Strength and Stiffness Development in Soft Soils: A FESEM aided Soil Microstructure Viewpoint

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Abstract. This paper opens with an overview of the debatable definition of soft soil that goes beyond a (CH) organic / inorganic clay and OH peat to include weakly cemented periglacial deposits of loess and alike. It then outlines the findings obtained from stiffness test on cement-stabilised soft clay. The findings are complemented with a microstructure viewpoint obtained using field emission scanning electron microscope (FESEM). Research also comprised of making cylindrical stabilised clay samples, prepared in the laboratory with various rubber chips contents and cement, and then aged for 28 days. The samples were then subjected to unconfined compressive strength (UCS) test and observations were also made of its microstructure using the FESEM. The impact of the soil microstructure on the stiffness result was studied both with the stabilized soil and also of some of the natural undisturbed loess soils. Sustainability aspect and the potential of the use of rubber chips and sand as additives to cement stabilisation are also discussed. The overall test results indicated that rubber chips and sand contributed to the improvement in unconfined compressive strength ($q_u$). The derogatory influence of moisture on the stiffness of the stabilised clay was studied simultaneously. SEM micrographs are presented that show bonding of cement, rubber chips/ sand and soft clay, granular units and aggregated / agglomerated units in loess. The paper concludes with observations on the dependence of soil microstructure on the soil strength and deformability and even collapsibility of the loess. Current practices adopted as engineering solutions to these challenging soils are outlined.

Keywords: Soft soil, ground modification, microstructure.

1. Introduction
Advances in geotechnical engineering has not yet been able to totally eradicate the occurrence of failures during construction or after a long term, particularly in challenging and even genetically (geologically) different challenging soils such as highly sensitive clays, organic clays or stiff fissured soils. The softness of a soil may be due to it being weak in strength / highly deformable or both. Embedded artefacts such as shrinkage cracks, fissures, bedding surfaces and alike are visible macro structural features that influences more so the strength (bearing capacity) of the ground rather than its deformability.

On the other hand, the morphology (size, shape, roughness and compressibility of the particles), fabric (spatial arrangement of the particles) and the level of cementation (bonding between the particles) contribute to the microstructure that further characterizes a sensitive or soft ground condition. Water is a prime cause of softness in soils. Quick clays have a liquidity index greater than one and a metastable clay microstructure resulting from the exchange of the marine pore water with fresh water. Peat has an excessively compressible organic matter content, which contains intracellular free water, inter-particle and adsorbed water in the particle cell structure. The mechanism of loess collapsibility on flooding is attributed to the breakdown of the weak cementation bonds subjected to stress concentrations. Hence a “soft soil” has a low Unconfined
Compressive Strength and high compressibility, because of relatively high water content and a fragile macrostructure and microstructure.

Loess is a yellowish, open structured, quaternary deposit of wind-blown silty sands and is widely distributed over the world, covering an estimated total area of 13,000,000 km². China has the most integrated topographic occurrence (63,000 km²; and mainly in northwestern China) of loess and the thickest loess deposit in the world. Sixty percent of these deposits tend to settle uncontrollably due to collapse of its internal structure when wetted under the action of certain pressure. This phenomenon is defined as water collapsibility [1]. Water is one of the factors attributed to the settlement of the loess when wetted. Loess, which has high strength and low compression at low natural water content, will deform uncontrollably and the strength will decrease sharply when the water content is increased (or immersed in water) [2].

The water sensitivity of loess has been the subject of investigation for a long time. The moistening uniaxial compression test and dehumidifying uniaxial compression test were taken out to investigate the variation in tendency of collapse deformation with the variation of the saturation and axial pressure [3]. The mechanism of loess collapsibility is a problem that needs further detailed study. Amongst the many theories postulated, one that is most plausible is that when the loess is soaked in water, the chemical reactions and physico-chemical reactions occur within the cementing materials. These reactions break down the particle bonding to reduce the structural strength of soil, and hence the reason for collapse of the loess. The pore diameter is often greater than the diameter of the surrounding soil particle, this overhead structure in loess is the inner condition for the collapse. Water infiltration further catalyzes the collapse. Therefore, the microstructure feature of loess is of importance for understanding loess collapsibility.

This paper presents some examples of microstructure of loess samples collected from Yucheng district of Shanxi province in China. The research study contradicts the popular belief that coefficient of collapsibility is proportional to the clay particle content.

