Mechanics of compression behaviour in shale

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ABSTRACT

A deep understanding of mechanics of behavior of shale is very important because of its properties when associated with other geomaterials and its suitability as a host materials for engineering applications. Investigating compression behavior is essential because geotechnical and geological problems can be analyzed using data obtained from one-dimensional compression tests. To achieve this, odometer tests were conducted on the reconstituted and intact samples of shale. In addition, chemical, microstructural and mineralogical analyses were studied. Shale is dominated by quartz, hornblende and calcites. The fabric is characterized by continuous clusters with few inter clusters voids. The compressibility is low and the yield stress is medium to high. The degree of enhance resistance in compression is positive.

1. INTRODUCTION

Investigating compression behavior of geomaterials is very essential because it is the means by which one of important parameter for computations in geotechnical engineering (compression index) can be obtained. The parameters obtained from compression tests on geomaterials are commonly used for the analyses of many geotechnical and geological problems. For example, settlement calculations which can result from several factors such mining subsidence, debris flow, slope failure and earthquake. So the knowledge about compression behavior has been important for the practitioners in geological and geotechnical engineering profession. The understanding has been widespread for sedimentary clays (e.g., Burland 1990; Gasparre and Coop 2008; Liu et al. 2103; Mataic et al. 2015) and other related geomaterials (e.g., Okewale and Coop 2017, 2018a, 2018b, 2020; Okewale 2019a, 2020a, 2020c; Rocchi et al. 2015, 2017). However, such studies are very limited for shale. Although some work have been done on mechanical properties of shale (strength, stiffness, brittleness) using uniaxial and triaxial compression tests as well as Brazilian tests (e.g., Cho et al. 2012; Arora and Mishra 2015; Wang et al. 2016; Hou et al. 2019) but majority of them are on shear loading and study on compression loading is still scanty despite its significance. Shale belongs to sedimentary geomaterials and its properties (e.g., very low permeability, sealing potential) make it suitable for various engineering applications. Therefore, studying compression behavior of shale is essential because geomechanical characteristics of
shale is of great interest in energy industries. This work presents the behavior of shale in compression and this was achieved by conducting odometer tests on the intact and reconstituted samples. Also, grading was studied using classification test, microstructure and chemical analyses were investigated using scanning electron microscope (SEM) equipped with electron dispersive spectrometer (EDS) and mineralogical analysis was conducted using X-ray diffractometer (XRD). This type study is rare and new for this geomaterial and also novel is the way in which the effects of geological structure are studied.

2. MATERIALS AND METHODOLOGY

The materials used are shale samples. They were collected as block samples from Dangote Coal Mine, Ankpa Kogi State, Nigeria. Samples were retrieved from mine face and typical profile of mine face showing sampling point is presented in Fig. 1. The sample is overlain by lateritic soil and underlain by coal which is being excavated and use as a source of energy for different plants.

![Profile of shale](image)

Fig. 1 Profile of shale

The particle size distributions were determined using a combination of wet sieving and sedimentation technique. The microstructure and chemical analyses were studied using a Phenom ProX scanning electron microscope (SEM) equipped with electron dispersive spectrometer (EDS). The samples were prepared by breaking the surface rather than cutting in order to reveal the true fabric of the sample. The samples were placed in sample holder and pushed into machine for the analyses. A Shimadzu XDS 2400H diffractometer equipped with JCPDWIN software was used for the mineralogy analysis. It operated at 40 kV and 55 mA. The minerals were identified in the range of $5^\circ \leq \theta \leq 70^\circ$ with Cu-$k\alpha$ radiation and samples were scanned at $0.02^\circ / 0.30$ s. The analyses were conducted on samples in powder form.
Conventional front loading odometer was used to study the compression of the samples. For the reconstituted samples, a closed base fixed confining ring of 50 mm diameter and 20 mm height was used. A floating ring of 30 mm diameter and 20 mm height was used for the intact samples and this allows higher stresses to be achieved in the tests. The load was applied incrementally from 0.1 kg to 50 kg. Reconstituted samples were prepared using natural moisture content and in these samples, natural bonding and fabric have been removed. Samples were mixed in a container, vibrated and then placed in confining ring. The initial height was then carefully measured under a small nominal load. The intact samples were prepared by careful trimming of the block samples in order to reduce the disturbance to the samples. The sample was excavated ahead of the ring and the ring pressed with a small downward pressure.

