Geomechanics and Engineering, *Vol. 20, No. 6 (2020) 537-546*
DOI: https://doi.org/10.12989/gae.2020.20.6.537

Geomechanical properties of synthesised clayey rocks in process of high-pressure compression and consolidation

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(Received September 11, 2018, Revised February 19, 2020, Accepted February 20, 2020)

Abstract. Oil and natural gas reserves have been recognised abundantly in clayey rich rock formations in deep costal reservoirs. It is necessary to understand the sedimentary history of those reservoir rocks to well explore these natural resources. This work designs a group of laboratory experiments to mimic the physical process of the sedimentary clay-rich rock formation. It presents characterisation results of the physical properties of the artificial clayey rocks synthesized from illite clay, quartz sand and brine water by high-pressure consolidation tests. Special focus is given on the effects of illite clay content and high-stress consolidation on the physical properties. Multi-step loaded consolidation experiments were carried out with stress up to 35 MPa on mixtures constituting of the illite clay, quartz sand and brine water with five initial illite clay contents (w=85%, 70%, 55%, 40% and 25%). Compressibility and void ratio were characterised throughout the physical compaction process of the mixtures constituting of five illite clay contents and their water permeability was measured as well. Results show that the applied stress induces a great reduction of clayey rock void ratio. Illite clay contents has a significant influence on the compressibility, void ratio and the permeability of the physically synthesized clayey rocks. There is a critical illite clay content w=70% that induces the minimum void ratio in the physically synthesised clayey rocks. The SEM study indicates, in the high-pressure synthesised clayey rocks with high illite clay contents, the illite clay minerals are located in layers and serve as the material matrix, and the quartz minerals fill in the inter-mineral pores or are embedded in the illite clay matrix. The arrangements of the minerals in microscale originate the structural anisotropy of the high-pressure synthesised clayey rock. The test findings can give an intuitive physical understanding of the deep-buried clayey rock basins in energy reservoirs.

Keywords: artificial clayey rocks; property characterisation; low permeability; clay content; porosity reduction

1. Introduction

The clayey rock is a typical porous medium that has been under investigation worldwide in mining, underground waste disposal, and petroleum and natural gas engineering. The clayey rock is a heterogeneous porous material constituted by the minerals such as clay mineral, quartz and calcite, etc. (Robinet 2008) . Its mechanical behaviours and physical properties are closely related to its microstructure and mineralogical compositions. Since the clayey rock is a naturally formed material, its microstructure and mineralogical composition are divergent and complex due to the variation of the sedimentary history and field geological conditions(Armand *et al.* 2017b, Liu *et al.* 2018b) . In this context, fully controlled consolidation experiments provide an alternative effective way to characterise the physical properties of deep-buried clay rich rock formations by mimicking the formation of the clayey rocks from its constituent minerals.

The formation of clayey rocks in nature involves a complicated consolidation process that is difficult to fully realize in laboratory due to the uncertainty of the recognised geological history. During the cementation of the constituents of the clayey rocks under consolidation, physical, chemical and combined reactions can be involved simultaneously (Akgün *et al.* 2015, Chai *et al.* 2016, Hamidi and Marandi 2018) . These reactions are closely related not only to mineralogical composition of the clayey rocks but also to stress conditions (such as stress history and stress path). Experimental investigations have announced and confirmed again that the properties of the synthesised materials are dependent on the type and amount of the raw materials as well as the method of consolidation (Chai *et al.* 2016, Hamidi and Marandi 2018, Koochak Zadeh *et al.* 2016, Mollins *et al.* 1996, Park *et al.* 2018, Sun *et al.* 2015) . As a result, understanding the formation of the clayey rocks should at first require progressive investigations on consolidation process, e.g., physical consolidation.

The compressibility of the clayey rocks is commonly revealed with the help of the compression curve obtained through the consolidation test by means of oedometer device. Void ratio is to describe the variation of compression deformation by the compression curve and also to directly reflect the deformation stiffness of the material. As mentioned previously, both physical and chemical reactions can result in decrease of void ratio.

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http://www.techno-press.org/?journal=gae&subpage=7
ISSN: 2005-307X (Print), 2092-6219 (Online)

Among all physical reactions, physical compaction is one of the simplest and most representative manner to induce void ratio decrease with depth and stress history in the clayey rocks (Armand *et al.* 2017a, Koochak Zadeh *et al.* 2016, Liu *et al.* 2016) . Earlier experimental investigations have been performed on the one-dimensional consolidation properties of some engineering clayey soils under different vertical stress (Di Maio *et al.* 2004, Djéran-Maigre *et al.* 1998, Engelhardt and Gaida 1963, Karig and Hou 1992, Marcial *et al.* 2002, Rieke and Chilingarian 1974, Shang *et al.* 2014) . The void ratio and consolidation parameters were reported to have correlations to the applied vertical stress (Akgün *et al.* 2015, Chen *et al.* 2017, Sobti and Singh 2017, Ye *et al.* 2014) . The correlation functions were reported to be linear in the low stress regime and non-linear in high vertical stress regimes. For example, Linear characters have still been observed from the compression curve (void ratio vs logarithm of consolidation pressure, i.e., e-lgσ_v) for some remoulded and natural clayey soils in the range of pressure from 10 to 60 MPa (Di Maio *et al.* 2004, Shang *et al.* 2014, Djéran-Maigre *et al.* 1998, Vasseur *et al.* 1995). Instead of that, nonlinear characters with decreasing rate of slope of the compression curve have been presented (Marcial *et al.* 2002, (Baille *et al.* 2010, Tripathy and Schanz 2007). Moreover, the slope of the compression curve for the sandy clay decreases with increasing consolidation pressure, whereas the slope gradually increases for the sand soil when consolidation pressure is above 1.6 MPa (Karig and Hou 1992). It can be noted that the characters of the compression curve are obviously dependent on the properties of clayey soils, especially on mineralogical composition. These experimental data provided good references for understanding the mechanical compressibility of the engineering clayey soils. However, the effects of mineralogical compositions were not well considered in those former works due to the presence of uncertainty in mineralogical compositions of the engineering clayey rocks. Thus, as a supplementary, further laboratory consolidation tests, especially under high pressures, were needed to investigate the effects of the original mineralogical compositions.

