented in Fig. 28 is obtained. This presentation clearly shows the progressive changes in the shape of the PSD curves when considering samples compacted at the same compaction stress with water content decreasing from 0.311 down to 0.081. Unsurprisingly, compaction of drier samples at the same vertical stress result in looser samples characterised by larger total intruded porosities, the wetter samples (with lower suction) being more compressible than the drier ones (with higher suction, see Fig. 10). In all curves, the e_w values corresponding of the water saturated void ratio are also represented.

When considering samples with decreasing water contents, the change in shape of the curves is progressive and well ordered with, as shown by the curved arrows also plotted in the figure, a regular rightwards displacement of the curves in the small porosity area coupled with a leftwards displacement of the curves in the large pores area. This shows that, with decreased water content, the modal entrance pore

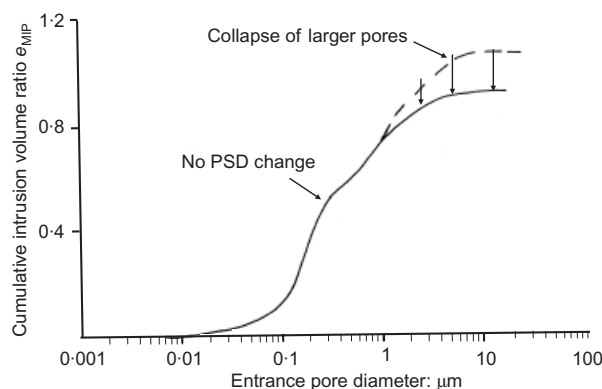


Fig. 27. PSD curves of samples compacted at various density with same water content at two densities (after Tarantino & De Col, 2008)

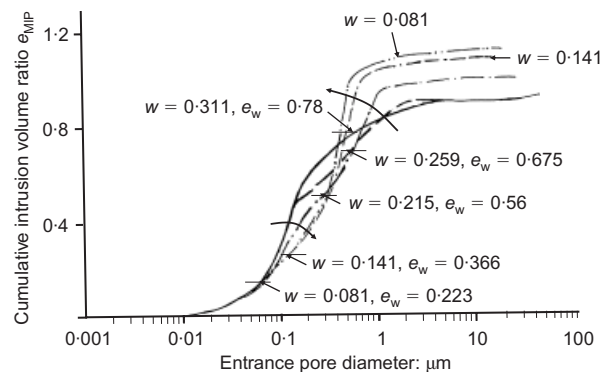


Fig. 28. PSD curves of samples compacted at various water contents under the same 1200 kPa vertical stress (after Tarantino & De Col, 2008)

diameter of the intra-aggregate porosity increases whereas that of the inter-aggregate porosity decreases, finally resulting in a well-graded unimodal PSD curve. The sample at $w = 0.311$ with a degree of saturation close to 0.9 (see Fig. 9) has a PSD curve more typical of samples compacted 'wet of optimum' (Ahmed *et al.*, 1974, Delage *et al.*, 1996) with around 40% of the total porosity being poorly graded between $3.5\text{ }\mu\text{m}$ (beginning of mercury intrusion in the sample through the largest inter-aggregate pores) and $0.15\text{ }\mu\text{m}$ (beginning of the intra-aggregate mercury intrusion according to Fig. 24(a)). A typical bimodal PSD curve is observed in the sample at $w = 0.259$ ($S_r \approx 0.75$), showing a trend typical of 'dry' compacted sample. The bimodal nature of the PSD curve is kept at $w = 0.215$, whereas it disappears with the two driest samples ($w = 0.141$; $S_r \approx 0.32$ and 0.081 ; $S_r \approx 0.18$) that present a unimodal shape.

In the two latter PSD curves, the beginning of mercury intrusion is very clearly defined with entrance diameters of 0.6 and $0.45\text{ }\mu\text{m}$ respectively. These diameters are significantly smaller than that of wetter samples. The unique well-graded pore population defined in the PSD curves (with modal entrance diameters close to 0.43 and $0.35\text{ }\mu\text{m}$ respectively) seems to be typical of very dry compacted samples. These samples have apparently been less frequently considered in terms of microstructure in the literature. According to Fig. 10, the corresponding suctions are close to 3000 and 5000 kPa respectively. To interpret the sample microstructures further, more information about the initial size of the clods obtained when wetting the powder prior to compaction would be helpful, and the authors probably have some comments to add in this regard. Scanning electron microscope observation should obviously be quite useful as well. In a first approach, it is suggested as a possible interpretation that the smaller entrance diameters measured in drier samples at the inter-aggregates level correspond to a denser assemblage of smaller grains made up of aggregated kaolinite particles strongly stuck by high levels of suction. It is likely that these grains have a fragile behaviour and may break into smaller grains during compaction. The question as to whether the aggregate microstructure is still observable in such samples arises, since no apparent distinction between intra-aggregate and inter-aggregate is observed.

The point of view of the authors about these points is certainly welcome.

Authors' reply

We wish to thank Professor Pierre Delage for the interest in our paper and for stimulating the discussion on the microstructure of compacted clay.

Concerning the experimental technique for sample dehydration, we performed our tests prior to the publication of

Delage *et al.* (2006) and were not aware of this improvement. We used liquid isopenthan to avoid boiling all around the immersed sample as discussed by Delage *et al.* (1996). The excellent comparison between the void ratio measured using Mercury intrusion porosimetry (MIP) and the void ratio measured in a traditional way by measuring the volume and water content of the sample from which the specimen for MIP was taken (Fig. 24) shows that the use of isopenthan was successful for the non-active clay that we investigated. However, we recognise that the procedure suggested by Professor Delage is simpler and may be important for dehydration of active (sensitive) clays.

