A micromechanical experimental study of kaolinite-coated sand grains

S.S. Kasyap, K. Senetakis*

Department of Architecture and Civil Engineering, City University of Hong Kong, Kowloon, Hong Kong SAR

ABSTRACT

The micromechanical behaviour of sand grains coated with a clay agent is examined in the study. The coating was applied using a suspension of sand grains and kaolinite powder with a wide spectrum of coating percentages and durations of vibration to achieve different classes of coating. Element composition, measurement of surface roughness and single grain crushing tests were used for the basic characterisation of the materials. From pure sand grains to lightly coated grains, there was observed a significant shift of the inter-particle friction but the differences were smaller between lighter and heavier coating. Microscopic images showed the creation of debris during shearing which might have contributed to the increase of friction from the uncoated to the coated grains.

1. Introduction

Advancements in the application of the discrete element method (DEM) in geomechanics [1] have necessitated more systematic micromechanical studies to be conducted in the laboratory. Such experimental studies can provide important information associated with the behaviour of geological materials at the grain scale, including grain strength and grain contact response, so that the findings from laboratory research can be further utilized in DEM analysis as input. Some of the important quantities to be explored in the laboratory include the tangential and normal load – displacement relationships as well as the inter-particle coefficient of friction [2]. Particularly for the inter-particle coefficient of friction, previous studies have highlighted its important influence on the macro-scale monotonic and cyclic behaviour of granular materials [3–6].

In terms of micromechanical studies investigating the normal or tangential load – displacement behaviour and inter-particle friction of real soil grains or reference grains, previous works [7–13] have focused, majorly, on clean and/or uncoated surfaces. This has been deliberately performed by cleaning the surfaces of the grains (for example in the studies by Refs. [7–9]) to remove any impurities that could influence the output from the micromechanical tests. Cleaning of the grain surfaces can help to isolate any unforeseen factors on the contact mechanics behaviour of soil grains and purely examine the frictional mechanisms at the grain contacts providing fundamental insights into the micromechanical behaviour of geological materials. In other micromechanical studies [11,14–16], it was not explicit whether the surfaces of the tested materials had been cleaned. Nevertheless, it can be considered that most of these works focused on uncoated grain surfaces, so that effects such as, for example, surface roughness, material type and composition or surface morphology could be examined in a straightforward way.

In nature, granular soils comprise a mixture of coarser and finer grains and it is common that the surfaces of the grains may be coated. This coating can be because of chemical or biological substances, weathering processes or fine grained material covering sand grains. The latter may be the case for sand-silt or sand-clay mixtures (which are called as gap-graded soils). The coating of the surfaces of geological materials is applicable not only within the context of soil mechanics applications, but it is also of major interest in the rock mechanics discipline. For example, rock joints are commonly filled with a coating material which may affect markedly the mechanical behaviour of gouges [17]. Using laboratory element testing or numerical analyses, researchers have studied the influence of the presence of finer geological materials, i.e. silt and clay fractions, on the frictional behaviour and shear strength of coarse grained soils [18–22]. This kind of studies also influence the research on the liquefaction behaviour of granular materials due to the presence of fines [23–25]. Rheological studies have also been carried out varying the concentration of the granular suspension, which have enhanced our understanding on problems related, for example, to debris flows [26]. Problems such as liquefaction or debris flows (i.e. granular flows) are of major interest in the disciplines of geotechnical and geological engineering. Even though many advancements have taken place recently in the coating techniques and studies on coating effects on the tribological behaviour of interfaces [27–32], there have been relatively limited studies related with the coating characteristics of geological materials. For example, Barrows et al. [33] studied the natural formation of coating on the surfaces of
limestone grains in soil due to wetting and drying cycles. The coating formed on the grains was observed to be characteristic with the surrounding soil and that the combination of effects such as weathering, recrystallization and adsorption were found to be the major reasons for the formation of coating. Scheidegger et al. [34] worked on coating silica sand with goethite (i.e. an iron oxide compound). Torrey et al. [35] described different procedures for coating of quartz crystals with nanoparticles. These works were biased either towards chemical interactions with soils or coatings at the nanoscale.