In classical soil mechanics, the compression curves of all types of clayey soils are considered to be characterized by a linear function when the vertical pressure applied is above a critical value, and the compression curves of a specific clayey soil with different disturbance degrees all pass through the specific point e=0.42e_0 (e_0 is the void ratio of the undisturbed soil in situ). Such feature is very useful to gain some important information on the clayey soil studied such as the pre-consolidation pressure and the field compression curve proposed by Casagrande. However, experimental studies have shown that the initially isotropic clay soil with large void ratio prepared by the slurry method (Howell *et al.* 1997) became an anisotropic one at high consolidation pressures (Djéran-Maigre *et al.* 1998, Shang *et al.* 2014) . Moreover, bilinear characters have been observed from the compression curve. The slope of the compression curve in the range of low pressure is greater than the one in a high-pressure range (Cotecchia and Chandler 1997, Dewhurst *et al.* 1996, Li *et al.* 2006, Marcial *et al.* 2002). Sometimes, nonlinear characters can also be observed from the compression curve of clayey soils as mentioned above. Therefore, conclusions obtained from the compression curve in the range of low pressure can’t be extend directly to the range of high pressure. The physical properties of the synthesised clayey geo-materials strongly depend on the imposed pressure. The physical properties during the low pressure and high-pressure consolidation were different. In addition, mechanical compaction of sand/clay mixtures is also strongly dependent on the expansibility of clay mineral (Cui *et al.* 2008, 2010, Koochak Zadeh *et al.* 2016, Mollins *et al.* 1996, Mondol *et al.* 2008, Revil *et al.* 2002, Shackelford *et al.* 1997, Sun *et al.* 2009). The content of expansive clay mineral was found to influence greatly the physical behaviours of the sand/clay mixtures. There is perhaps a critical clay content to induce a minimum porosity during mechanical consolidation of the clay rich soils, inspired by the works (Vallejo and Mawby 2000, Abichou *et al.* 2002, Kenney *et al.* 1992). However, the data obtained from the artificial soils may be not valid for artificial clayey rocks obtained by high-pressure consolidation. The validation has not fully confirmed yet due to the unavailability of sufficient experimental data. Thus, more experiments are in need to confirm the existence of a critical clay mineral content inducing minimum porosity of the synthesised clayed rocks. Moreover, swelling is a basic property of expansive clay minerals (Liu *et al.* 2020, Yang *et al.* 2013, 2018, Liu *et al.* 2018a, Roberts 1990, Zhang *et al.* 2013) . This effect could also affect the physical consolidation process of the clay materials and its microstructural change, which should be considered in laboratory tests. Thus, it is necessary to have a prescribed mineral composition to investigate the effects of the mineral compositions and the imposed stress.

With the above considerations, this paper is devoted to a laboratory characterization of the effect of vertical stress and illite clay contents on the physical properties of the clayey rocks synthesised with prescribed compositions of raw minerals. The synthesised samples are artificially made in laboratory in an autonomous high-pressure oedometer from the illite clay, quartz sand and brine water. This work puts emphasis on the physical property characterisation during the consolidation process of the clayey rocks from its two major constituent minerals. Further work will focus on the consolidations with combined physical and chemical reactions.

2. Materials and sample preparation

2.1 Raw materials

The artificial clayey rock was obtained in laboratory by consolidation of the mixtures of the pure illite clay, pure quartz sand and brine water. Illite other than bentonite was used in this study to avoid as much as possible the swelling effect of the clay minerals with water interaction (Cui *et al.* 2010, Sobti and Singh 2017, Sun *et al.* 2015) . In addition, the brine other than pure water also was used for the reason of abating the swelling effect of the clay mineral with water interaction due to the present of Na cation (Baille *et al.*

Table 1 Basic physical properties of the synthesised clay rocks with five different illite contents

Group	Mass (g)	Illite content (*wt* %)	Length (mm)	Diameter (mm)	Density (g/cm^3)	Void ratio		
						Initial	Final	Reduction
G1	121.95	85	50.20	37.01	2.26	1.05	0.248	0.802
G2	123.08	70	49.87	37.00	2.29	0.846	0.176	0.670
G3	128.30	55	51.25	37.01	2.33	0.591	0.220	0.371
G4	178.56	40	72.33	37.03	2.29	0.587	0.253	0.334
G5	185.68	25	79.98	37.00	2.16	0.548	0.418	0.130

2010, Cui *et al.* 2010, Komine and Ogata 1996, Maio and Fenellif 1994, Tripathy and Schanz 2007). Detail information about the three components of the raw materials is as follows.

The pure illite was obtained from the French company "Argiles du Bassin Mediterraneen". It was dried into powders through a rotational tempered steel dryer heated by gas without surpassing 100°C. The maximal grain diameter is 45 μm, and the specific gravity density is about 2.7 g/cm^3. The liquid limit of the powdered illite was evaluated by means of the fall cone test on the powdered soils mixed with the saline water of the concentration of sodium chloride to 3.5%. The liquid limit is determined as 46%.

