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점토-골재 벽돌 경화에 있어 효소 사용의 효과 Effect of Enzyme Stabilization on Hardening of Clay-rock Brick

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This study investigates enzyme stabilization in clay-rock bricks through mechanical tests and image processing. Appropriate soil mixtures were designed using clay/crushed rock with ratios of 70/30, 60/40, 50/50, 40/60, and 30/70 by weight to verify the strength of the enzyme brick and soil compaction. The maximum compressive and flexural strengths in the 60/40 ratio mixture were found to be 5MPa and 1.25MPa, respectively; however, the maximum dry unit weight of 2.073g/cm³ was found in the 50/50 clay/gravel ratio mixture. Generally, the strength of the enzyme brick was improved by 27%. The paper concludes that in order to achieve optimal strength, soils should be mixed with the 60/40 clay/gravel ratio, which provides an adequate strength, while 50/50 ratio should be used for achieving more compaction. The SEM-EDX observation and Matlab image processing verified how the bond structure appeared after enzyme stabilization. It was found that enzymes created bond with the clay soil and the crushed rock for rendering strength and stability.

키워드: 효소 점토벽돌, 압축강도, SEM-EDX 관찰, 점토 **Keywords**: Enzyme brick, Strength, SEM-EDX observation, Clay

1. INTRODUCTION

Rocks and bricks have been used for buildings, monuments, and tombstones based on their availability or beauty(Franke 2009). As urbanization increases, demand for bricks gradually rises. In developing countries, urbanization has led to replacement of the old structures with new structures, resulting in the production of demolition wastes in a large scale(Rekha and Potharaju 2015). Brick is one of the important materials for the construction industry(Raut et al. 2011). Clay as a building material plays a major role in improving the environmental efficiency and sustainability of buildings and contributes to the economic prosperity and infrastructural development worldwide (Oti and Kinuthia 2012). Clay is necessary to achieve sufficient strength in a block to function without excessive breakage

(Montgomery 1998). Clay bricks and panels are commonly used for external cladding application as clay has shown good resistance to aging and to external actions(D'Orazio et al. 2014). Brick used for masonry is a low cost material with good sound and heat insulation properties(Kaushik et al. 2007). Construction house built from un burnt clay has been found to be significantly cheaper than fire clay bricks(Kasperiūnaitė et al. 2009). The high-temperature consumes a significant amount of energy and releases greenhouse gases(Zhang 2013). Soil stabilization is an accurate way to improve the soil strength(Yusuf et al. 2012). Soil stabilization is an accurate way to improve the soil strength(Yusuf et al. 2012). From the Eko-soil manual, it can be used to increase soil density and to lower permeability and compaction efforts. The enzymes can be absorbed by colloids, enabling them to be transported through the soil electrolyte media(Velasquez et al. 2005).

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Several studies have reported that bricks have been designed to become homogeneous, harder, and stronger(Phonphuak and Thiansem 2012). As masonry is constructed using locally available materials, its behavior in terms of strength and stiffness varies from region to region(Basha and Kaushik 2014). Compressive and flexural strengths of masonry unit increase over time(Costigan and Pav a 2009). Compaction control for construction is mostly based on the requirement that the contractor meets a certain maximum dry density obtained from the standard test procedure. As a result, laboratory specimens compacted to the minimum specification are used to assist in making decisions regarding the adequacy of the specifications required for the field. To characterize the soil compaction, physical parameters such as the bulk density, porosity, soil strength, and reduction of aeration have been used(Nawaz et al. 2013). There are a few internationally recognized standards for compressed earth block sizes. However, in general, block sizes vary widely(Morel et al. 2007). However, there is an approximate mathematic relationship of length, width and height of 6:3:2, which allows for bonding in any direction. One of the roles of the enzyme is minimizing the double layer absorption by increasing the electrolyte concentration or cation exchange with higher valence(Rathore and Pinky 2012). Cross-linking and copolymerization are some of the methods for immobilization of enzymes(Sayler et al. 1992). Enzymes are surfactants(ionic surface active agents) that change the hydrophilic nature of clay to hydrophobic(Lim et al. 2012). The active site of enzyme plays a significant role in orientation of the substrate and then promotes strain development such that some of the bonds are easier to break, thus allowing the formation of the new bonds(Rathore and Pinky 2012). Ettringite is a white needle—like crystal mineral found in cement and lime treated soils, occurring because of the reaction between solid materials containing calcium, aluminum, and iron, especially in the presence of sulfate enriched environment(Rajasekaran 2005). The microstructure of an enzyme clay brick reveals that the enzyme acts as a carrier or a cross—link and enables the creation of a bond. The cross—linking and bonding ability of the enzyme in a clay brick help bind the soil particles together, thus imparting strength to the soil mixture and increasing its dimensional stability(Mitikie et al. 2017).