In this research work, an attempt was made to study in the laboratory the micromechanical behaviour of sand-sized grains coated with clay minerals in order to quantify the frictional and normal contact response of coated sand grain interfaces. For this purpose, the commercially available kaolinite was used as the coating compound, and Leighton Buzzard sand (LBS) was used as the coarse-grained material, which quartz sand has been thoroughly examined in previous works [8,9,13,36]. The three mainstream objectives of this work were: (i) to develop a new technique in coating natural sand-sized grains with clay powder, (ii) to provide characterisation of the coated grains in terms of element composition, surface roughness and grain strength and (iii) to examine the effect of the coating presence and amount on the tribological behaviour of natural grains.

2. Material description

Leighton Buzzard sand (LBS) of fraction 2.36–5.00 mm was used for both coating studies and micro-mechanical testing. This is a typical quartz sand which consists of Silicon (Si) and Oxygen (O) as the primary elements. Traces of other elements such as Aluminium (Al) and Iron (Fe) are also found in the particles. Typical energy-dispersive X-ray spectroscopy (EDS) test results of pure LBS are shown in Fig. 1. Average percentage weights of elements for fifty pure LBS particles were used in the current study, which are also shown in the same figure. These results from the different particles showed consistent values with small deviations. Wang and Coop [37] described the LBS grains as rounded and translucent white or yellow in colour with a greasy lustre. On the surface of the LBS grains, coating of clay minerals was applied using the commercially available kaolinite powder. The chemical composition of the kaolinite is Al₂Si₂O₅(OH)₄ as a repeating unit, whereas, quartz has SiO₂ as a repeating unit and hence the elemental analysis of ‘kaolinite coated LBS particles’ should show an increase in Al percentage. This is a key parameter used in the current study to quantify the amount of coating on the surfaces of the grains. The EDS results and scanning electron microscope (SEM) images of pure kaolinite are also shown in Fig. 1. It can be observed that the kaolinite particles have flaky shape and varying sizes. This could be used as a reference to observe the adsorption of kaolinite on the LBS grains. The liquid limit and plastic limit of the kaolinite are 30% and 15%, respectively, resulting in a low to medium plasticity material with Plasticity Index (PI) of 15%. Kaolinite was made as a solution for coating and distilled water was used as the solvent. The adopted procedure for coating is explained in the next section. From Sandeen and Senetakis [38], the average surface roughness (Sₖ) of pure LBS grains was observed to be around 0.223 μm with a standard deviation of ± 0.061 μm as measured from the optical surface profiler of the City University of Hong Kong. This value was confirmed from representative tests carried out by the authors using the same optical profiler.

As an established practice, the LBS grains were cleansed with Butanone before testing [8,9] in order to remove any oil or other impurities from their surfaces that could disorder the actual behaviour. For this purpose, ultrasonic cleaner with distilled water at a temperature of 50–60 °C was used for 30 min to remove any kind of impurities from the material surface before the application of the coating. Hence the sole effects of the coating material could be studied without the intervention of pre-occupied debris on the surfaces of the grains.

3. Coating procedure

Six different concentrations and three different time intervals were chosen to examine the influence of the coating material on the amount of adsorption on the surfaces of the LBS grains. The LBS grains were made in contact with 25, 50, 100, 150, 200 and 300 mg/ml concentrations of kaolinite solution for time intervals of 100, 500 and 1000 s.

At a required concentration, a uniform solution of kaolinite was prepared with distilled water as the solvent by shaking the mixture. The temperature of the distilled water used in this process was around 40–50 °C to achieve uniformity more readily. Shaking of the solution was carried out using an orbital shaker which moves its platform in a horizontal orbital motion for set time and at set RPM. The beakers with mixture are fixed to this platform (Supplementary Fig. S1). To avoid the formation of lumps, kaolinite powder was gradually added in small amounts to the distilled water which was already in motion. During this process, it was observed that the clay particles were not always completely dissolved in the water, particularly for lower concentrations. Because of this, the mixture should be kept in constant motion within the total duration of the coating procedure to maintain a uniform solution. Thereafter, the LBS grains were dropped into that uniform solution. After a few trials, an RPM of 220–250 was found to be appropriate for the solution to be uniform and for minimum impact of the grains. After the time of interaction was set, the grains were separated from the solution and they were oven dried. The effectiveness of this coating procedure was evaluated qualitatively with SEM and digital microscope images and quantitatively with energy dispersive spectroscopy (EDS) graphs.