The pure quartz sand used in the tests is the sand GA39 produced by the company SIBELCO. It is consisted of 98.81% of quartz (S_iO_2). The specific gravity density of the particle is about 2.65 g/cm^3.

The brine was obtained by adding sodium chloride into the distilled water, in which the salt concentration in mass was set 3.5%. This value agrees with the concentration of general seawater in the marine offshore petroleum exploitation.

2.2 Material composition design

Five groups of illite clay and quartz sand mixtures with different illite contents (G1:85%, G2:70%, G3:55%, G4:40% and G5:25%) were tested to investigate the effect of illite clay content on the physical properties of the synthesised clayey rocks. The raw materials were mixed homogeneously with the brine water in a stirrer to form a flowable slurry (Howell *et al.* 1997) so that the solid mixtures were fully saturated by brine water. In slurry form, mixed raw materials can fill with the minimized void in the cylindrical oedometer device. In addition, the slurry form approaches the actual original state of the sedimentary deposits in marines.

All the samples were prepared at an initial solution content higher than the liquid limit of the raw materials in order to realize a good plasticity of the slurries. Due to significant difference on absorptivity of bound water between pure sand and powdered illite clay, the liquid limit of the raw materials was thus strongly dependent on the mass content of the illite. Therefore, the liquid limit among the five mixed raw materials differs from each one due to the difference in clay content. The amount of the saline solution needed for the preparations of the slurry mixtures was determined based on the mass content of the solids, largely on the illite clay content. The mass ratio of the saline solution to the total mass of solid mixtures was 0.5 for the sample in group G1, G2 and G3, and was 0.4 for the other two groups G4 and G5 (See Table 1) in order to realize a good plasticity of the slurries and avoid segregation effect between sand and illite.

3. Experimental device and method

3.1 High pressure oedometer device

A self-designed high pressure oedometer was developed at the Laboratory of Mechanics of Lille to characterise the large deformation of the high compressibility of the artificial clayey rocks at high pressures. The device is made up of two independent parts: pneumatic pressure oedometer and hydraulic pressure oedometer as illustrated in Fig. 1. The functionality of the two types of oedometers is similar. Each oedometer is composed of loading system, computer recording system, drainage system, and test chamber. The height of the chamber is about 140 mm with inner diameter of 37 mm. Two *LVDT*s (Linear Variable Differential Transformer) with measurement range of 50 mm and accuracy of 0.01 mm were used to acquire the displacement of sample during application of vertical stress applied by the loading piston at both ends of the sample (see Fig. 1).

The consolidation pressure was applied either by a gas pressure regulator or by a hydraulic pressure pump (Gilson®) adjusting to the compressibility of the artificial clayey rock. At the start of the test, the slurry mixtures can undergo a very large deformation. The gas pressure regulator was used to apply the vertical stress, usually lower than 5 MPa, since it can adapt with the large displacements at initial compaction of the raw materials. However, if the vertical stress supplied by the gas pressure regulating system is high, the gas pressure regulating system has a high risk of leakage and variation with environmental changes. In this way, the hydraulic pressure pump system (up to 60 MPa) was used to realise a constant vertical stress for high-pressure consolidation. The pressure sensors and a data acquisition centre were used to obtain and record the consolidation pressure.

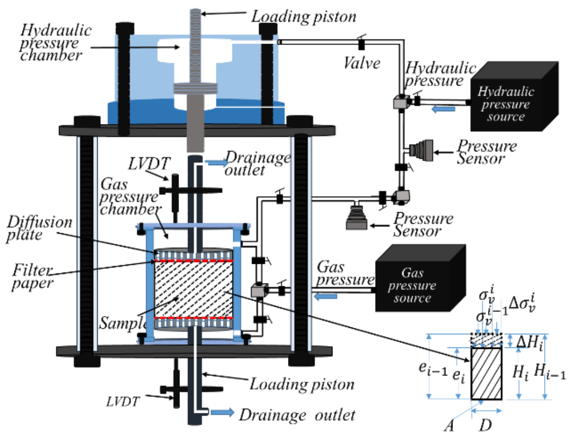


Fig. 1 Schematic illustration of high-pressure oedometer

The double-pressured system of the oedometer allows the fast generation of a constant pressure by the gas regulator to adapt initial large sample deformations and the application of a very high vertical stress by a hydraulic pump for high pressure consolidation. At the alteration of the two-pressure systems, an unloading-reloading cycle was carried out after the stabilization of the primary consolidation.

It should be noted that there is an additional source of friction caused by each external piston at top and bottom of the apparatus related to the hydraulic pressure pump system. The additional friction force generated by the hydraulic pressure chamber was evaluated and about 740 N, which was equivalent to a vertical consolidation stress of 0.4 MPa on the sample. This additional friction effect has been deducted in calculation of the applied vertical consolidation stress.