The second point discussed by Professor Delage concerns the effect of compactive effort on pore size distribution. He reinterpreted data by Sridharan *et al.* (1971) by superposing the cumulative pore size distributions in the range of low pore-entrance size. The pore size distributions (PSD) associated with five compactive efforts clearly show that compaction at constant water content occurs at the expenses of the large inter-aggregate pores. This is an important complement to our data as only PSDs for two compactive efforts were investigated. We do not have any specific comment on this point apart from thanking Professor Delage for integrating our paper with this significant piece of information.

The last point discussed by Professor Delage is the effect of compaction water content on pore-size distribution. He replotted our MIP data in terms of cumulative pore size distribution again by superposing the curves in the range of low pore-entrance size. It was observed that the pore size at which mercury starts intruding the soil is lower in drier specimens ($w = 0.081$ and $w = 0.141$) than in wetter specimens (for example $w = 0.311$). He suggested a possible interpretation of this phenomenon and raised the question on whether an aggregate structure still exists at lower water contents.

We have a different interpretation about the lower initial intrusion pore size in drier specimens and perhaps an answer to the question raised by Professor Delage about aggregation at lower water contents.

To prepare the samples, we spread air-dried kaolin powder over a large surface, sprayed it with demineralised water and hand-mixed the moistened powder. Aggregation occurred upon water spraying, the size of the aggregates depending on the final water content. At high water contents ($w > 0.30$) very large aggregates formed and, when sieving the moistened powder on the sieve having 1 mm aperture size, about 20–30% of the material was retained on the sieve. The sieved material still had large aggregates as shown in Fig. 29 and, hence, there was a large amount of intra-aggregate pores as shown in Fig. 25 ($w = 0.311$ at $d \sim 0.2 \mu\text{m}$). The aggregate size was well distributed in the range $d_{\text{aggregate}} < 1 \text{ mm}$ as shown in Fig. 29, and this gave rise to a relatively well-graded inter-aggregate pore size distribution as shown in Fig. 28 (curve $w = 0.311$ at $d > 0.2 \mu\text{m}$).

As water content was decreased, the size of the aggregates forming upon spraying decreased as shown in Fig. 29. At water contents lower than 0.22–0.24, almost all the moistened powder passed through the 1 mm aperture size sieve. The decrease of the aggregate size resulted in a decrease in the amount of intra-aggregate pores as shown in Fig. 25 ($w = 0.215$ and $w = 0.259$ at $d \sim 0.2 \mu\text{m}$). As the largest aggregate size decreased, the inter-aggregate pore size distribution tended to become more uniform, as shown in Fig. 28 ($w = 0.215$ and $w = 0.259$ at $d > 0.2 \mu\text{m}$).

Eventually, at very low water contents ($w = 0.086$ and $w = 0.141$), there was no or little aggregation as shown in Fig. 29. It is interesting to note that aggregation at very low water contents ($w = 0.10$), as observed by visual inspection,

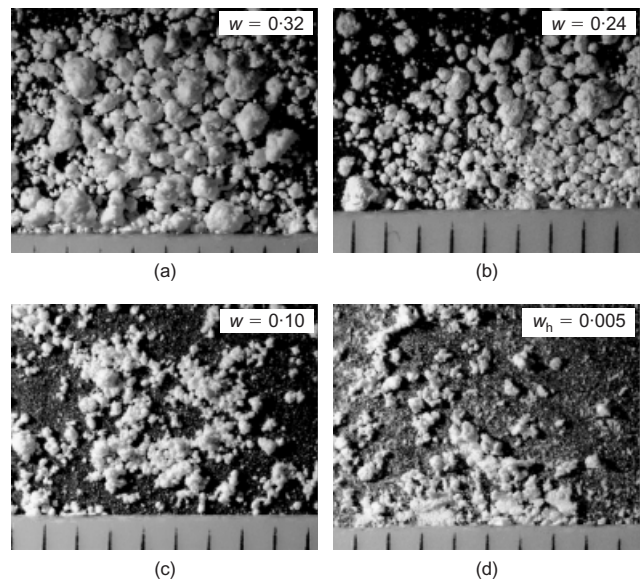


Fig. 29. Aggregation forming upon water spraying to different water contents as observed in the optical microscope (ticks are 1 mm spaced)

does not seem to differ significantly from that occurring to the kaolin powder under hygroscopic conditions as shown in Fig. 29. The hygroscopic water content w_h is here defined as the water content of the soil in equilibrium with laboratory air at relative humidity of 40–50%.

At very low water contents, the pores were probably inter-particle pores. The cumulative pore size distribution was uniform (Fig. 28) as is the grain size distribution. In other words, the small pore size at which mercury first intrudes the samples compacted at lower water contents ($w = 0.086$ and $w = 0.141$) would be associated with the small pores between the quasi-dry particles. In contrast, at higher water contents, the elementary unit is not the particle but the aggregate. The larger the aggregates, the larger will be the pores and, hence, the pore size at which mercury would start intruding the sample.

Nonetheless, we agree with Professor Delage that scanning electron microscope will prove invaluable in elucidating this issue.

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