COMPRESSIVE MECHANICAL BEHAVIOR OF RED LOESS/BLACK-RICE HUSK ASH COMPOSITE MATERIAL UNDER VARIATIONS OF FIRING TEMPERATURE

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Abstract

Red loess and black-rice husk ash (B-RHA) composite was investigated for its compressive strength. The loess was collected in Khon Kaen University and sieved through mesh No.60. Clay components of red loess were enhanced up to 9% by gravitational settling. This oriented loess was then mixed with black-rice husk ash (B-RHA) at a dry solid weight ratio of 1:1. The mixture was casted in a cylindrical mold with a diameter and height of 1.7 cm and 3.4 cm, respectively. The green bodies were fired under different fixed temperatures of 900, 1000, 1100, and 1200°C. Tridymite and cristobalite were dominant silica polymorphism found under the applied firing temperatures especially at 1200°C. Uniaxial compression tests were performed on un-oriented loess, unmixed oriented loess, and oriented loess/B-RHA composite. The oriented loess was found to be more rigid than the un-oriented loess and the loess composite. The elastic modulus of the oriented loess was found to be as high as 4 GPa, which is twice as much compared to that of the un-oriented loess at the same firing temperature of 1200°C. It can be surmised that the structural network of red ferric oxide-clay loess stabilizes the strength of the material when fine-grained silica is reduced, resulting in a higher strength of loess after orientation. Fired bodies of loess/B-RHA composite showed a significant decrease in its elastic modulus which is approximately 45 times less compared to that of the unmixed oriented loess. This is a result of an increased amount of voids of the composite due to the incineration of B-RHA. In addition to the reduction of its elastic modulus and compressive strength, the composite also shows extreme brittleness. However, the relationship of both yield strength and elastic modulus of the composite as a function of temperature shows a strong linear behavior in contrast to that of the un-oriented loess and unmixed oriented loess. It can be inferred that B-RHA in the composite decomposes into voids at a similar rate that quartz transforms to higher phases during the firing process.

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Introduction

Loess, silt-sized sediment or aeolian sediment, is considered a challenging material in engineering construction and has been categorized as a collapsible soil. It is developed by the collection of wind-blown sand-silt sediment. In the northeast of Thailand, red loess can be found extensively; in Khon Kaen Province alone it is found to cover 122 sq.km. with a volume of 244 Mcu.m (Department of Mineral Resources, 2009). Due to its notable problematic behavior in construction, its utilization and identification of strength has been limited which has been caused by wetting. Khon Kaen loess under various conditions causing the foundation damage of building on this soil are reported. A critical impediment for several constructions is mostly found on loess deposits. Physically, their deposits in Thailand are sorted by color, red and yellow loess because of differences in oxidation state (Phien-wej *et al.*, 1992). However, their lithological properties and mineralogy are similar. Commonly, high elevation regions including Khon Kaen are covered with thick loess deposits with the thickness of a few meters to greater than 6 m. The majority of the area in the northeast of Thailand is covered by loess (Punrattanasin and Gasaluck, 2008) this also includes the soil within Khon Kaen University main campus which is known as Mor-Din-Daeng soil (Boonsaner, 1980). Due to the lack of knowledge, loess has been used in construction and has been reported to cause damage such as at Lam Chuang Krai Dam, Lam Sam Lai Dam, and Lam Mun Bon Dam, the latter being most widely known nationally.