3.2 Experimental method and procedure

Several loading steps are designed to investigate the effects of the vertical stress and the illite clay content on the physical properties of the synthesised clayey rocks. The experimental protocol is as follows:

a) Mix the pure sand and pure illite with brine for 20 min to form a homogenous slurry according to the prescribed compositions;

b) Apply evenly a thin layer of the Vaseline on the inner surface of the consolidation chamber to reduce frictional effects, and place a filter paper and a diffusive stainless-steel plate at the cell bottom before the slurry mixture is poured into the pneumatic oedometer cell;

c) Place a filter paper and a diffusive stainless-steel plate at the other end of the slurry mixture and then seal the consolidation cell;

d) Expel possible air bubbles trapped into the slurry mixture with a vacuum pump to minimize artefact-originated pores;

e) Apply a pressure of 0.3 MPa until the completion of primary consolidation to realize a compacted and well-shaped cylindrical sample;

f) Increase the gas pressure up to 1.6 MPa with the step of 0.4 MPa and maintain the vertical pressure until the completion of primary consolidation;

g) Realize an unloading phase to allow change from pneumatic loading to hydraulic loading;

h) Reload successively the consolidation pressure by hydraulic pumps following the planned values: 0.5, 1.6, 3.3, 4.7, 8.2, 12.7, 17.7, 22.7, 27.7 and 34.8 MPa. The duration of each stress is assured by the completion of the primary consolidation phase identified by the Casagrande method.

i) Stepwise unload the consolidation stress after the completion of primary consolidation under the maximum vertical stress arrived. The duration of each unloading step was set so that no rebound displacements can be observed by the LVDTs;

j) Remove the synthesised samples from the test cell, and take their weight and size before installing them in a triaxial cell for permeability measurement;

k) Apply confining pressure to the value of 4.0 or 9.0 MPa and maintain it;

l) Apply interstitial pressure at both outlet and inlet of the sample to allow average and differential interstitial pressure respectively of 2.0 MPa and 0.6 MPa. The permeability of artificial clayey rock samples can be obtained by Darcy's flow method.

The height of the raw materials in slurry form was difficult to measure initially. It was calculated according to the final height of the artificially made clayey rock sample and the compression displacements. The gas odometer sealing ability was additionally checked during consolidation of the sample G3, which induced that its loading steps in the low stress regime showed a slight difference with the others. Two parallel consolidation tests were performed for each prescribed raw material composition. There is no significant difference in the measured results during consolidation, thus only one test is discussed below for clarity.

4. Experimental results

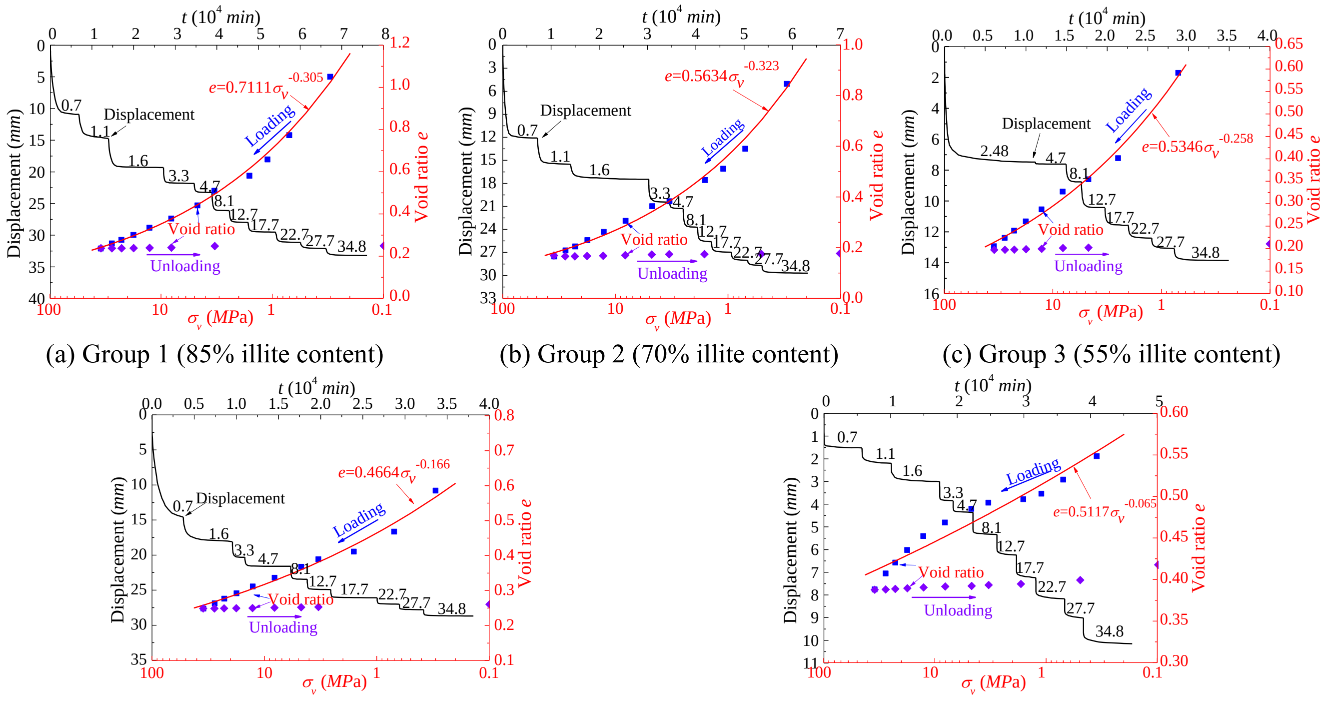
4.1 Compression curves

With incompressibility assumption of mineral particles, the void ratio (or porosity) of the sample at each consolidation pressure can be calculated based on the back-calculated method (Karig and Hou 1992) with the final sample dimensions and its weight, density and content of each solid phase, and compression displacement. During calculation, the unloading induced rebound deformation was also taken into account although it was very small. The geometry and physical properties such as the void ratio of the artificial clayey rock samples can be calculated as given in Table 1. The initial void ratio and the final void ratio were the void ratios respectively for the equilibrium consolidation pressure of 0.3 MPa and the finally compacted synthesised clayey rocks after removing out of the chamber of consolidation cell. The curves of compression displacements versus the logarithmic of time were shown in Fig. 2(a)-2(e), respectively, for the tests with clay content of 85%, 70%, 55%, 40% and 25%. These curves can be used to evaluate the void ratio and compaction modulus. Fig. 3 indicated that the reduction of the void ratio of the samples was induced importantly by consolidation pressure. The compression curves presented a nonlinear character which was similar to those of the clays reported by (Djéran-Maigre *et al.* 1998) , but different from those reported by (Marcial *et al.* 2002; Shang *et al.* 2014) . These differences might be induced by the mineralogical composition of the raw materials and the chemical solution.