The main objective of this study is to investigate the effect of the enzymes on clay-rock brick strength and compaction effect with different proportions of the clay/rock mix.

2. MATERIALS AND METHODS

2.1 Materials

The materials used in this study were clay, rock, and enzyme. The clay contained bacteria capable of reacting with the enzyme. The rock used in this study was of the igneous type and provided the strength and stability of the brick, it was crushed to less than 10mm in size. The enzymes used in this study were obtained from the Eco Enviro Services(Aust) Pty Ltd. (Australia).

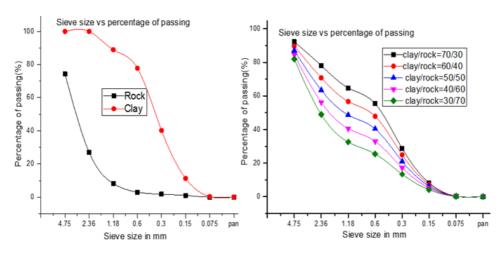


Fig. 1. Grain size distribution and sieve analysis result

Table 1. Characterization of the grain size distribution of the soil mixture

	Analysis		Clay/rock	Clay/rock	Clay/rock	Clay/rock	Clay/rock
Sieve	Rock	Clay	70/30	60/40	50/50	40/60	30/70
D60	4.03	0.46	0.89	1.46	2.08	2.68	3.16
D30	2.51	0.25	0.31	0.37	0.44	0.55	0.98
D10	1.29	0.14	0.16	0.17	0.19	0.21	0.25
Cu	3.12	3.28	5.56	8.58	10.94	12.76	12.64
Сс	1.21	0.97	0.67	0.55	0.49	0.54	1.22
Finesse modulus	4.85	1.82	2.73	3.03	3.33	3.63	3.94

where CU is the uniformity coefficient, CC is the Coefficient of Curvature, and D10, D30, and D60 are the diameters of which 10%, 30%, and 60% of the sample is finer respectively.

Table 2. Chemistry of the clay soils used for enzyme brick from the SEM-EDX

Chemistry	SiO ₂	TiO ₂	Al ₂ O ₃	Cr ₂ O ₃	Fe ₂ O ₃	SrO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	SO ₃	LOI
Rock	56.4	0.9	16.8	0.02	5.4	7.3	2.23	4.4	2.01	4.1	0.2	0.2	0.04
Clay	56.5	-	25.6	-	5.9	-	1.2	0.7	2.6	5.3	-	1.3	0.9

known under the trade name of Eko-Soil. The number of companies supplying the enzyme products for soil stabilization is limited. There are several enzyme products, such as permaenzyme, Tera enzyme etc. However, the Eko-soil is widely used in the Asia-Pacific region. As the product's name implies, it is fermented for engineering the soil used for the road surfaces and brick houses. It is a multiple enzyme-based product fermented from organic materials with non-hazardous, non-toxic, and biodegradable properties. Enzymes can break the weak oxides from the clay soil and can combine with other higher valence elements to form strong oxides that can make the block structures stable and strong. The active site of the enzyme can break Na₂O, which is weak in the clay structure; then ${\rm Ca_2}^+$ and ${\rm Fe_3}^+$ can replace Na⁺ to form CaO or ${\rm Fe_2O_3}$, which has higher bondage structure.

To find the exact value of the fineness modulus for coarse aggregates, mechanical shaker and sieve analyzers were used. From Table 1, the fineness modulus of the rock is seen to be 4.85, which means the average size of a particle of a given coarse aggregate sample lies between the 4th(0.6mm) sieve and the 5th(1,18mm) sieve.

