Chemical Stabilization of Loess in Northeast Thailand Using the Mixture of Calcined Marble Dust Waste and Sugarcane Bagasse Ash Waste

Phongthorn Julphunthong

Department of Civil Engineering, Faculty of Engineering, Naresuan University, Phitsanulok, Thailand Research Unit for Innovative Construction Materials, Faculty of Engineering, Naresuan University, Phitsanulok, Thailand Research Center for Academic Excellence in Applied Physics, Faculty of Science, Naresuan University, Phitsanulok, Thailand E-mail: pop_civil@hotmail.com

ABSTRACT: The main objective of this study was to investigate the possibility of utilizing calcined marble dust waste and sugarcane bagasse ash waste as the chemical stabilizer for loess. Loess is widely distributed throughout the northeast area of Thailand. This soil type has a large loss of shear strength and a significant increase in compressibility upon wetting, which are obstructive behaviors for construction materials. The study was divided into two sections. The first section dealt with the study of synthesizing cementitious materials from calcined marble dust waste and sugarcane bagasse ash waste. The second section focused on studying the stabilization of loess. This consisted of observing the effect of stabilizer on the basic properties of soil-stabilizer mixtures and the improvement in its mechanical properties. The additive ratios of stabilizer which were studied were between 0-25 percent by weight. The physical and chemical properties of loess, calcined marble dust waste and sugarcane bagasse ash waste were studied by X-ray diffraction, X-ray fluorescence, thermogravimetric analyzer, differential thermal analysis, laser diffraction particle size analyzer, Brunauer, Emmett and Teller, transmission electron microscope and scanning electron microscope analytical techniques. After analyzing the physical and chemical properties of the stabilizing materials, the sugarcane bagasse ash waste and the marble dust waste which was calcined at 1000 °C for 2 h, mixed with the ratio of 1:1, and then selected as the stabilizer for loess improvement. It was found that the liquid limit and plastic limit of the treated soils had been decreased with the percentage increase of stabilizer. The optimum moisture content tended to increase while the maximum dry density was reduced. This corresponded to an increasing of the stabilizer fraction. By adding stabilizer at 25%, the soaked and unsoaked California bearing-ratio were increased by about 185% and 140%, respectively. Unconfined compressive strength of specimens with 25% stabilizer was dramatically increased with 28 days curing time.

Keywords: Loess, Calcined marble dust waste, Sugarcane bagasse ash waste, Cementitious materials.

1. INTRODUCTION

It has been estimated that loess covers nearly 10% of the surface area of the earth [1]. Loess soils are usually found in arid or semiarid climates and their physical and chemical characteristics mainly depends on their geological origin. In Thailand, it has been estimated that the area covered by loess, especially in the northeast of the country, exceeds 168,800 km² or about one-third of the total area of Thailand [2]. Loess is composed of predominantly silt-sized particles with a tiny amount of clay. The silt-sized particles are not in contact with each other, but are separated by clay coatings. For an undisturbed structure in an unsaturated state, the cohesion is developed due to the capillary tension in the clay coating. However, if the water content increases and the clay binder become saturated, the clay bonding tends to be relatively weak resulting in high decrease in shear strength [3]. The loss of shear strength with increased moisture content of loess is a significantly obstructive factor for the use of loess as a construction material.

There are several techniques for improving the engineering properties of soils for use in construction. One of the extensively used techniques for the improvement of problematic soil is treatment with cementitous additives such as cement. Cement is often used as an additive to improve strength and compressibility of soils in tropical areas. Stabilization of soil with cement enhances its strength and also reduces its compressibility compared to that of untreated soil [4-6]. Arrue et al. [7] extensively investigated the stabilization of loess with cement for civil engineering applications. The study results showed the unconfined compressive strength of treated soil can be increased by 450% compared to untreated soil. Moreover, cement addition increased the stability of the soil and protected structural collapse. They suggested that the samples composed of loess material and cement, with cement contents above 7.5%, have physical and mechanical properties suitable for sustainable employment in different civil infrastructure projects. Though cement shows high performance for stabilized soils, an application of cement as an additive for soil stabilization is

expensive since the cost of cement material is around of 57 percent of the total cost [2].