Universiti Tun Hussein Onn Malaysia (UTHM) in general is located in an area consisting of soft soil deposits whose depths exceed 40 m. The low yield stresses, high compressibility, low shear strength and low permeability character of this soft soil deposit fall short of the desired construction specification criteria. Such properties can be improved by introducing a sustainable element within the existing various stabilization techniques. Rubber chips derived from waste rubber tyres and sand were used as additives together with cement to stabilize the clay.

Many researchers have studied the inclusion of tyre waste into soil or directly into concrete. Emiroglu et al[4] investigated mixture of tyre fibres and concrete. They studied the compressive strength and split tensile strength and both of them gave a lower strength compared to that of the plain concrete. They also concluded that increased rubber content will cause the rubberised concrete to be weaker in strength.

Segre and Joekes [5] used the Scanning Electron microscope (SEM) to characterize the rubber particles and to observe the interface between the rubber and the cement matrix. The micrographs were obtained using two different detectors: one with backscattered electrons (BEI), which are capable of distinguishing the cement (inorganic material) from the rubber (organic material) through the contrasting differences in the backscatter of the electrons, and the other using secondary electrons (SEI), which showed better surface detail.

2. Research Materials

Materials used in this study were coded, viz. loess (L), soft clay (SC), rubber chips (R), sand (S), and cement (C). Relevant information about the individual materials are given below.

2.1 Loess (L)

Microstructure images of loess samples from different regions in China can be classified into three representative types: grain structure, aggregation structure and grain-aggregation structure. The loess in Yuncheng of Shanxi province has the typical grain structure. The microstructure images of Yuncheng specimens obtained from different depths are given in Fig.1. From these images, it can be seen that the most of the solid composites in the specimen are single particles or fragmented grains. There is only a small quantity of cementing materials visible in the specimens from 14m depth. The micrograph Fig. 1a for specimen at a depth of 3m shows a coarser particle distribution as well as a slightly larger average aspect ratio of the grains in that they appear more angular than the grains in Fig. 1b (14m deep specimen).
In Fig. 1a the particle or grain contact are varied in the way of contact; point to point, point to side or point to face. These cause some open voids to exist in the microstructure. Open void is formed by loosely arranged particles or grains in loess soil. The diameter of the open void is greater than the diameters of the particles around the void. The particles are likely to fall into the void when the fabric is destroyed caused by some destabilizing action such as loading. So the existence of the open void points towards microstructural instability. Thus the microstructural features of Yuncheng specimens in 3m depth can be concluded as: grain, contact connection, and open void. The collapsibility of the loess soil at 3 m depth is high.

Specimen from a depth of 14m (Fig. 1b) show microstructural features that imply grains with the coexistence of contact connection and cement connection (there are a light cement materials between the solid composites), and embedded voids. Hence the loess soil at this depth are different demonstrating them to be non-collapsible.

2.2 Soft clay (SC)
The soft clay was retrieved in a disturbed but bulk form from a depth of 1.5m in the ground at the Research Centre for Soft Soils (RECESS) site, located beside the university. The particle size distribution of this soft clay used in the study is shown in Fig. 2. The clay contained some organic materials, roots and small fragments of decaying wood. The size of all particles was less than 0.02mm with an effective particle size D10 of 0.001 mm.

The collected disturbed bulk samples were then remoulded in the laboratory as soon as it was collected from the site and then wrapped with a few layers of cling film. Remoulding process preceded the laboratory sample preparation to ensure that the entire soft clay will be uniform and constant in moisture content. It was then placed in a tightly sealed plastic container to maintain the original moisture contents, and was stored in a temperature controlled room, set at 25°C.

Some physical and chemical properties of typical RECESS soft clay are shown in Tables 1 and 2 respectively. This soft clay was classified as CH or OH in accordance with the Casagrande plasticity chart. The electron micrograph of the soft clay shown in Fig. 3 shows thin hexagonal particles and is composed of agglomerates of clay particles with thin platy flakes.
Figure 2. Grain size distribution of RECESS soft clay, rubber chips and sand.

**Figure 3.** SEM Micrograph of soft clay (x5000).

**Figure 4.** Rubber chips used in this study.

| Properties                        | RECESS soft clay |
|-----------------------------------|------------------|
| Average water content before remoulding | ± 80 %          |
| Specific gravity (Gs)             | 2.60             |
| Soil pH                           | 3.5              |
| Colour                            | Light grey       |
| Liquid limit (LL)                 | 68 %             |
| Plastic limit (PL)                | 32 %             |
| Plasticity index (PI)             | 36               |
| Clay fraction (percentage by weight passing 2 µm sieve) | 29 % |
| Activity ($A = PI / Clay fraction$) | 1.24            |