2.2 Enzyme brick production process

The appropriate soil mixture was designed first, based on

the clay/gravel ratio. The clay soil was crushed into smaller pieces using a disk mill 120 device to obtain appropriate grain-size distributions to ensure sufficient reactions with the enzymes. Each ingredient was then sieved to remove the particles with odd sizes. The clay must be grinded to help it undergo an ionic reaction with the enzyme. The second step was the stabilization and mix preparation: The soil had to be mixed with the appropriate proportion of water content that was same as the enzyme/water ratio. Water content depended on the finesse of the materials used. The enzyme stabilizer was thoroughly mixed with water and then added to the soil. Then, the soil was mixed with the selected proportion of the enzyme/water mixture to maximize its performance. Compaction: The mixture then had to be compacted to ensure strength and quality using the hydraulic press machine. A constant compaction force was provided by the hydraulic press machine and then the block was carefully removed from the mold. Finally, the bricks were cured in the room(not heated or oven dry needed, because heat destroyed enzyme and clay bacteria). The enzyme brick was cured for 28 days for better strength.

2.3 Method and tests

Tests were performed to measure the compressive strength, flexural strength, and the compaction of enzyme rock fill with

Table 3. Material mix proportion and require
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Clay/rock ratio	70/30	60/40	50/50	40/60	30/70
Enzyme/water ratio	1/100	1/300	1/500	without enzyme	
Test required	Compressive stre	ngth	Flexural strength		Compaction
Number of samples	8×4×5=	160	8×4×5=	3×4×5=60	

(Note:8 is the number of samples, 4 is for the enzyme/water ratio and 5 is for the clay/rock)

clay at different enzyme/water ratios. Though the SEM observations were used to observe the structure, the focus of this study was primarily on mechanical stabilization.

Any oven drying was forbidden for natural clay and rock. To control the drying condition, the room was maintained at 58% average humidity and 18°C temperature. 28 days curing time was found sufficient for the enzyme brick. As the size of the brick was 190mm x 90mm x 60mm, it satisfied the standards from the mathematical approximation of 6:3:2(length: width: height). The universal testing machine was used for testing the compressive strength of the bricks. To test the specimens the bricks were placed in the compression testing machine. For each tested specimen, the failure load was noted and the compressive strength was estimated by the following formula:

$$Compressive strength = \frac{(1000).(P)}{A} \tag{1}$$

Where the compressive strength is in MPa, P=failure load(KN) and A= net cross-sectional area(mm²). The flexural strength test was conducted using designation ASTM C42(ASTM 1999). Flexural strength represents the highest stress experienced within the material at its moment of yield and is given by:

$$Flexural strength = \frac{(P).(L)}{(w).(b^2)} \tag{2}$$

Where P is the maximum load(N), L the distance between the supports(120mm), w the width(80mm), and b is the height of the specimen(50mm). The compaction of the soil into the mold at various moisture contents was based on the standard compaction test AS 1289(Australian standards). The soil was compacted into a mold in three equal layers, with each layer

receiving the constant blows of a hammer weight equivalent to 2745KN-m/m³ compactive energy. This value is nearly the same as 2700KN-m/m³ from the ASTM D698(Standard 2007). The rectangular compaction mold of 125cm³ and 1.555kg of the rectangular hammer fell down 300mm for 25times on the material cast.

3. RESULTS AND DISCUSSION

3.1 Compressive and flexural strength

The compressive strength, flexural strength, and compaction are mechanical properties of enzyme stabilization of the clayrock application. The compressive strength of brick results is accepted by the Indian standards, which have a minimum value 3,5MPa, and the British standards with the minimum value of 5MPa(Lynch 1994; Standard 1992). The flexural strength of the enzyme brick is positively correlated to the compressive strength as per the results of this study. The important property in the structural design of brick is the compressive strength.