There are several uses of loess in the various industries. Loess is used as a composite material for an additive to develop swelling properties of superabsorbent network. It mixed with xanthan gum and acrylic acid to synthesis organic-inorganic composite materials (Feng *et al.*, 2014). A specific loess composite is fabricated by blending loess, wood fragment, and water of 40, 40, 20% by volume, respectively. This mixture is applied to construct support plates and insulation sheets (Oh, 2002). The mixtures with loess are efforts to use its good property. For example, chemical components of loess consist of hydrous magnesium aluminium silicate. This hydroxyl functional group obtains hydrophilic property. Thus, loess is an ultimate solution for the inorganic material mixing with both organic and inorganic components. Rice husk (RH) from the rice mill is also a target material for the loess mixture. Rice husk ash (RHA) produces nano-size amorphous silica which can be combined into composites with loess to improve its mechanical property. RHA is normally utilized to improve soil structure since amorphous silica can be easily extracted by heat treatment (Della *et al.*, 2006). Moreover, RHA consists of crystalline tridymite and α-cristobalite, and is preferred to be used as an option for silica source (Prasetyoko *et al.*, 2006; Kordatos *et al.*, 2008). The goal of this research is to obtain understanding on the strength of fired-loess/black-RHA composites with variations in compositions and firing temperatures of 900, 1000, 1100, and 1200°C. RHA contains silica in the form of α and β quartz, tridymite, and cristobalite which are natural polymorphs that can exist metastably. Therefore, the strength depends on the firing temperature since different temperatures will transform silica into different minerals.

Methodology

Red Loess Purification

Red loess, the main body of the composite, is collected from a foot hill slope of a main lake in Khon Kaen University. It is refined by suspension method with clean water. Refining process is performed by mixing loess in water at 15 wt% and allowing for gravitational settling. The settled particles are separated and discarded. After 4-5 sedimentation cycles, the supernatant suspension is decanted off with purified loess.

This suspension is filtered and dried in an oven at 110ºC. Dried loess is ground and sieved through mesh No.60, obtaining particle sizes finer than 0.25 mm. Loess from gravitational settling is called oriented loess. Loess without this refining procedure is called un-oriented loess.

Pretreatment of Black-Rice Husk Ash

Rice husk ash is prepared by acid treatment, drying, and sieving process. Rice husk is boiled in 5vol% of hydrochloric acid at 150°C for 1 h to remove heavy metal impurities. After soaking in hot-clean water overnight, it is sintered and transformed into ash in the oven at 250°C for 24 h to eliminate the majority of volatile matters. During this process, rice husk will change from yellow (fresh rice husk) to black (or black-rice husk ash). The ash will be ground and sieved through the mesh No.60 according to the ASTM C136/136M-14 guidelines (American Society for Testing and Materials, 2014). The prepared ash is stored in dry containers.

Oriented Loess/Black-Rice Husk Ash Mixture

Oriented loess is blended with black-rice husk ash (B-RHA) and continuously stirred to develop a uniform mixture before adding clean water. This very viscous slurry is cured for 24 h. The mixing composition of the oriented loess and B-RHA is maintained at a 1:1 ratio. The mixture is casted through a cylindrical mold which has the height twice its diameter and then left overnight. The casted block is dried in the oven at 110°C for 24 h. This loess-mixed block is fired at four different temperatures at 900, 1000, 1100, and 1200ºC. The final firing temperature is maintained for 6 h and then the fired-loess block is left to cool in a desiccator at ambient temperature for about 12 h.

Methods of Characterization

Water Absorption

Two tests are conducted for comparison, one with un-oriented loess (without B-RHA) and one with oriented loess with B-RHA. These blocks are fired at four different temperatures at 900, 1000, 1100, and 1200ºC before water absorption testing. Water absorption implies the internal structure of the body which relates to the mechanical strength. Description of this method is as described by the ASTM C20-00 guideline (American Society for Testing and Materials, 2015).

Mineral Composition

X-ray Diffractometry (Bruker AXS D8 ADVANCE X-Ray Diffractometer, XRD) is used to analyze mineral compositions of the fired un-oriented loess and the oriented loess/B-RHA mixture. The Cu-Kα radiation is selected for this powder diffractometer. Diffractograms are achieved and recorded by step scanning from 4-70º2θ, with a step size of 0.02º and counting for 2 sec per step. The area under a curve of the maximum peak position of each mineral is quantified and presented as a semi-quantitative analysis.

Mechanical Compressive Strength

Mechanical property of dry-fired single blocks is investigated according to the ASTM C773-88 guidelines (American Society for Testing and Materials, 2011). Uniaxial compressive strength test is accomplished by using the Instron 5567A Universal Testing Machine (UTM).