Specific volume \( v = (1 + e) \), where \( e \) is void ratio, void ratio \( e \) and porosity \( n \) are very important in describing the behavior of geomaterials together with stress. However, specific volume is used here because of the framework adopted. Different methods were used to determine specific volumes to improve the confidence and the equations are similar to those used by Okewale and Coop (2017, 2018a, 2018b, 2020) and Okewale (2019b, 2020a). Initial weight, moisture content, height and diameter were used to determine initial specific volume and final specific volumes were estimated from final measurements, back calculating the initial value using volumetric strain measured in the tests.

3. RESULTS AND DISCUSSIONS

3.1 Grading

Figure 2 presents the particle size distributions of the sample determined from wet sieving and sedimentation. The demarcations in the figure were used to divide the sample into various fractions (clay, silt and sand). The sample is well graded. The mean particle size \( d_{50} \) is 0.04 mm and the coefficient of uniformity \( C_u \) is 72.85 which shows well graded nature of the sample. The clay content (fraction with particles diameter less than 0.002 mm) is 15%, silt content (fraction with particles diameter between 0.002 mm and 0.063 mm) is 49% and sand content (fraction with particles diameter between 0.063 mm and 2 mm) is 26%. The sample can be classified as sandy silt.
3.2 Microstructure and chemical analyses

The fabric and composition of the sample were determined using SEM equipped with EDS. Figure 3 shows the SEM images of sample in horizontal and vertical planes. The micrographs are shown for 100 μm field of view for proper comparison. In horizontal plane, the fabric is dominated by flat particles and in some places the particles aggregated to form clusters (Fig. 3a). There are many inter particle and intra cluster voids. There is no specific particle orientation and fabric is heterogeneous. In vertical plane, the fabric is characterized by particle aggregation forming continuous clusters with few intra cluster voids (Fig. 3b). The fabric seems more robust in vertical plane and the fabric is heterogeneous. Comparing the fabrics, it seems the degree of anisotropy in the samples is small.
Table 1 presents the elemental composition of the samples. The values shown in the table are the average for two samples. The sample is dominated silicon followed by aluminum with other elements in trace amount. The details of mineralogy are given in Table 2. The mineralogy is dominated by quartz, hornblende and calcite in that order. Other minerals present are plagioclase, kaolinites, chlorite and hematite.

Table 1. Details of chemical composition

<table>
<thead>
<tr>
<th>Element/symbol</th>
<th>Atomic (%)</th>
<th>Weight (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon/Si</td>
<td>63.32</td>
<td>59.93</td>
</tr>
<tr>
<td>Aluminium/Al</td>
<td>19.04</td>
<td>17.24</td>
</tr>
<tr>
<td>Sulphur/S</td>
<td>5.79</td>
<td>6.23</td>
</tr>
<tr>
<td>Iron/Fe</td>
<td>2.72</td>
<td>5.10</td>
</tr>
<tr>
<td>Potassium/K</td>
<td>2.93</td>
<td>3.86</td>
</tr>
<tr>
<td>Calcium/Ca</td>
<td>1.51</td>
<td>3.86</td>
</tr>
<tr>
<td>Titanium/Ti</td>
<td>1.06</td>
<td>1.70</td>
</tr>
<tr>
<td>Phosphorous/P</td>
<td>0.73</td>
<td>0.76</td>
</tr>
<tr>
<td>Magnesium/Mg</td>
<td>1.34</td>
<td>1.09</td>
</tr>
<tr>
<td>Sodium/Na</td>
<td>1.16</td>
<td>0.90</td>
</tr>
<tr>
<td>Zircon/Zr</td>
<td>0.37</td>
<td>1.12</td>
</tr>
</tbody>
</table>

Table 2. Details of mineralogy

<table>
<thead>
<tr>
<th>Minerals</th>
<th>Composition (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>41.1</td>
</tr>
<tr>
<td>Plagioclase</td>
<td>2.51</td>
</tr>
<tr>
<td>Kaolinites</td>
<td>2.34</td>
</tr>
<tr>
<td>Calcite</td>
<td>16.74</td>
</tr>
<tr>
<td>Chlorite</td>
<td>5.89</td>
</tr>
<tr>
<td>Hornblende</td>
<td>20.61</td>
</tr>
</tbody>
</table>
3.3. Intrinsic behavior

Figure 4 presents odometer tests for the reconstituted samples. The compression behavior of reconstituted sample is termed intrinsic behavior. The constituent particles are solely responsible for this behavior. This is very important because the compressibility parameter needed for design is obtained from it. The reconstituted compression paths and one-dimensional normal compression line (1D-NCL) are shown in Fig. 4. The 1D-NCL refers to the intrinsic behavior derived from reconstituted samples. The 1D-NCL was estimated using compressibility parameters (intercept N and compression index Cc). The NCL is assumed to be straight line within the range of stresses studied. The compression paths converge to unique line, similar to sedimentary clays (e.g., Burland 1990) and other geomaterials (e.g., Okewale and Coop 2017; Rocchi et al. 2017). However, more tests are needed particularly at different initial densities for the shale samples. The N and Cc values for the samples are 2.01 and 0.12 respectively. Comparing to other geomaterials (e.g., clays, weak geomaterials), the compressibility of shale is very low.