2.3 Rubber chips (R)
Rubber chips (Fig. 4) used in this study was obtained from discarded / used truck tyres by crushing and the subsequent removal of the textiles and metal fibers. The rubber chip sizes used were between 2 to 5 mm (refer to Fig. 2). It was obtained from Yong Fong Rubber Industries Sdn. Bhd., Klang, Malaysia which produces reclaimed rubber such as rubber powder, rubber chips and rubber shreds. Rubber chips are elastic material, with Poisson’s ratio of 0.5 and elastic modulus is about 4 to 6 MPa (averaged over 0 % to 15 % strains) (Mitarai et al. 2006) [6]. Fig. 5 shows the FESEM of rubber chip that shows a characteristic rough
and irregular surface. At lower magnification, viz. 1000 times, some longitudinal shapes can be seen. Thus though the particle size distribution depicts a uniform particle size, with a uniformity coefficient <3, the particles had different shapes with a wide range of aspect ratios.

Figure 5. SEM Micrograph of rubber chips (x5000).

Figure 6. SEM Micrograph of sand (x5000).

2.4 Sand (S)
Clean river sand was used in this study and its gradation curve is also given in Fig. 2. This sand was obtained from a local building material distributor in Batu Pahat, Johor, Malaysia. The sand was oven dried before testing. The type of sand chosen for the study was easily available locally. It is noteworthy that the size distribution of the sand was chosen so as to be similar to the size distribution of rubber chips to ensure that both materials have a similar particle size range. Fig.6 shows the sand also to consist of a mixture of particle shapes; spherical and angular grains.

2.5 Ordinary Portland cement (C)
Ordinary Portland cement is a widely used stabiliser, either on its own or admixed with other additives. It has a specific gravity of 2.86 and was purchased from Holcim Malaysia Sdn. Bhd., Johor Bahru. Fig. 7 shows a micrograph observed for the cement. In cement systems, CH forms are comparatively much more finely dispersed particles throughout the microstructure. The thin platy CH crystals are frequently seen in Portland cement systems.

Figure 7. Micrographs of cement (x5000)
Table 2. Comparison of RECESS soft clay, cement, rubber chips and sand chemical composition (in percentage) by X-ray Fluorescence (XRF) test [7].

| Chemical Composition | RECESS soft clay | Cement rubber chips | Sand |
|----------------------|------------------|---------------------|------|
| SiO2                 | 59.1             | 18.30               | 96.00|
| Al2O3                | 27.5             | 4.68                | 3.44 |
| Fe2O3                | 3.66             | 2.32                | -    |
| CaO                  | 0.18             | 66.80               | 44.87|
| MgO                  | 1.09             | 1.59                | -    |
| Na2O                 | 0.18             | 0.28                | -    |
| K2O                  | 1.96             | 0.57                | -    |
| SO3                  | 5.25             | 5.03                | 6.89 |
| TiO2                 | 0.63             | 0.21                | 0.29 |

3. Experimental Methodology & Investigations

3.1 Field Emission Scanning Electron Microscope (FESEM)

FESEM is a microscope that differs from an optical microscope because it works with electrons (i.e. particles with a negative charge) instead of light. These electrons are liberated by a field emission source. The object is scanned by electrons in a random and a zigzag pattern. It can discern and help visualize very small topographic details on the surface or entire or fractioned objects as small as 1 nanometer (i.e. billionth of a millimeter).

The working principle of FESEM: A narrow scanning beam of electrons is bombarded on the surface of the sample. As a result, secondary electrons are released from each impact spot on the sample. A detector catches the secondary electrons and produces an electronic signal providing a video scan-image that can be seen on a monitor or as a digital image that can be saved and processed further. The detector used in this study was an In-lens detector.

Prior to microscopy observations, small but representative pieces were collected from one of UCS samples that replicates others and then oven dried for approximately 24 hours. A similar small piece of cement-rubber chips specimen was mounted on a copper specimen holder and then coated with a thin layer of gold coating about 15 nm thick to provide surface conductivity by using sputter coater.

When handling the small piece of clay with cement-rubber chips specimen, it was made certain that a flat surface (as the top side) was chosen as a specimen before coating. A flat surfaced specimen is needed to enhance the capture on the micrograph. The coated pieces were placed in FESEM Carl Zeiss (SUPRA 40 VP with GEMINI column) operating at 5 kV to observe the micro-structural changes in the matrix of the stabilised specimens and to visually examine the resulting products of hydration.

For fine particles such as cement-sand specimen, the steps for coating of the particles are not needed because of its fine size particles. The FESEM examination was performed on a similar small portion of the specimen (± 4 mm), but it is believed to be representative of the reaction process between rubber chips and cement matrix or sand and cement matrix.