Results and Discussion

Water absorption of the oriented loess is significantly less than the oriented loess/ B-RHA mixture with a difference of at least 5 times (Table 1). The minimum water absorption of both types of samples is found from specimens fired at 1200ºC because the shrinkage of voids in the bodies by the high temperature. However, the overall water absorption of the mixture between the oriented loess and black-rice husk ash is found to be significantly higher than unmixed oriented loess, especially at 1000ºC (Figure 1). An explanation for this occurrence is that organic carbon in black-rice husk ash (B-RHA) decomposes to carbon dioxide gas and other volatile matters. Thus, the interior space within the B-RHA structure is created and the volume of voids increase after firing.

At various temperatures, different minerals start to transform, however, tremendous changes of physical property and mineral characteristics can be observed at 1000°C (Figures 2 and 3). XRD results in Figure 2 show that quartz is the main mineral found in both un-oriented loess and oriented loess/B-RHA. However, additional minerals become more profound at 1200°C, such as mullite, tridymite, and cristobalite. These three novel minerals are transformed by thermal interaction. Mullite transformation occurs from alumino-silicate minerals (clay minerals, such as kaolinite and illite). However, tridymite and cristobalite transform from β-quartz. Supplement of black-rice husk ash can increase cristobalite up to 35%, especially at the temperature of 1200°C.

According to the compressive stress-strain curve, the un-oriented loess presents higher resilience than the oriented loess with a smooth *S*-shape (Figure 4(a)). More elongation and resilience appear at 900°C. However, the oriented loess proves to be more rigid and cracks during compressive strength test at 1000 and 1100°C as shown in Figure 4(b). It is believed that the structural network of red ferric oxide-clay loess stabilizes the strength of the green bodies. Mechanical properties of the mixture (oriented loess and black-rice husk ash, oL/R) becomes extremely brittle and show no sign of plastic deformation (Figure 4(c)), moreover, there is a significant reduction of yield strength and elastic modulus compared to both un-oriented loess and unmixed oriented loess (Figure 4(d)). The lowest yield strength and elastic modulus are found in the oL/R mixture (Figure 5). The reduced yield strengths of the oL/R mixtures are mainly affected by voids inside their bodies. Black-rice hush ash in the oriented loess converts into voids after heating. Thus, their water

Table 1. Water absorption of oriented loess (oL) and oriented-loess/black-rice husk ash (oL/R) at different temperatures ($T_{oL\text{-}1200}$ and $T_{oL/R\text{-}1200}$ are specimens fired at 1200°C)

Temperature (°C)	Water Absorption (%)		Ratio of Water Absorption		
	oL	oL/R	(oL/R) : oL	T_{oL} : $T_{oL\text{-}1200}$	$T_{oL/R}$: $T_{oL/R\text{-}1200}$
900	18.09	95.31	5.3	1.5	1.1
1,000	18.07	102.18	5.7	1.5	1.2
1,100	12.98	90.24	7.0	1.1	1.1
1,200	11.83	85.57	7.2	1.0	1.0

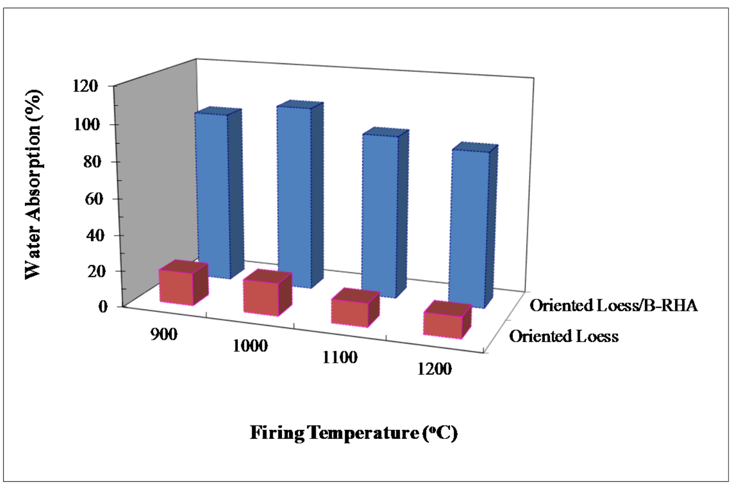

Figure 1. Water absorption as a function of firing temperatures at 900, 1000, 1100, and 1200°C of oriented-loess/black-rice husk ash and oriented loess

absorptions are remarkably higher than plain oriented loess (oL). The loose structure which traps water in their interstices results in reduced strength despite the increased presence of cristobalite and tridymite which are known to have higher strength. By contrast, its yield strength and elastic modulus as a function of temperature show a more linear behavior than the un-oriented loess and unmixed oriented loess.