![Fig. 4 Compression behaviour of reconstituted samples](image)

3.4 Intact behavior and effects of structure

Figure 5 presents the intact compression paths and 1D-NCL. The paths plot below NCL due to low in-situ specific volume. The yield stresses range between 400 kPa and 800 kPa.
The conventional effect of structure is increase in strength and stiffness as a result of bonding and fabric during compression. It is assumed in this study that structures depend on geological history of the sample. The effects of structure are evaluated using normalizations (void index $I_v$ and normalized volume $v_n$) suggested by Burland (1990) and Coop and Cotecchia (1995).

$$I_v = \frac{e - e_{100}^*}{e_{100}^* - e_{1000}^*}$$ (1)

where $e$ is current void ratio, $e_{100}^*, e_{1000}^*$ are the void ratios on the 1D-NCL corresponding to vertical effective stress at 100 kPa and 1000 kPa respectively.

$$v_n = \exp\left(\frac{\ln(v) - N^*}{\lambda^*}\right)$$ (2)

where $N$ and $\lambda$ are intercept and slope of NCL.

$$\ln(v) = N^* - \lambda ln p'$$ (3)

where $p'$ is mean normal effective stress $p'$ and it was estimated for one-dimensional compression using

$$p' = \frac{\sigma_v' + (2\sigma_k'k_o)}{3}$$ (4)

where $k_o = 1 - \sin\phi'$ as proposed by Jaky (1994).

Both $I_v$ and $v_n$ normalize the compression behavior for grading and mineralogy thereby making the ICLs to be unique and allows the effects of structure to be compared. The effects of structure are indicated by the degree to which compression curves of the intact samples cross the ICL and it can be seen that the curves do not reach a state outside ICL due to low initial $I_v$ and $v_n$ (Figs. 6 and 7). The tendency of an apparently no effect of structure has been highlighted as an artefact of normalization rather than true indication of effect of structure by Gasparre and Coop (2008). This type of behavior has been seen in different geomaterials (e.g., Okewale and Coop 2018a).
Another quantitative way to determine the effects of structure is by computing stress sensitivity as proposed by Cotecchia and Chandler (2000). However, the methods cannot be used here because the ICL is defined outside the intact compression curves. In order to obtain quantitative effects of structure, the methods proposed by Okewale and Grobler (2020) is used.

The definitions are given in the schematic plot in Fig. 8. The two methods assumed that stress sensitivity is ratio of stress at yield for the intact sample to stress on ICL of the
reconstituted sample at the same specific volume. The methods are based on the assumption that, at high stress, samples have been destructured and will behave like reconstituted samples.

![Figure 8 Definition of stress sensitivity (After Okewale & Grobler, 2020)](image)

Positive effects of structure can be seen using the two approaches (Figs. 9 and 10). The magnitude is similar to what has been reported for other related geomaterials (e.g., Okewale and Coop, 2017). The post yield behavior of the samples have been presented in Figs. 9 and 10. This is achieved by presenting the current values of stress sensitivity with vertical stress (Figs. 9 and 10). The current stress sensitivity is calculated using the current point on the post yield compression paths. This is simply quantifying structure degradation of the intact sample in compression. The lines of demarcation, where stress sensitivity is one, indicate the points of complete structural breakdown in samples. The samples have been completely destructured at the highest stresses reached in the tests.
4. CONCLUSIONS

The mechanics of compression behavior of shale have been investigated by conducting odometer tests on the reconstituted and intact states. Apart from this, physical, microstructure, chemical and mineralogy were also studied. The sample is well graded and can be classified as sandy silt. The fabric is characterized with flat particles and continuous clusters with inter particle and intra cluster voids. The fabric is heterogeneous and anisotropy is low. The composition is dominated by silica and alumina and mineralogy is dominated by quartz, hornblende and calcite. The samples have
convergent behavior and the compressibility is low. The yield stress is medium to high. Using normalizations, apparent no effect of structure was found due to low initial void index and volume but the effect of structure is positive using stress sensitivity.

REFERENCES