4. Coating analysis

EDS analysis provides a spectrum of elements as well as their percentage weights available in the functional scan area of the grain. With respect to the size and shape of the grains, the maximum possible surface was scanned for the analysis by adjusting the magnification. For each combination of concentration and time, five grains were coated and analysed. As discussed in the previous section, the coated LBS grains should show an increase in aluminium (Al) content because of the chemical composition of the materials. This statement could be supported, primarily, with EDS graphs of coated LBS grains. Typical spectra obtained at different concentrations are shown in Fig. 2. It is observed that the peak value of Al increased with increasing concentrations. Hence, the governing parameter could be either the aluminium percentage on the coated surface or the change in the aluminium percentage after coating. Finally, the change in aluminium percentage was arbitrarily chosen for the current study as the trend for both parameters was similar. Fig. 3 shows the variation of the change in percentage weight of aluminium with the increase in concentration at three different exposure times. Their associated standard deviations and polynomial trendlines for bar graphs are also shown in the same figure. The trends were fitted very well to the data with admirable R² values. There was observed a strict increase in the adsorbed aluminium content with increase in concentration for given time of exposure. Also, there was an increase in the adsorption, though small, with the increase in time of exposure. As shown in Fig. 3, from 500° to 1000°, the responses were within the standard deviations of each other and the adsorption was observed to reach a saturation level in the range of 200–300 mg/ml of concentration. This was not the case for 100° time of exposure where no hint of saturation was observed even up to 300 mg/ml. Based on the results from the coating analysis, it was understood that heavier concentrations were required to reach saturation at 100° of exposure time. At lower concentrations, all the curves started with a saturation behaviour irrespective of the time of exposure. This explains that any concentration below 25 mg/ml would show similar values of the adsorbed aluminium contents. The said phenomenon could not be
supported experimentally since the adsorption phenomenon at concentrations lower than 25 mg/ml was very unrepeatable and unreliable for the given material combination (i.e. LBS with kaolinite). Hence, the range of concentrations chosen for the current study had a lower bound of 25 mg/ml.

Supplementary Table 1 gives a summary of the numerical values of the bar graphs and their standard deviations as illustrated in Fig. 3. The standard deviation values are in considerable range with a maximum of ± 27% and a minimum of ± 2.5%. Precisely, very small deviations are observed for the 300 mg/ml condition. The morphology of the grains could be, perhaps, one of the primary aspects that was accountable for non-uniform adsorption of the coating material on the surface of the LBS grains. After the grains were shaken in the kaolinite solution, the solution was observed to be stagnant in the micro troughs on the surface of the grains. After oven drying, deposition of excessive kaolinite in troughs than in crests occurred. This phenomenon was observed more extensively for medium concentrations. At lower and higher concentrations, the adsorption was comparatively uniform. Typical microscopic images of the grains after coating are shown in Fig. 4 at two different magnifications and two extreme concentrations. It is observed that with increasing concentration, the adsorption of the clayey material is very much visible to the naked eye. It is shown that the LBS surface was completely covered with flaky kaolinite particles for higher concentration and that at lower concentration the LBS surface was scarcely covered.

The important role of surface roughness on the frictional behaviour of variable types of interfaces has been well acknowledged in the literature [7,38–40]. The root mean square deviation of the heights on the surface is a typical parameter used for roughness quantification. The surface roughness of the coated LBS grains at selected concentrations (i.e. 25 and 300 mg/ml at 500° exposure time) was evaluated. For this purpose, a set of five particles for each concentration were analysed and the surface roughness values for sub-regions of 20 μm × 20 μm area at five distinct locations on a single coated grain were found. The peak of a grain is the prime region for micromechanical sliding tests and so the selection of sub-regions was biased towards peak. The roughness values are noted after flattening the curved surface profile of the particle. Fig. 5 shows 3-dimensional images of flattened surface profiles at said concentrations. The texture of the kaolinite on the surface of the LBS grains is clearly visible. The average surface roughness values observed
are provided in Supplementary Table 2. There was a significant increase of the roughness from pure LBS ($S_q = 0.223 \pm 0.06 \mu m$) to the coated grains prepared in suspension with 25 mg/ml powder concentration ($S_q = 0.572 \pm 0.22 \mu m$) and 300 mg/ml concentration ($S_q = 1.013 \pm 0.14 \mu m$).