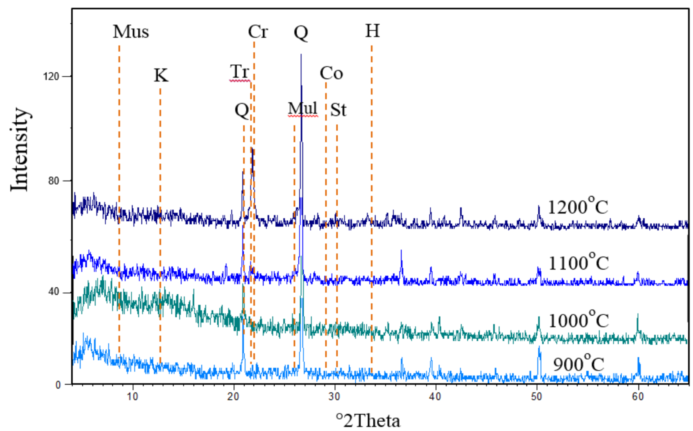

Figure 2. Diffractogram of oriented loess mixed with black-RHA (1:1 ratio) at 900, 1000, 1100, and 1200°C (Q = Quartz, Co = Coesite, St = Stishovite, Tr = Tridymite, Cr = Cristobalite, K = Kaolinite, Mus = Muscovite, Mul = Mullite, H = Hematite)

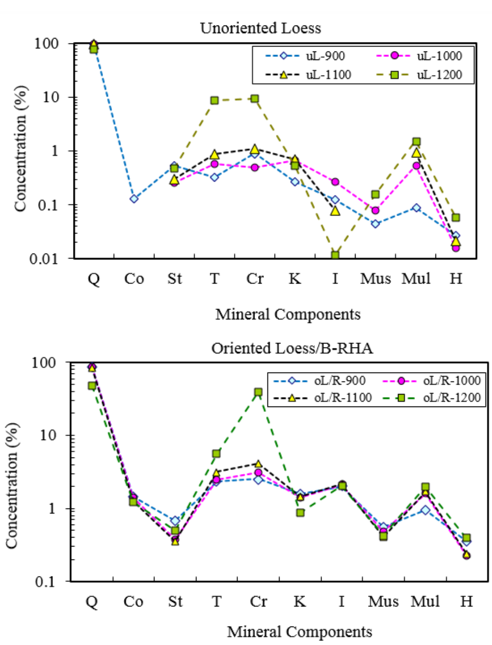

Figure 3.Concentrations of each mineral component for: un-oriented loess without black-RHA (above)and oriented loess mixed with black-RHA at 1:1 ratio (below), fired at 900, 1000, 1100, and 1200°C (Q = Quartz, Co = Coesite, St = Stishovite, Tr = Tridymite, Cr = Cristobalite, K = Kaolinite, I = Illite, Mus = Muscovite, Mul = Mullite,

Conclusions

The compressive mechanical behavior of a composite material consisting of red loess and rice husk ash (RHA) was investigated. This red loess was sieved through 60-mesh (250 μm). Clay minerals of red loess were increased up to 9% by gravitational settling. This oriented loess was then mixed with black-rice husk ash (B-RHA) at 1:1 ratio by weight. The green