4. Results and Discussions

According to past research, the collapsibility of loess decreases with the increasing clay particle content. However the results of the investigations on loess collected from Yucheng and Kelan of Shanxi province, Xi’an of Shanxi province in China (shown in Fig. 8), there does not appear to be an obviously marked relationship between collapsibility coefficient and clay particle content.
Coefficient of collapsibility is a parameter that quantifies the collapsibility of the loess. According to Chinese code for building construction in collapsible loess regions, GB 50025-2004 [8], the loess is called as collapsible loess when coefficient of its collapsibility is greater than 0.015.

On the whole, the correlation between the collapsibility of loess and the content of the clay particles is not good enough. The clay particle content of some samples with great collapsibility coefficient is greater than 20%, even more than 30%. It contradicts with the traditional common view that the loess with the clay particle content exceeding 15%~20% will not collapse. Hence the authors demonstrate that it is difficult to assess the collapsibility of loess from simple observation of its clay particle content alone. There lacks a one-to-one correspondence between the collapsibility and the clay particle content. However, the microstructure analysis indicates that the states of the clay particles existing in the loess have great influence on its properties.

Grain and aggregate are the main types of solid composites for loess soil. The aggregate is the group of grains or particles stuck together by cementing materials like clay particles; it acts like a single particle or grain. It is impossible to judge the collapsibility of the soil only depending on the type of solid composite. The connection types of solid composites include contact and cement. Even cement connection may be more stable than contact connection; further research is needed to judge the collapsibility of the loess soil only based on its type of connection of solid composites.

There are two types of voids, i.e. open void and embedded void, for the loess microstructure. A soil with open voids is loose, and its microstructure is not stable. If there are many open voids existing in the loess soil, it would collapse when the soil is immersed in the water and under the action of certain load. Therefore, the existence of the open voids is the definitive internal factor for collapsibility of the loess, while both loading and wetting are the definitive external factors responsible for collapse of loess.

Figs. 9 and 10 show the microstructure of the stabilized clays SC-5C-5R and SC-10C-5R after 28 days of curing. From these two figures, it is evident that the amount of calcium silicate hydrates (CSH) and calcium aluminate hydrates (CAH), which are two cement hydration products formed in the clay stabilized with cement is greater and its fabric appeared less porous than that of untreated clay. When comparing stabilized clay to the untreated clay, it can be seen clearly that a bonded structure exists, whereby the homogeneous texture of clay particles interact with cement hydration products. As a result of such inter-particle cementation, an increase in small-strain shear modulus can be expected [9].

Figure 8. Research observations between collapsibility coefficient of Loess and clay particle content.
SEM micrographs clearly indicate that cement interacts with the clay minerals to form new cementing materials, viz. calcium silicate hydrates (CSH) and aluminate hydrates (CAH). Interface bonding between cement-rubber chips and cement-sand can be observed within the stabilized clay. This consequently produces high stiffness and strength but often a low resistance to fracture, i.e. brittle behavior. These new materials functioned as additives that filled the inter-granular clay voids or sometimes coat the clay particles that result in decreased soil porosity and permeability.

The micrograph for the SC-5C-5R (sand cement sample with 5% cement and 5% rubber chips) (Fig. 9) seem to be more evenly distributed in the mix as compared to the SC-10C-5R mix. The micrograph (Fig. 10), for the latter sample shows that the matrix is more porous and the particles are agglomerated or aggregated. This can be due to the higher moisture content in the soft clay that reacts with the increased cement content (10 % C). The addition of cement to soft clay makes it more aggregated and flocculated, as compared with untreated soil. Stacks of clay particles can still be seen, indicating that not all soft clay particles are cemented. On the other hand at 10 % cement, the presence of cement seems obviously distributed throughout the mixture. These observations also complement and agree with the laboratory UCS test, where it was noted and is also known that the higher cement content will give higher strength and stiffness [7].

Fig. 11 shows a micrograph with a larger magnification (x10000) of a similar sample as that in Fig. 10. It is observed that larger pores represent a looser bonding between cement-rubber chips samples. Figs. 12 and 13 show micrographs for samples SC-5C-10S and SC-10C-10S after 28 days, respectively. As observed, both FESEM images look similar even though the cement content has been doubled in the case of Fig. 13. As compared to the cement-rubber chips samples, the cement-sand sample pores are well-knitted together and infilled with hydration products [10]. The processes of cement hydration; alite (C3S–Ca3SiO5) and belite (β-C2S–Ca2SiO4) are the main components in Portland cement, were virtually undetected in Fig. 13, confirming that primary hydration was completed within 28 days. Portlandite, naturally occurring form of calcium hydroxide (Ca(OH)2) or the peak of calcium aluminum oxide (Ca-Al-O) can also be seen in Fig. 12 with higher cement content.
When more cement is added, particle agglomeration takes place with the cementation due to secondary particles. Similar observations were also reported by Hossain and Sakai [11]. The SEM images published by Emiroğlu et al. [4] showed that the addition of waste tyre rubber in normal concrete does not affect the C-S-H formation in concrete (viz. C-S-H morphologies of normal and rubberized concrete are the same).