The void ratios during unloads were also shown in Fig. 2. The rebound void ratio during unloading indicated that the consolidation process was mainly plastic with few elastic recovery void ratios.

4.2 Permeability

After being undergone compaction with consolidation pressure of 34.8 MPa and then being taken out from the chamber cell of the oedometer device, the permeability of



(a) Group 1 (85% illite content) (b) Group 2 (70% illite content) (c) Group 3 (55% illite content)

(d) Group 4 (40% illite content) (e) Group 5 (25% illite content)

Fig. 2 Consolidation displacement versus time under stepwise loaded stress and void ratio versus consolidation stress

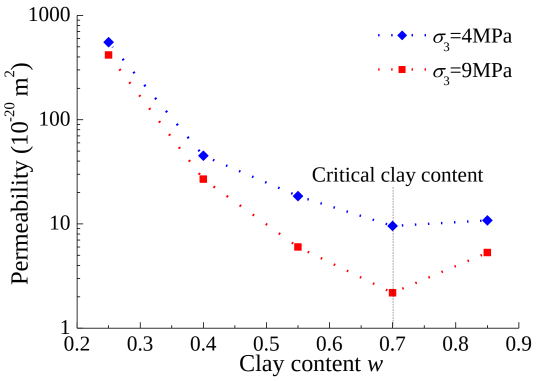


Fig. 3 Evolution of permeability with illite content

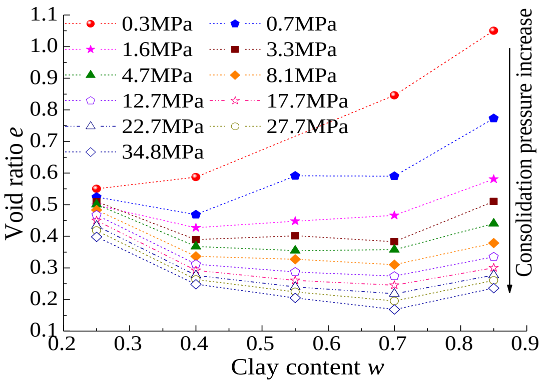


Fig. 4 Variation of void ratio with illite content

the five artificial clayey rocks have been measured by the constant water flow method in a triaxial cell under two isostatic stress of 4 and 9 MPa. The obtained permeability values are shown in Fig. 3. The permeability of artificial clayey rock G5 with w=25% is 5.5*10^{-18} and 4.2*10^{-18} m^2 respectively for isostatic stress of 4 and 9 MPa, and that of artificial clayey rock G4 with w=40% is 4.5*10^{-19} and 2.7*10^{-19} m^2.

The permeability values of the five groups of artificial clayey rocks are closely related to the clay content as shown in Fig. 3. The synthesised clayey rock G2 with w=70% other than G1 has the smallest value of permeability and the artificial clayey rock G5 has the biggest value of permeability. These results agree well with the differences in the void ratio of the five groups of synthesised clayey rocks shown in Fig. 4 and in Table 1.

It is also indicated Fig. 3 that the permeability of each group of synthesised clayey rocks is decreased by the isostatic pressure. The reduction amplitude of the permeability is different for the five groups of clayey rocks when isostatic stress increases from 4 MPa to 9 MPa. The permeability reduction of the clayey rock G2 is more important than that of the others. Therefore, the permeability of the artificial clayey rocks is influenced by the original mineralogical composition. There is a critical illite content w=70% inducing a minimum permeability, when the artificial clayey rocks have been compacted with consolidation pressure of 34.8 MPa or probably more than 8.1 MPa.

5. Discussions

The effects of vertical stress and clay content on the void ratio reduction and modulus change are discussed in this section for the artificial clayey rocks obtained from the high-pressure oedometer tests.

5.1 Effect of consolidation pressure

The void ratios of the five groups of consolidation tests

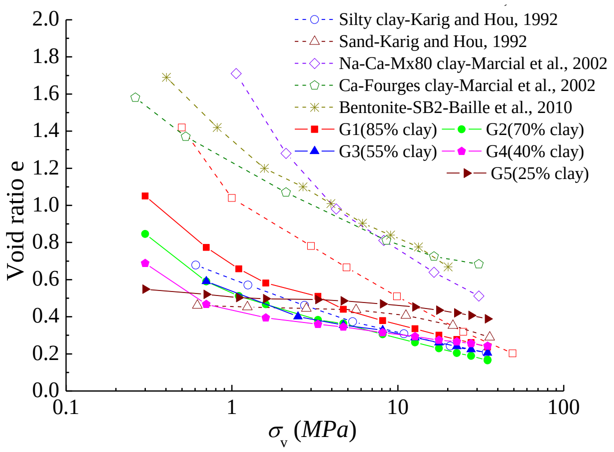


Fig. 5 Comparison of void ratio reduction of clay rich materials under compaction