Recently, a number of waste materials have been used in the stabilization of soils in order to reduce both environmental problems and the requirement for traditional additives such as lime, fly ash and rice husk ash. For instance, Eren et al. [8] demonstrated that the use of 4-10% lime increased the mechanical strength of soil while significantly reducing water absorption. Three types of chemical reactions have been identified as occurring in the soil: when lime is added, when ion exchange occurs, and when a pozzolanic reaction and carbonation of lime occurs. In the case of waste biomass ash, many researchers have reported that rice husk ash consists of a large amount of silica with a high specific surface that is very suitable for activating the reaction of soil and lime [9]. Some researchers have shown that rice husk ash is a promising pozzolanic material to improve lime or cement-stabilized soils [10, 11]. After being surveyed, there were two interesting by-product materials which are possible for use as soil-stabilizing materials. The first one is marble dust waste (MDW), which is obtained as a by-product of marble sawing and industrial shaping. Saboya et al. [12] explained that the calcium carbonate (CaCO₃) in marble dust is initially decomposed to calcium oxide (CaO) or free lime at around 800 °C. Hence, calcined marble dust waste (CMDW) could be used as a soil stabilizing additive similar to lime. The next material which was characterized from a physical and chemical properties point of view for use as an additive was sugarcane bagasse ash waste (SBAW). Several reports suggest that SBAW, which consists of high silica oxide composition [13-15], could probably be used as a soil stabilizing additive like the rice husk ash. For the above reasons, the combination of CMDW plus SBAW as loess stabilizers is very interesting. Therefore, this study aims to synthesize calcium oxide from MDW. The chemical and physical properties of loess, CMDW and SBAW were studied. The additives composition proportions effect on the engineering properties of stabilized loess was investigated.

2. MATERIALS AND METHODS

2.1 Materials

Loess, MDW and SBAW were used as materials in this study. The soil used in the present study is red loess obtained from Khon Kaen University, Thailand. MDW and SBAW in this research were produced in a local marble factory company and sugar factory company in Kamphaeng Phet, Thailand. The raw materials from the source of production are shown in Figure 1.

2.2 Methods

The experimental procedure of this work has been divided into three parts. In the first part, some physical-chemical tests of MDW, SBAW and loess were performed. Physical properties including specific gravity, determination of Atterberg limits, sieve analysis, and hydrometer test results were obtained by ASTM methods [16]. The chemical composition of the raw materials was analyzed using an X-ray fluorescence (XRF) analyzer. Mineralogical analysis was done by X-ray diffraction (XRD) using monochromatic Cu-Ka radiation at a scanning speed of 1.5° (2 θ) min⁻¹ in a conventional diffractometer. The crystalline phases were identified by comparing the intensities and positions of the Bragg peaks with those listed in the JCPDS databases. For better physical characterization of the MDW and the SBAW, their grain size distributions and specific surface area were measured using a laser diffraction particle size analyzer (PSA) and a Brunauer, Emmett and Teller (BET), respectively. A scanning electron microscope (SEM) and a transmission electron microscope (TEM) were used to observe the morphology and the particle size of the raw materials. The second part of the experimental procedure was concerned with the chemical reaction of MDW at high temperature. The reaction of the MDW at high temperature was investigated by thermogravimetric analysis and differential thermal analysis (TGA-DTA) using a heating rate of 10°C/min from room temperature to 1,400°C. After that, the MDW powders were calcined at various calcination temperatures ranging from 200 to 1000 °C with a soaked time of 2 h to ensure a complete chemical reaction. In the third part, the experimental procedure focused on the soil stabilization. The MDW which was calcined at an optimum temperature was mixed with an equal amount by weight of SBAW for use as a soil stabilizer. This stabilizer was then used to treat samples of loess, by mixing them together with various proportions of stabilizer. Comparisons of the engineering properties of resultant loess mixture with the various proportion of the stabilzer, from 0% (untreated loess) to 25% by weight in 5% steps were performed using the consistency limit test (ASTM D4318), the standard compaction test (ASTM D698), the CBR-test (ASTM D1183) and an unconfined compression test (ASTM D2166).