Some previous researchers [12] have discussed about the strength of the stabilised clay. Fig. 15 shows the compressive strength (qu) obtained from laboratory testing for untreated soft clay is 13.48 kPa. Also, SC-10C-5R sample shows no change or only a slight reduction in the strength for the field test result. Another observation is that the qu at 28 days for laboratory, semi-controlled and field test are very close to one another within 6 to 12 % of each other. A very promising field test result was observed for SC-10C-15S result that showed improvement in the properties. This shows that mixing sand into the stabilized clay gives a stiffer stabilized soil compared to the rubber chips addition. Both SC-10C-5R and SC-10C-15S showed an increase in strength as the curing days increased except in the field test result for cement-rubber chips after 28 days.
Figure 15. Undrained shear strength for SC-10C-5R and SC-10C-15S samples [12].

The results from SEM showed that a discontinuity can be observed in the rubber-matrix interface, indicating rubber adhesion to cement paste was poor, while for NaOH-treated rubber particles, an adhered joint was noticeably present. The powdered tyre rubber particles were surface treated with NaOH aqueous solution for 20 minutes, which enhanced the bonding of the rubber particles to the cement matrix. The mechanical behaviour of soil is strongly influenced by the shape, size and surface characteristics of the soil particles [14]. According to Lee and Lee [13], the soil microstructure is also a function of the individual particle properties, and strongly influences the soil behaviour. Such complementing details on particle properties can only be observed with the aid of a SEM. Prior to sample preparation the pore water in the soil needs to be removed through replacement or by freeze drying. This process of removal of pore water is very difficult to be done without disturbing the structure of the sample and some debate still exists as to what method is most suitable for different types of soils.

SEM result for bonding of kaolin-cement was that the addition of cement causes the kaolin to be less aggregated but more flocculated as compared with the untreated one [13]. They observed that 5 % or less of cement addition did not have significant effect on the mechanical properties of the stabilised soil, when observed through the SEM photomicrographs.

Hossain and Sakai [11] studied the formation of secondary particles that accompanied the cementing reaction after 7 days curing. They reached this conclusion through SEM observations of the formation of larger sized particles in the soil when 0.2, 0.4 and 0.6 % cement was added. This floculation brought the clay particles together by cementing them to form a compound or secondarily agglomerated particle.

Horpibulsuk et al [10] also observed that in cement-stabilised silty clay, there was a strength gain with time. They observed that the large pores were filled with the cementitious products that resulted in a pore volume which was smaller and consequently the total pore volume decreased.

The terms aggregation/ agglomeration is used synonymously in this paper to refer to a process of particle collection into an unorganized entity of a larger size. Flocculation is used in the context of the process of individual particles of clay aggregates becoming clotted mass or precipitated into small lumps. Flocculation occurs as a result of chemical reaction between clay particles and salt water. Conversely, dispersion is the process of breakdown of grouped particles into individual particles and evenly distributed.
5. Conclusion
The analysis of the microstructure of loess soils from different regions in China shows that loess collapse happens when the soil mainly possesses open void which is the prime inner cause of the collapsibility of loess. Overall, the FESEM observations help to demonstrate bonding between cement, rubber chips/sand and soft clay. Larger pores were seen in enlarged FESEM image that occurred for cement-rubber chips sample, which represent a looser bonding between cement-rubber chips samples as compared to the cement-sand sample. Whereas, the microstructure of cement-sand is more closely bonded compared to cement-rubber chips samples and therefore influences the geotechnical properties of the stabilized clay. Sand seems to give better result in unconfined compressive strength than the rubber chips in the semi-controlled and field test result. The potential use of rubber chips and sand in this research is not so obvious and the cement content needs to be increased in order to give greater improvement to the strength of the soft clay.

Acknowledgment
The authors thank Universiti Tun Hussein Onn Malaysia for supporting this research under the Postgraduate Incentive Research Grant. The research on Loess received financial support from NSRF in China and NSRF of Shanxi province.

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