With the observations from the SEM and microscopic images, EDS analysis and roughness values, two extreme concentrations i.e. 25 and 300 mg/ml were chosen for the conduction of the micromechanical loading tests. All the samples were prepared at 500" of exposure time. Hereafter the said two cases of the coated particles are referred as Type – L and Type – H (i.e. 25 and 300 mg/ml, respectively), where “L” and “H” denote light and heavy concentrations.

5. Crushing behaviour of coated LBS particles

The study against grain crushing is important to be obtained for a complementary understanding of the behaviour of geological-granular materials. Crushing is part of the inherent process of sedimentation and lithification where soils are compacted at greater pressures, as well as part of variable phenomena including granular flows or compression-shearing of soils under the application of external loads. This work provided a preliminary study into grain crushing of the selected materials to understand the role of coating on their compression and subsequent brittle failure.

The crushing tests were conducted on pure LBS and Type – H
particles to examine and compare extreme cases of uncoated and heavily coated grains. The apparatus and procedure described by Wang and Coop [37] were used for the tests but only digital micro cameras were used for the monitoring of the tests (i.e. high-speed camera was not used in the study). For the crushing tests, the grains were chosen to be fairly spherical in outline so that the effect of morphology could be relatively diminutive. The expression proposed by Cavarretta and O’Sullivan [41] was used for calculating stresses (Eq. (1)) and also the survival probability of the grains was calculated (Eq. (2)) with the expression proposed by McDowell and Bolton [42] based on Weibull [43]. From the Weibull survival probability, plots similar to Lim et al. [44], the Weibull modulus (m), which explains the variability of strength within the population, and the characteristic strength ($\sigma_c$), which describes the minimum strength that 37% of the population possess, were calculated.

$$\sigma_l = \frac{0.9^N}{d^2}$$  \hspace{1cm} (1)

$$R_i = \exp \left[-\left(\frac{\sigma}{\sigma_c}\right)^m\right]$$ \hspace{1cm} (2)

In Eq. (1), N is the peak load or failure load and d is the geometric mean of $d_2$ and $d_3$ ($d_2$ and $d_3$ are the intermediate and smallest dimensions of the smallest cuboid circumscribing the grain). In Eq. (2), $\sigma$ is the peak load obtained for a test and $\sigma_c$ is the characteristic strength for the population.

Pure LBS and Type – H particles were tested with 30 samples each at a loading speed of 0.01 mm/min. Fig. 6 shows the survival probability against the peak stress for the LBS and the Type – H particles. Fig. 7 shows typical normal load-displacement curves obtained for pure LBS and Type – H particles. Though there was some scatter in the crushing behaviour of the particles, it was observed that the Type – H particles had extended failure displacements than that of pure LBS with a more plastic behaviour in the initial stage of crushing. This plastic deformation stage in the beginning of the trend has smoothly transformed into nonlinear behaviour to reach the failure point. There were no
abrupt changes in the trend that can signify any differences in the crushing behaviour of kaolinite-coated and pure LBS particles. One of the reasons for this behaviour could be that the coating material, between the platen and the LBS surface both at top and bottom, is under continuous compression with the increase of load. This could be observed from the images of a representative test while crushing proceeds as shown in Fig. 8, where chipping of the coating material on the sides of the particle could be observed before the actual failure was reached. This plastic deformation however did not affect the failure load of the grains and its variability. This can be observed from the comparison of the survival probability of both pure LBS and Type – H particles in Fig. 9. It is observed that the Weibull's modulus and the characteristic strength for both classes of grains are similar. The $\alpha$, and $m$ values obtained in this study are in good comparison with the values obtained by Ref. [37] for 2.36–5.00 mm pure LBS grains. For a complete characterisation of the crushing behaviour of coated LBS, additional number of tests at different concentrations, dealing with all the parameters governing the strength and also understanding mechanisms is required, which was out of the scope of the current study.