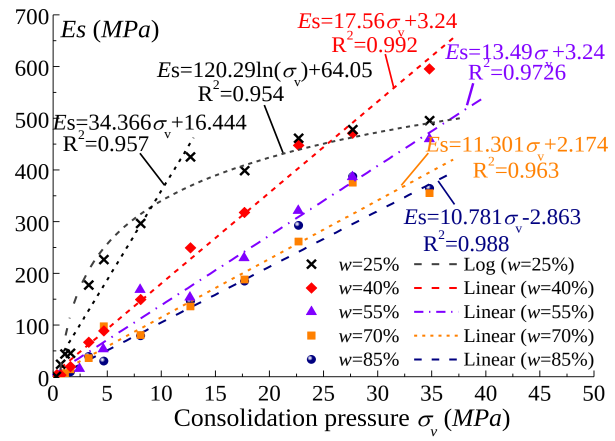


Fig. 6 Evolution of compaction modulus with consolidation pressure

are shown in Fig. 4 for each vertical stress realized. It is shown that the void ratio decreases with increasing consolidation pressure and arrives at its minimum value under vertical stress of 34.8 MPa. The raw materials with higher clay content can absorb more water than those with lower ones during the preparation process due to the difference in water absorption potentials of illite clay and quartz sand particles. All pores of the slurry raw materials can be fully filled by brine water. Thus, the void ratio of the synthesised clayey rock is closely related to its clay content under initial low stress of 0.3 MPa. In addition, the reduction of the void ratio is also influenced by the illite clay content. The higher is the clay content, the more important is the void ratio reduction. However, it is also indicated in Fig. 4 that, as the consolidation pressure increases, the reduction characters of the void ratio are different among the five groups of tests. The void ratios show a minimum value at clay content *w*=70%.

The reduction properties of void ratio of the synthesised samples are generally similar to those obtained during consolidation of clay rich soils, such as kaolinite (Vasseur *et al.* 1995) , silty clay and sand-smectite (Karig and Hou 1992) , Na-Ca-Mx80 clay and Ca-Fourges clay (Marcial *et al.* 2002) , and bentonite-SB2 (Baille *et al.* 2010) in previous publications as presented in Fig. 5. The relationship between the void ratio and vertical stress shows a double linear or nonlinear in the $e - lg\sigma_v$ coordinates. This relationship can be well described by a power law equation as presented prior in Fig.3 for the synthesised samples. The differences between the present study and the former ones lie mainly in the void ratio values. Comparisons between the void ratios of those materials suggested that higher content of swelling clays such as smectite and bentonite can lead to larger void ratio under the same consolidation pressure with the same total clay content. Further, the presence of sand can largely reduce the porosity of the artificial clayey rock samples since the sand grains are filled or embedded in the clay sheets and platelets.

Therefore, vertical stress reduces importantly the void ratio of the synthesised clayey rocks during consolidation. The relationship between the void ratio and vertical stress is highly nonlinear and can be described by a power law.