3. RESULTS AND DISCUSSION

3.1 Chemical and physical analyses of raw materials

The chemical composition of the materials, which consist of loess, MDW and SBAW is shown in Table 1. The chemical components of loess comprised mainly silicon dioxide, aluminium oxide and ferric oxide. The main composition of silicon dioxide suggests that loess contains significantly large amounts of quartz. In the case of MDW, it should be emphasized that this material consists of pure calcium oxide which indicated that this powder comes from a calcite-type marble. The XRF results show that the major pozzolanic components of $SiO_2 + Al_2O_3 + Fe_2O_3$ for SBAW is 90.70%, hence satisfying the requirements of standard specification for coal fly ash and raw or calcined natural pozzolan for use in concrete (ASTM: C618-12a).

Table 1 Chemical composition (% by mass) of loess, MDW and SBAW

Chemical composition	Loess	MDW	SBAW
SiO ₂	56.24	-	79.26
Al_2O_3	24.99	-	9.21
Fe ₂ O ₃	12.38	-	2.23
CaO	0.95	100	1.97
SO ₃	0.41	-	0.50
MnO ₂	0.29	-	0.16
Na ₂ O	-	-	-
K ₂ O	3.27	-	5.06
P ₂ O ₅	-	-	1.09
TiO ₂	1.32	-	0.51

The suggestion of large amount of quartz in loess is emphasized again in the XRD analysis (Figure 2(a)). The diffraction patterns show that loess consists of main crystalline phases of quartz (Refer to JCPDS file no. 46-1045). The matching position of diffraction patterns observed in Figure 2(b) demonstrated the mineralogy of SBAW is quartz similar to loess. The lower level in the intensity peaks of SBAW compared with loess indicates that the Si-O band structure of SiO₂ in SBAW is disordered which caused the crystal silica to be transformed to amorphous phase [17].



Figure 1 Raw materials of the study; (a) red loess, (b) MDW, and (c) SBAW



Figure 2 XRD patterns of materials; (a) loess, and (b) SBAW

The particle size distributions of the studied materials are depicted in Figure 3. In the case of loess, the particle size was analyzed using sieve analysis and hydrometer analysis. It can be observed that more than 70% of samples are sand size particles and 27% are silt size particles. Grain size distribution curves of SBAW and MDW were obtained from PSA. It is found that the particle size distribution curves of SBAW and MDW are similar. D_{50} of SBAW is 10.47 µm being almost the same as that of MDW (Table 2). The physical properties such as specific gravity, Atterberg limits and specific surface area of the raw materials used are given in Table 2. Specific gravities of loess, SBAW and MDW are 2.70, 2.02, and 2.86, respectively. The uniformity coefficient and the coefficient of gradation values suggest that the loess sample is well-graded soil. The Atterberg limits results reveal loess is non-plastic soil and can be classified as SM.



Figure 3 Particle size distribution curves of loess, MDW, and SBAW

Table 2 Typical physical properties of loess, MDW and SBAW

Physical properties	Loess	SBAW	MDW
Specific gravity	2.70	2.02	2.86
D ₉₀ (μm)	4,750	29.93	32.68
D ₅₀ (μm)	290	10.47	10.81
D ₁₀ (μm)	8.7	3.80	3.28
Uniformity coefficient (C_u)	44	3.16	4.10
Coefficient of gradation (C_c)	1.8	1.35	1.54
Liquid limit (%)	18.98	-	-
Plastic limit (%)	NP	-	-
Soil classification (USCS)	SM	-	-
BET specific surface area (m^2/g)	-	38.85	7.93

Figure 4 shows the TEM micrographs of loess. At low magnification, loess showed a sub-angular to sub-round shape (Figure 4(b)). Tiny amount of clay particles can be observed by using very high magnification (inside ellipse marking in Figure 4(b)). Figure 5 and Figure 6 provide the SEM micrograph of SBAW and MDW, respectively. The SBAW powders were <u>comprised</u> mainly of coarse and angular, sub-angular, and irregular particles with a wide range in particle size distribution (Figure 5(a)). When using higher magnification, it can be observed that larger particles result from the agglomeration of the small particles (Figure 5(b)).