6. Grain contact testing program and results

6.1. Micromechanical apparatus and testing program

A custom-built micromechanical loading apparatus (Supplementary Fig. S2), present at the City University of Hong Kong, was used in the study [36,45]. The apparatus consists of three major systems, one in the vertical direction which is used for the study of the normal load – displacement behaviour and the application of the confinement at grain contacts, one in the horizontal direction for the conduction of shearing tests and one in the out-of-plane horizontal direction. Each system consists of micro linear actuators, load cells (100 N of capacity) and eddy current displacement sensors ($10^{-5}$mm of resolution). The grains, after mixing with coating solution and oven drying, were glued on the brass mounts, and fixed rigidly into the brass wells. One brass well is placed on the sled of the apparatus which is supported by ball bearings beneath and the second brass well is fixed on a rigid frame. Images of coated grains during the setting of a sliding test are shown in Supplementary Fig. S3. The apparatus is housed inside a Perspex chamber and a humidity controller was used to maintain the humidity at about 60% during the experiments.

Two classes of inter-particle shearing tests were conducted (i.e. Type – L and Type – H) varying the concentration of the coating material on the surface of the LBS grains. Pure LBS test results were extracted from Sandeep and Senetakis [13,38] to compare with the behaviour of the coated grains. Details of the testing program and a summary of the results are given in Table 1. For each class of grains, monotonic shearing tests were conducted at five different normal loads between 1 N and 10 N, using a new pair of grains for each test. A total of forty-five tests was conducted with the variation of normal load and concentration of coating as presented in Table 1. Both the normal load application and shearing were carried out in a displacement-controlled manner with a rate of 0.3 mm/h and 0.1 mm/h, respectively. The tests were carried out for a shearing displacement of around 50 $\mu$m where a steady state or micro-slip condition was observed. These tests on coated grains were repeated for three different pairs at each given normal load, to assess the repeatability of the normal and tangential load behaviour. With the said tests, the normal contact response and the frictional
behaviour of coated LBS grains at different concentrations and normal loads were studied and compared with the behaviour of pure LBS. For selected tests, digital microscope images were taken after the completion of the shearing to observe possible damage to the surfaces.

6.2. Normal load – displacement behaviour

After the grains are fixed in position, the top grain is moved downwards towards the stationary bottom grain at a required rate. The movement is stopped after the required load is attained. Fig. 10 gives a comparison of normal load-displacement behaviour of pure LBS, Type – L and Type – H cases for 10 N loading condition. The range of the load-displacement behaviour is shifted towards increasing displacements from the pure LBS to the heavier coated grains and in general, the normal contact response becomes softer for the heavier coated grains. In the maximum loading case of 10 N, the final displacement is less than 10 μm for pure LBS, but for Type – H particles, this displacement ranges from about 20 to 35 μm and beyond that. For each type of grains (i.e. Table 1

<table>
<thead>
<tr>
<th>No.</th>
<th>Kaolinite coating</th>
<th>Code</th>
<th>Normal load (N)</th>
<th>Tangential load (N)</th>
<th>Inter-particle coefficient of friction - μ</th>
<th>Friction Angle (deg.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Pure LBS</td>
<td>Pure LBS</td>
<td>25</td>
<td>0.25</td>
<td>0.25</td>
<td>10.2°</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td></td>
<td></td>
<td>5</td>
<td>0.95</td>
<td>0.19</td>
<td>13.9°</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td></td>
<td>7</td>
<td>1.26</td>
<td>0.18</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td></td>
<td></td>
<td>10</td>
<td>1.7</td>
<td>0.17</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>25 mg/ml</td>
<td>Type – L</td>
<td>1</td>
<td>0.21</td>
<td>0.29</td>
<td>0.21</td>
</tr>
<tr>
<td>7</td>
<td></td>
<td></td>
<td>2</td>
<td>0.24</td>
<td>0.29</td>
<td>0.21</td>
</tr>
<tr>
<td>8</td>
<td></td>
<td></td>
<td>5</td>
<td>0.5</td>
<td>0.19</td>
<td>0.21</td>
</tr>
<tr>
<td>9</td>
<td></td>
<td></td>
<td>7</td>
<td>1.1</td>
<td>1.35</td>
<td>0.22</td>
</tr>
<tr>
<td>10</td>
<td></td>
<td></td>
<td>10</td>
<td>1.3</td>
<td>1.35</td>
<td>0.22</td>
</tr>
<tr>
<td>11</td>
<td>300 mg/ml</td>
<td>Type – H</td>
<td>1</td>
<td>1.2</td>
<td>1.3</td>
<td>0.22</td>
</tr>
<tr>
<td>12</td>
<td></td>
<td></td>
<td>2</td>
<td>1.1</td>
<td>1.3</td>
<td>0.22</td>
</tr>
<tr>
<td>13</td>
<td></td>
<td></td>
<td>5</td>
<td>1.4</td>
<td>1.3</td>
<td>0.22</td>
</tr>
<tr>
<td>14</td>
<td></td>
<td></td>
<td>7</td>
<td>1.7</td>
<td>1.7</td>
<td>0.22</td>
</tr>
<tr>
<td>15</td>
<td></td>
<td></td>
<td>10</td>
<td>2.2</td>
<td>2.4</td>
<td>0.26</td>
</tr>
</tbody>
</table>