Bricks mixed with 60/40 clay/rock with higher proportions of the enzyme/water tend to exhibit higher strengths of 5Mpa

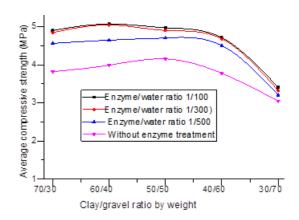


Fig. 2. Average compressive strength of the enzyme brick

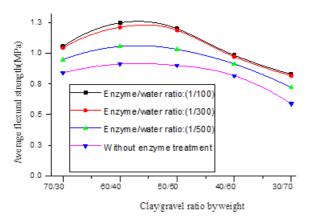


Fig. 3. Average flexural strength of the enzyme brick

(Fig. 2). The bricks without enzyme have less compressive strength caused by the weakening of micro structural bonds The strength of the enzyme brick is improved by 27% over the non-enzyme brick. Increasing the enzyme amount above this limit has no effect on the result. Therefore, the enzyme to water ratios of 1/100 & 1/300 result in the same strength value. The compressive strength value of enzyme brick depends on the clay/gravel as well as enzyme/water ratio. The gravel is very important for strength and stability, but an increase in more than 50% has resulted in a lower strength of brick. Bricks with 30/70 clay/rock ratio by weight continued to show decreasing compressive and flexural strength, the main reason being the improper mix with enzyme water applied to more gravel with less clay. The flexural strength shows its ability to resist failure in bending and its value was 1,25MPa with the clay/rock ratio of 60/40. Generally, it can be concluded that the 60/40 clay/gravel mixture may be the more promising mixture of the raw materials for maximization of enzyme stabilization in compressive strength and flexural strength

3.2 Compaction, air-voids and maximum dry unit weight

Compaction is a function of water content. Water added to the soil during compaction acts as a softening agent on the soil particles. Comparison was made among samples with different clay/rock ratios with different enzyme combinations. The maximum dry unit weight was controlled by the water

Table 4. Maximum dry unit weight of different clay/gravel ratios in compaction

Enzyme/water ratio	1/100	1/300	1/500	without enzyme
Clay/gravel ratio	N	Maximum dry	density(g/cm ³)
70/30	2.00	1.980	1.980	1.809
60/40	2.00	2.00	1.980	1.795
50/50	2.073	2.070	2.003	1.813
40/60	2.037	2.036	1.980	1.808
30/70	2.038	2.038	1.996	1.804

Table 5. Optimum moisture content for maximum dry weight in different clay/gravel ratio

Clay/gravel ratio	Optimum moisture content
70/30	12%
60/40	11%
50/50	10%
40/60	8%
30/70	6%

content and the air voids content.

Curves for different air voids contents can be added to the maximum dry density/water content plotted. The grain size of the material in the mix increases from the 70/30 to 30/70 clay/rock combination by weight. The compaction curve, maximum dry density, and optimum moisture content are highly dependent on the size of particles in soil mix or the clay/rock ratio.

The optimum water content can be seen to decrease from 12% to 6% as the clay/rock ratio changes from 70/30 to 30/70. The dry unit weight corresponding to the maximum point on the moisture content/dry density curve is the acceptable value.

There is an overall increase in the dry unit weights in all the samples when the whole samples are compacted. The water content used for compaction is highly dependent on the clay/rock ratio rather than the enzyme/water ratio. Generally, the compaction values increase as the gravel increases to 50% with clay and then tend to decline. Compaction can be applied to improve the properties of an existing soil sample by filling the voids and to decrease permeability. In the enzyme/ water mix, enzyme lowers the surface tension of water by breaking the double water molecules in their active site. The

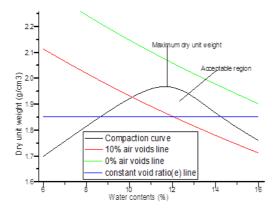


Fig. 4. Compaction curve with 70/30 clay/gravel

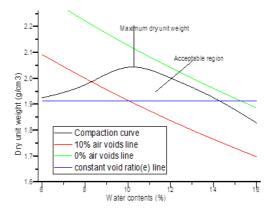


Fig. 5. Compaction curve with 60/40 clay/gravel

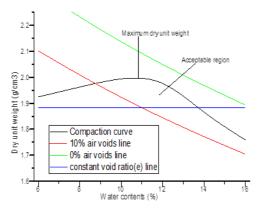


Fig. 6. Compaction curve with 50/50 clay/gravel

hydrated bond will then be strong if a double layer of hydrogen bond is removed using an enzyme. Enzyme reacts with the adsorbed water layer of clay particles and reduces the thickness around the soil particles because of the clay particles' orientation with the enzyme. The enzyme has lubricant effect that allows the soil to compact highly with a constant