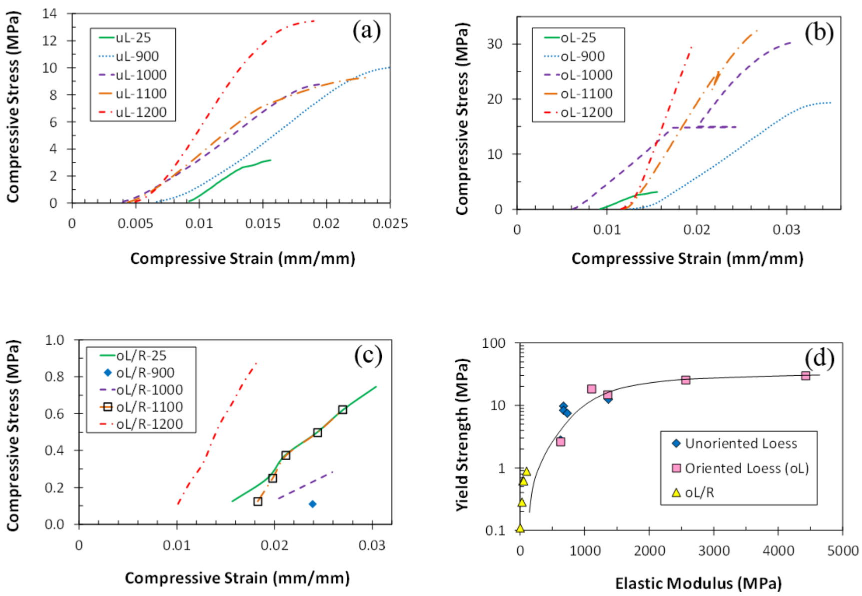

Figure 4. **Compressive stress as a function of strain for (a) un-oriented loess (uL), (b) oriented loess (oL), (c) mixture of oriented loess and black-rice husk ash (oL/R), and (d) relationship between yield strength and elastic modulus of uL, oL, and oL/R at each firing temperatures . Note that -25 stands for un-fired specimens (green bodies)**

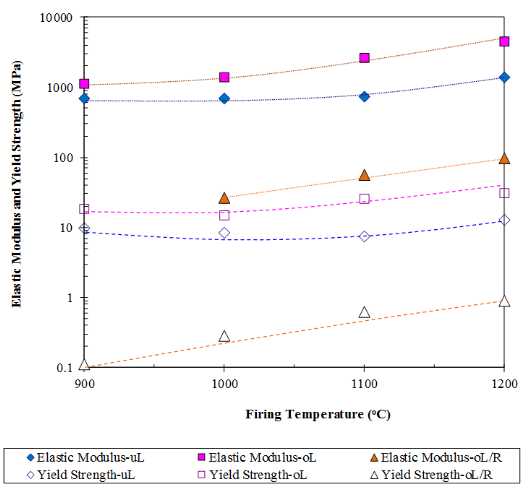

Figure 5. **Yield strength and elastic modulus as a function of firing temperature for all types of specimens**

bodies were fired under fixed temperatures of 900, 1000, 1100, 1200°C. Water absorption of the mixture between the oriented loess and black-rice husk ash was found 5-7 times higher than oriented loess. Tridymite and cristobalite are dominant minerals found under high firing temperatures. Uniaxial compression tests were performed on un-oriented loess, unmixed oriented loess, and oriented loess mixed with black-rice husk ash. The elastic modulus of the oriented loess was found to be as high as 4 GPa, compared to the un-oriented loess at the same firing temperature of 1200°C. Relationship of the stress-strain curve reveals that the oriented loess has higher yield strength than un-oriented loess. It is considered that red ferric oxide-clay loess stabilizes the strength of the material when fine-grained silica is diminished resulting in a higher strength of loess after orientation. However, fired blocks of the oriented loess/B-RHA composite presents a decrease in its yield strength and elastic modulus for approximately 45 times less compared to that of the unmixed oriented loess. It can be concluded that the remarkable amount of voids presence in the composite strongly affects the mechanical properties by reducing both the yield strength and elastic modulus in addition to resulting in a brittle material. Interestingly, the composite shows a strong linear relationship between the compressive strength and firing temperature. This is possibly due to the rate of B-RHA depletion being similar to the rate of quartz transformation into higher phases.

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