* Pure LBS values considered after Sandeep and Senetakis [13,38].

Fig. 8. Type – H particle under crushing test (a) before loading (b) Chipping of coating material while loading (c) particle breakage at peak load.

Fig. 9. Survival probability curves showing Weibull modulus and characteristic strength.
LBS, Type – L, Type – H), there was observed a scatter to the data, which has also been reported for other types of geological materials \[13,14\]. It is possible that this is influenced by the variabilities of the grain surface characteristics for each pair of grains which is reasonable to be expected for natural-geological materials. However, the trends of the normal load – displacement curves are clear and consistent as the amount of coating increases.

Fig. 11 shows the normal displacements at which the required normal loads were reached for Type – L and Type – H and also the variation in the displacements for the total set of tests at each given maximum normal load. A slight variation in the normal displacement with normal load can be observed and the probable reason, as discussed above, is because of variabilities of the surface characteristics for the different pairs of grains at a given concentration. It is also observed that this variation is greater for Type – H than Type – L because of heavier adsorption on Type – H. It is required to note that measurement of the thickness of the coating on the surface of LBS particles was not carried out.

6.3. Tangential load – displacement behaviour

After the required normal load is reached, the lower particle is moved horizontally at a target rate in a displacement-controlled mode against the surface of the upper-stationary grain, maintaining the normal load in a force-controlled mode. The grains were displaced for about 40–50 μm where a steady state or a micro-slip condition was observed. Fig. 12 shows typical tangential load – displacement curves at different normal loads for Type – L and Type – H. For a given concentration, it is observed that the steady state (or maximum reached tangential load) increases with the increase of the normal load, which is the general case observed as well for uncoated sand grains \[13\]. In contrast to the displacements reported by Sandeep and Senetakis \[13\]...
for uncoated LBS, the coated grains reach a steady state at much greater displacements. Ni and Zhu [46] described that the asperities breakage occurring continuously would cause a micro-slip condition and that breakage at the macro level would cause a steady state behaviour, which observations have been confirmed for geological materials [12,13]. Micro-slip condition was observed more severely for few cases, particularly at higher normal loads (Fig. 12, curves at 10 N of normal load). The higher displacements that were needed to reach a steady state or the micro-slip condition could be attributed, partly, to the soft flaky clay particles present in the contact region which particles were compressed between the top and bottom grains. A significant shift of the required displacements to reach a steady state (or micro-slip behaviour) in comparison to reported data on LBS, was also observed by Sandeep et al. [47] examining the micromechanical behaviour of weathered volcanic granules. These granules have a heavy natural coating on their surfaces which consists, majorly, of clay to silt sized grains. It is hypothesised that the softer nature of the Type – L or Type – H grains, similar to the weathered volcanic granules tested by Ref. [47], may have contributed to this shift of the displacements. A comparison of the tangential load – displacement curves of pure LBS and coated grains at a normal load of 5 N is given in Fig. 13(a). There is observed a shift of the curves towards lower displacements in the initial regime of behaviour for the LBS grains. The shape of the curves reaching a steady state, or the micro-slip condition, also differs for each class. The coated grains showed a softer behaviour, whereas the pure LBS behaviour was observed to be very stiff reaching, for the example of Fig. 13, a steady state at very small displacements.