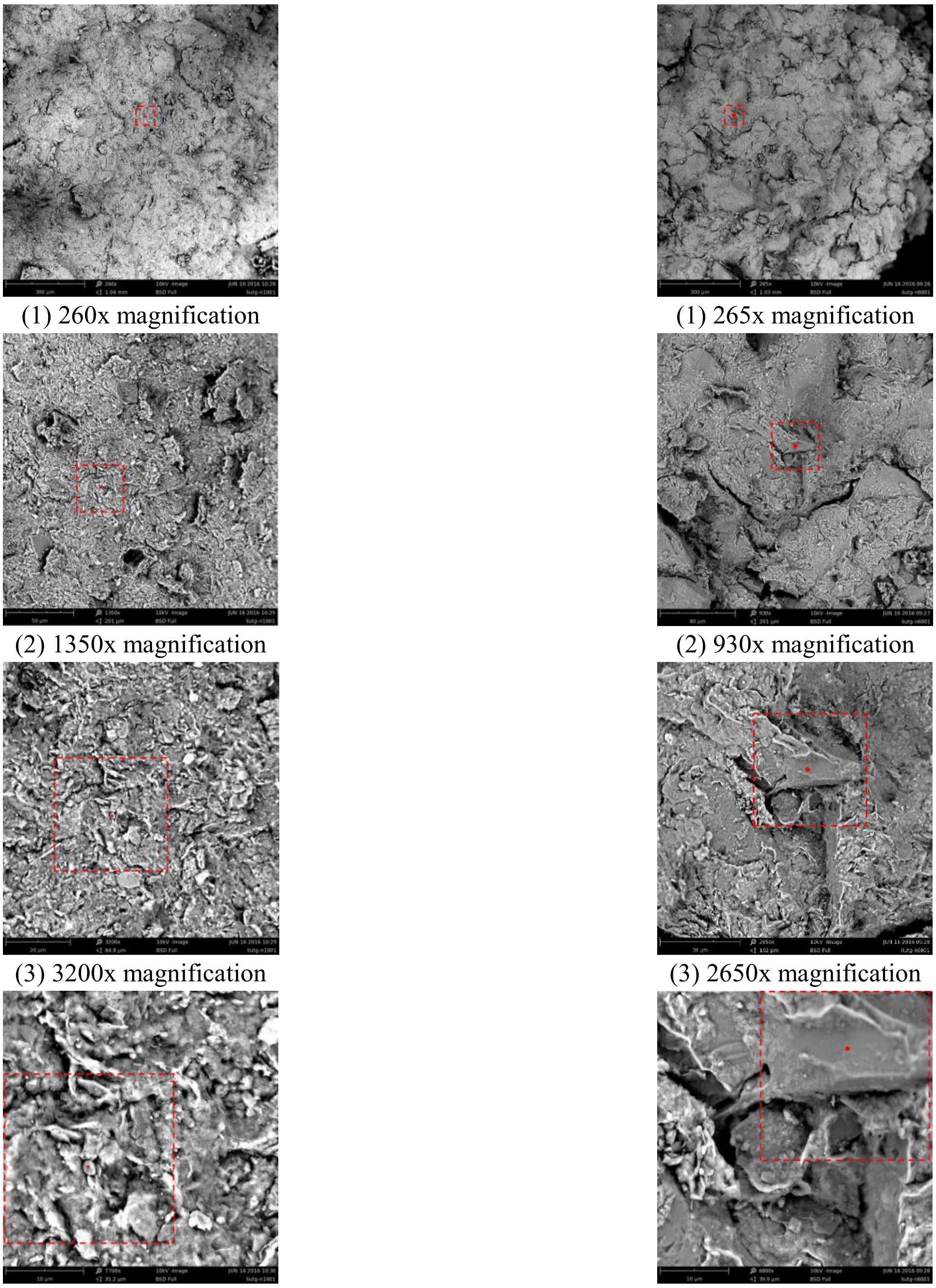
5.2 Effect of clay content

The compaction moduli of the raw materials during consolidation under each consolidation stress are shown in Fig. 6 for the five groups of tests. The compaction modulus is defined as the ratio of the variation of vertical stress to the corresponding variation of vertical strain in a material in condition of confined compression, also commonly known as constrained modulus. The quantification of the relationship between the compaction modulus and consolidation pressure is also given in Fig. 6. It is shown that the compaction modulus increases with increasing consolidation pressure for all the five groups of tests of different illite contents.

These quantification equations are different for the five artificial clayey rocks due to the influence of illite clay content as indicated in Fig. 6. The log functions describe best the quantification of group G5 with clay content *w*=25%. However, linear equations with different coefficients describe best the other four groups of tests with higher clay contents during consolidation.

It is also observed in Fig. 6 that lower values of clay content induce greater values of compaction modulus for the raw materials under the same consolidation stress with values smaller than 25 MPa. Due to the nonlinearity of the quantification equation for G5, the influence of the clay content on compaction modulus is complicated under high consolidation pressures greater than 25 MPa. Therefore, the clay content has an important effect on the change of compaction modulus of the mixed raw materials under consolidation.

The above phenomena are probably induced by the deformation mechanisms of the mixed materials during consolidation. The deformation mechanisms are strongly and sensitively associated with the skeleton structure (Li *et al.* 2019). The artificial clayey rocks are synthesised from the quartz sand and illite clay mixtures saturated by brine solution. When the quartz sand and the illite clay mixtures are mixed with brine solution during sample preparation, water fill in the pores between the solid particles. This type of water is named as the gravity water in the mixed raw materials. Moreover, the clay minerals can absorb and thus



(1) 260x magnification (1) 265x magnification

(2) 1350x magnification (2) 930x magnification

(3) 3200x magnification (3) 2650x magnification

(4) 7700x magnification (4) 6800x magnification

(a) G1 (w=85%) (b) G4 (w=40%)

Fig. 7 Microstructures of two representative samples

store an extra-large amount of brine water in the layers of illite clay platelets in micro scale. This type of water, named as the bound water (adsorptive and film water), is absorbed in the clay platelets and thus causes swelling of the clay minerals. This type of water is difficult to expel during consolidation compared with the gravity water.

For the ones with low clay content (G5 for example), the majority of water is the gravity water filling in the pores among the quartz particles. During consolidation, the gravity water in the mixed raw materials is firstly expelled with the application of low consolidation pressure. In this phase, large rate of void ratio reduction occurs and compaction modulus increases rapidly. During this process, the compaction modulus increases quasi-linearly with expel of gravity water. The deformation is induced mainly by the compaction of the pores filled by gravity water. Expel of the bound water on the other hand only starts at higher consolidation pressures. When the consolidation pressure increases up to an even higher value, the compaction of the compacted raw material becomes more difficult with the same stress increment. As a result, the void ratio decreases much slowly. Finally, the compaction modulus of the synthesised sample will approach a saturated value that corresponds to the modulus of the dense mineral grains of the raw materials. Therefore, compaction modulus of the synthesised clayey rock G5 with w=25% shows a nonlinear phase during the consolidation process in Fig. 6. Similar to the other four groups, there is also an evident linear phase prior to the nonlinear phase of G5 when the stress is lower than 13 MPa.

The nonlinear relationship between the compaction modulus and the synthesising stress can be described by a log function for the test G5, unlike a linear relationship for the other groups of tests. The absorbability of the raw materials with higher clay content is much greater than those with lower clay contents. Compaction of raw materials during consolidation with higher clay content is thus much easier than that with lower ones. This is also indicated by the difference of the void ratio reduction of the five groups of tests shown in Fig. 4. The saturated values of compaction moduli are much greater for the artificial clayey rocks with higher clay contents. Therefore, the compaction moduli of the four groups of tests with high clay contents only show a linear relationship with the applied vertical stress. The applied stress is insufficient high to induce a nonlinear phase for the tests with high clay content. In consequence, the relationship between the compaction modulus and pressure is affected by the clay content.