Figure 4 TEM micrographs of loess; (a) original magnification ×9,700, and (b) original magnification ×197,000



Figure 5 SEM photographs of SBAW; (a) original magnification ×1,000, and (b) original magnification ×3,000

In Figure 6(a), the SEM micrograph demonstrates that MDW exhibits a narrow particle size distribution compared with SBWA. Although the average particle size of SBAW and MDW is similar, the specific surface area of SBAW is significantly higher than the MDW surface area (Table 2). This indicates SBAW has a higher porosity than MDW.



Figure 6 SEM photographs of marble dust waste; (a) raw marble dust waste, (b) marble dust waste calcined at 600 °C for 2 h, (c) marble dust waste calcined at 800 °C for 2 h, and (d) marble dust waste calcined at 1,000 °C for 2 h.

3.2 Chemical reaction of MDW

The TGA–DTA curves for investigating the chemical reactions of the marble powder are shown in Figure 7. A sharp weight loss occurs from about 600 $^{\circ}$ C – 900 $^{\circ}$ C which corresponds to a strong endothermic peak at 873 $^{\circ}$ C in the DTA curve. These observations related to the reaction when calcium carbonate (CaCO₃) decomposes to produce quick lime (CaO) and carbon dioxide as shown in Eq. 1 [18].

$$CaCO_{3(s)} \longrightarrow CaO_{(s)} + CO_{2(g)}$$
 (1)



Figure 7 TGA-DTA curves of MDW

Figure 8 illustrates the XRD patterns of raw marble powder and marble powders calcined at various calcination temperatures between 200 and 1000 °C with the soaked time for 2 h. The XRD patterns of raw marble powder suggested that the material consists of pure calcite (CaCO₃), which was consistent with the chemical composition analyzed using XRF analyzer. The XRD patterns maintained their original characteristics even though the sample was calcined at a temperature of 600 °C. The diffraction peaks of quick lime (CaO, indexed as portlandite) were initially observed when the sample was calcined at 800 °C. When increasing the calcining temperature to 1000 °C, the diffraction patterns indicated that the CaO phase content was increased and showed a major component phase. There was very little of the CaCO3 phase remaining in the system, which was observed at the diffraction peak at 2θ of 29° . The variation of CaCO₃ and CaO phases was consistent with the thermal analyses results.

The SEM photographs of the marble powders calcined between 600 °C-1,000 °C are shown in Figure 6. It can be observed that calcination of the marble powders at 600 °C caused the CaCO3 particles initially to melt and merge into larger particles (Figure 6(b)). In Figure 8 (c)), the CaCO₃ was decomposed and small crystallite size particles of CaO were formed. By increasing the calcination temperature to 1000 °C, the grain growth of CaO became increasingly active (Figure 6(d)). This trend is consistent with those reported by Fischer [19] and Dash et al. [20]. Moreover, the result from the TGA-DTA investigation, XRD analysis and SEM study were consistent with each other. From the result of this part, the MDW which was calcined at 1000 °C for 2 h (CMDW1000) shows potential characteristics for use as a soil stabilizer due to the calcite being completely transformed to CaO or quick lime. When compared to Ordinary Portland Cement (OPC), which is a common soil stabilizer, CMDW1000 is a more convenient synthesizing process. This is especially true when the energy consumption and CO₂ emissions that are required for OPC production are taken into consideration. When considering the chemical composition, physical properties, undesired grinding process, lower synthesizing temperature compared with OPC, CMDW1000 becomes an interesting alternative material for use as a soil stabilizer cooperating with the pozzolanic materials.



Figure 8 XRD patterns of marble dust waste (Raw-MDW) and marble dust waste calcined at various temperatures

3.3 Engineering properties of the mixture of CMDW1000 and SBAW stabilized loess

After the physical and chemical properties of the stabilizing materials were analyzed, SBAW and the CMDW1000 were selected as the co-operating stabilizers for loess improvement. Since this work is a preliminary study to demonstrate possible use of a mixture of CMDW1000 and SBAW as a soil stabilizer, the SBAW and CMDW1000 were weighed and mixed into a ratio of 1:1 and tested. The stabilizer was used to stabilize the loess with stabilizer/loess soil (S/L) ratios between 0 - 25% by weight of dry mass of loess. The effect of the stabilizer fraction on the consistency limits of the stabilized loess specimens is shown in Figure 9. It was found that as the amount of stabilizer was increased from 0 to 25 %, the liquid limit and plastic limit of the specimens slightly increased while the plasticity index changed insignificantly. The variation of Atterberg limits of the treated soil can be related to the stabilizer's influences on consistency. The addition of SBAW, which is non-plastic material, reduces the plasticity index of the loess [21]. Adding CMDW1000, which is also referred to quick lime, leads to increasing plasticity limits and to a reduced plasticity index [22].