The envelopes of the tangential load – normal load for the Type – L and Type – H grains along with the envelope for pure LBS are given in Fig. 13(b). Each data point on the graph represents a shearing test on a given pair of grains and the slopes of the trendlines correspond to the average inter-particle coefficient of friction (μ) for each class. It is observed that the coated grains have greater μ values in comparison to pure LBS. The increase of μ was of the order of 35% (from 0.181 to 0.246) from LBS to Type – L, while this increase was comparatively smaller between Type – L and Type – H. Ref. [38] reported that the increase of the surface roughness and/or the decrease of the apparent Young’s modulus of the contacted surfaces may lead to an increase of
the inter-particle coefficient of friction. Qualitatively, the results in Fig. 13 can be attributed to the softer nature of the coated LBS grains, which in turn increases the area of contact, as well as the greater roughness for Type – L and Type – H in comparison to pure LBS. A summary of the results, in terms of inter-particle coefficient of friction for each given test as well as the average inter-particle friction values for the different classes are given in Table 1. This increase can be ascertained from the coefficient of inter-particle friction and friction angles shown in Table 1.

Representative microscopic images showing the damage on the grain surface after shearing (Type – H at 2 N and 10 N) are shown in Fig. 14. After shearing, the coating material on the surface of the particle was scraped in the direction of shearing and it was accumulated at the end of the traverse. The image of the surface of the particle shows the accumulation of the coating material at the end of the shearing path. It is hypothesised that the material being scraped provided additional resistance against sliding since debris was created between the contacted surfaces and thus, increase in tangential load was observed. Damage analysis of pure LBS was studied by researchers and at higher normal loads, the damage on the surface was severe and was termed as ploughing [13], which ploughing behaviour was also noticed by Yang et al. [11]. Since the coating material is not an integral part of the particle, scraping phenomenon was observed even for lower normal loads in the present study. The area of contact between the grains is expected to be greater at higher normal loads, so that the amount of damage occurred to the kaolinite coating on the surface of the grains is also wider, which is confirmed from the images in Fig. 14.

7. Summary and conclusions

In this study, the normal and tangential load-displacement behaviour of artificially coated LBS particles was examined. Commercially available clay mineral, kaolinite, was used as the coating agent. Orbital shaker was used to apply the coating on the LBS particles using different concentrations of solutions and various times of exposure. Coating efficiency was analysed using EDS analysis and SEM images as well as Fig. 13. Effect of coating compound on the frictional response of LBS grains: (a) comparison of tangential load-displacement behaviour at 5 N normal load showing shift in steady state or micro-slip condition (b) tangential load versus normal load variation corresponding to steady state.
surface roughness measurements of the grains. EDS results showed an increase in adsorption (change in Al contents on the surface of the particle) with increasing concentration for a given time of exposure. Increased roughness values were observed for coated particles compared to pure LBS. Representative particle crushing tests were carried out for pure LBS and grains with heavier coating. The peak crushing loads, the characteristic strengths and the Weibull’s moduli were found to be similar between pure LBS and coated grains. However, for the coated grains, notable plastic deformation was observed which shifted the normal-load displacement curves to greater displacements. This is due to the deformation of soft coating material on the surface of LBS. Two extreme cases of coating (lowest and highest, i.e. 25 and 300 mg/ml with 500° exposure time) were selected to conduct micromechanical sliding tests and also pure LBS results from the literature were used for comparison. Similar to particle crushing tests, higher normal displacements were required to reach a given normal load. This trend was more apparent as the amount of coating increased. In the tangential direction, the curves were shown to be softer for heavier coating, and particularly at greater normal loads, micro-slip was observed rather than a steady state sliding for many of the experiments. The tangential displacement requirements to reach a given normal load were more for the experiments on gneiss sand quartz minerals. Soils Found 2013;53(5):746–57.

Additional references:


[42] Bar-Hen M, Etsion I. Experimental study of the effect of coating thickness and


[35] Torrey JD, Kriehling TL, Greenlee LF. Processing and characterization of nanoparticle coatings for quartz crystal microbalance measurements. J. Research of National Institute of Standards and Technology 2015;120.


[38] Sandeep CS, Senetakis K. Effects of Young’s modulus and surface roughness on the inter-particle friction of granular material. Materials 2018;31(2).