Therefore, the clay content has direct influence on the reduction of void ratio of the mixed raw materials during consolidation. There is a critical illite clay content that induces minimum void ratio after high stress (more than 8.1 MPa) consolidation. In this study, the synthesised clayey rock sample with illite clay content of w=70% has the minimum void ratio. Lower values of illite clay content induce greater values of compaction modulus for the raw materials under the same consolidation stress with values smaller than 25 MPa. The compaction modulus variation is coupled influenced by the illite clay content, the vertical stress and the mineral grain properties.

5.3 Microstructure implications

As mentioned above, the influences of illite clay content on void ratio reduction, compaction modulus and permeability are not monotonic. Thus, there must be other factors that stand during the consolidation. Due to the availability of data and not lose generality, we presented here SEM analysis of two representative samples, one with high illite clay content and the other with low illite content.

Fig. 7 shows two groups of microphotographs of the representative artificial clayey rocks G1 and G4 by the SEM technique. In each microphotograph, both a red-filled circle and a rectangle with red dash line are used to get an exactly position in the scanning cross section of the tested block, where the red-filled circles marked in the four microphotographs of the artificial clayey rock G1 (Fig. 7(a)-1 to Fig. 7(a)-4) or G4 (Fig. 7(b)-1 to Fig. 7(b)-4) are located in the identical position of the same section, and are also to clearly and conveniently distinguish pore size at different scales with different magnifications.

It is shown that the pore properties between the two artificial clayey rocks are different. The pores of the artificial clayey rock G1 are mainly formed by the inter-granular space between two illite platelets, and the pore size is obviously less than size of the illite platelet and quartz particles as shown Fig. 7(a). However, a number of interface pores can be noticed in Fig. 7(b) between the quartz-illite particles and quartz-quartz particles. The pore sizes are larger than the distance between two illite platelets.

The synthesised clayey rock G1 has a high illite content of 85%, indicating that there is sufficient illite minerals to fulfil the surface of the quartz particles and their inter-granular void spaces, and to form a cementing force with the presence of water during high stress consolidation. As a result, the quartz particle can be well surrounded by or embedded in the illite matrix. Thus, there is no obvious defects in Fig. 7(a) at the interface of the quartz-illite minerals after the high-pressure consolidation.

On the contrary, the illite content of the clayey rock G4 is relatively low. There are inadequate illite platelets to include all the quartz particles and thus there are some direct contacts between quartz-quartz particles as shown in Fig. 7 (b). These features will induce a relatively large pore size of this group of materials compared with the one shown in Fig. 7 (a). Moreover, the quartz particles are generally incompressible compared to the illite platelets. The compaction is thus more difficult in the raw materials with more quartz-to-quartz particle contacts than in those with more quartz-to-illite platelet contacts. Consequently, it can be inferred that the skeleton structure of the synthesised clayey rock G1 is totally constructed by the illite clay platelets rather than by the sand particles for the synthesised clayey rock G4.

Therefore, the void ratio of the synthesised clayey rock G1 is smaller than that of the G4 after the consolidation test. The reduction of void ratio during compaction of the raw materials of G4 is smaller than that of G1. Therefore, besides the illite content, the compaction properties and permeability of the artificial clayey rocks are also influenced by the rearrangements of the particles during mixing and high-pressure consolidation.

6. Conclusions

The physical properties of the synthesised clayey rocks have been characterised by the specific autonomous testing device. The effects of illite clay content and consolidation stress have been demonstrated and interpreted by the test data and the SEM analysis. Conclusions can be made as follows:

(1) Consolidation stress can reduce importantly the void ratio of the clayey rocks during consolidation. The relationship between the void ratio and vertical stress is highly nonlinear and can be described by a power law for the synthesised clayey rocks in this study.

(2) The illite content has a direct influence on the reduction of void ratio of the synthesised clayey rocks. There is a critical illite clay content of w=70% inducing minimum void ratio after high stress (higher than 8.1 MPa) consolidation in this study.

(3) Consolidation pressure can enhance importantly the compaction modulus of synthesised clayey rocks. The relationship between the compaction modulus and

consolidation pressure is affected by the clay content. And the relationship can be simulated well by a linear function for all the artificial clayey rocks, except for the G5 (w=25%) where a log function describes more appropriately.

(4) Permeability of the synthesised clayey rocks undergone compaction with consolidation pressure of 34.8 MPa is importantly related to illite content. There is a critical clay content (w=70%) that induces minimum permeability. The permeability is also influenced by the isostatic stress.

(5) In the synthesised clayey rocks with high illite clay content, the sand particles are totally surrounded by the illite platelets, and dispersed randomly and homogeneously inside the matrix formed by the illite minerals. The pores size existing in the clayey matrix is smaller than that of the illite platelets. In the case of low illite content, there are some interface voids between the quartz-illite minerals and quartz-quartz particles since the bulk volume of the clay will only occupy a portion of the sand void space. The skeleton structure of the artificial clayey rocks varies depending on clay content and transforms from the skeleton constructed by sand particles to the one formed by illite clay platelets with increase of clay content. Future work will be carried out on the consolidation of different raw materials with combined physical and chemical reactions.

Acknowledgments

This work was supported in part by Total Company, in part by the Science and Technology Research Project of the Department of Education of Jiangxi Province (No. GJJ190979), in part by the Fundamental Research Funds for Central Universities of China (N180105031), the Young Talent Program of Liaoning Province (XLYC1807094), the Research and Development Program of Anhui Province (no.1804b06020361) and Sichuan Province (no.2019YFG0047), in part by the 111 Project (B17009), and in part by the Sino-Franco Joint Research Laboratory on Multiphysics and Multiscale Rock Mechanics. The authors thank Mr. Jean Secq for technical supports in preparation and development of the testing devices.

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