Figure 9 Variation of consistency limits with stabilizer percentage

The moisture-density relationships of untreated and treated soil with different percentages of stabilizer are shown in Figure 10. The figure shows that adding stabilizer increased the optimum moisture content (OMC) and diminished the amount of the maximum dry density (MDD). The increase in OMC is probably attributed to the high specific surface area of SBAW (38.85 m²/g), which increases the amount of water needed to be adsorbed in order to compact the soil-stabilizer mixtures [23]. The addition of higher stabilizer percentages will reduce the MDD. The changing of MDD due to an increase of stabilizer can be considered in two ways. Since both CMDW1000 and SBAW stabilizers exhibit smaller particle size compared with soil, the stabilizer will fill the voids of the soils resulting in an increased maximum dry density. On the other hand, increasing the percentage of stabilizer will decrease the MDD as a result of the lower specific gravity of the SBAW which replaces the soil in the mold and then reduces the mass of material for the same volume. However, adding stabilizer reduces the MDD of the soilstabilizer mixtures in this study. Although the MDD was decreased when the stabilizer fraction increased, the increase of stabilizer percentage caused the soaked and unsoaked CBR values to enhance (Figure 11). The soaked and unsoaked CBR were increased by about 185% and 140%, respectively for the soil-stabilizer mixtures at 25% stabilizer percentage. These results suggest a CMDW1000-SBAW mixture is an effective stabilizer for the stabilization of loess for pavement materials application.



Figure 10 Compaction test results due to the addition of stabilizer to the soil



Figure 11 Effect of stabilizer addition on CBR

Figure 12 illustrates the strength development of the soilstabilizer mixtures after 1, 7, 14 and 28 days of curing. In general, there is an increase in unconfined compressive strength (UCS) of the specimens with an increase of the stabilizer fraction. Further, it can be observed that, the specimens prepared with a high fraction of stabilizer have shown to have a marginally long term compressive strength compared to those of specimens with a low stabilizer fraction. The increased compressive strength of specimens prepared with a high percentage of stabilizers can be attributed to the pozzolanic reaction of calcium hydroxide, which occurred because of the hydration reaction of CMDW1000 or quick lime (Eq. 2), and SiO₂ or Al_2O_3 from SBAW, which helps the formation of cementitious compounds in the specimens.

$$CaO + H_2O \rightarrow Ca(OH)_2$$
 (2)

The dramatic increase of compressive strength of the specimens with high stabilizer with curing at 14 and 28 days suggest the pozzolanic reactions occurred at a slow reaction rate.



Figure 12 Relationship between UCS and stabilizer content with various curing time

4. CONCLUSION

In this study, the chemical and physical properties of CMDW and SBAW were also studied. The results indicate that SBAW has a large amount of the oxides required for pozzolanic reaction, while CMDW1000 shows a major phase of portlandite or CaO. The mixture of CMDW1000 and SBAW can be used as a soil stabilizer because their pozzolanic reaction produces cementitious materials. A mixture of CMDW1000 and SBAW was selected as a stabilizing agent and mixed with some selected loess from the northeast part of Thailand. The liquid limit and plastic limit slightly increased while the plasticity index insignificantly changed with increasing stabilizer percentages. The degree of stabilizer fraction caused the decrease and increase of maximum dry density and optimum moisture content of compacted soil-stabilizer mixtures, respectively. The CBR values were greatly enhanced to approximately 185% for a soaked sample and 140% for an unsoaked sample when treated with 25% stabilizer. The 28 days unconfined compressive strength of non-stabilizer specimen was 38 ksc and dramatically increased to 110 ksc by the addition of 25% stabilizer. The results of the unsoaked CBR test, soaked CBR test and unconfined compression test demonstrated the potential of the CMDW1000 and SBAW mixture for use as a loess stabilizer.

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