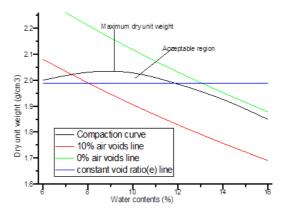


Fig. 7. Compaction curve with 40/60 clay/gravel

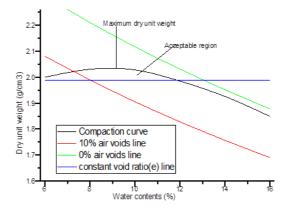


Fig. 8. Compaction curve with 30/70 clay/gravel

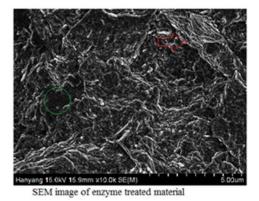


Fig. 9. Enzyme treated brick with 60/40 clay/gravel ratio using Matlab image processing

compaction effort, thereby improving the strength.

Compaction curve with given clay/gravel ratio at 1/100 enzyme/water ratio (Acceptable region, zero air void line, compaction curve, maximum dry unit weight and optimum moisture content for each clay/rock ratio are estimated.

For the SEM-EDS microscopic imaging and chemical composition studies, the sample was selected from 60/40 clay/gravel with the enzyme/water ratio of 1/100. The scanning electron microscope (SEM) provided more information about the sample including the external morphology(texture), chemical composition, and crystalline structure of the materials present in the sample. SEM techniques with a magnification of 10,000 × could show the enzyme reacting with the clay particles and the flake-like particles as well as a weak structure of ettringite. From the SEM image, it can be seen that enzyme reacts with clay and tries to form structures like ettringite to fill the voids in the brick. The clay soil is rich in silicon and aluminum oxide but has lower amount of calcium oxides. Therefore, the SEM image could not show delayed ettringite and calcium silicate gel. The image shows flake or cross-linking structures.

The difference between the enzyme stabilization and the

non-enzyme stabilization can be seen in Fig. However, from Fig. 6, the ettringite structure seems to be in the early stage and is different from ettringite in concrete. Concrete has high calcium from cement and sulfate due to gypsum, therefore very long needle-like structure will be observed. Based on the SEM-EDX observation, the percentage of elements can be estimated. SiO₂ and Al₂O₃ are the most dominant chemical oxides in the structure. The elemental composition of enzyme brick(wt, %) was determined by energy dispersive spectroscopy (EDS). The SEM observation is mostly expressed qualitatively than quantitatively. SEM with a couple of EDX can show the oxide composition in addition to the structural image. The chemistry of enzyme clay-rock is estimated,

However, the SEM image can show internal structure using Matlab image processing. The higher valancy oxides of silicon, aluminum and iron have better bondage structure than the

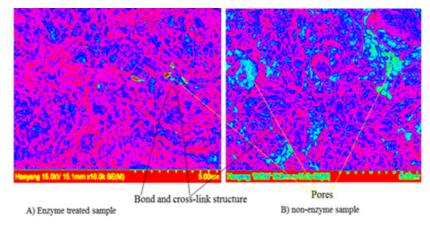


Fig. 10. Cross-link and bond like structure between the enzyme and the clay bacteria in brick structure

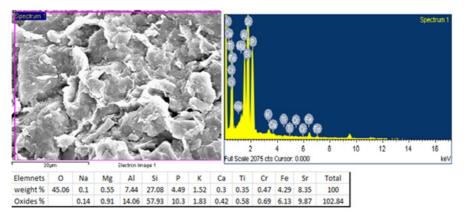


Fig. 11. SEM-EDX structure of enzyme brick with 60/40 clay/rock ratio

oxides with less valancy, like sodium, potassium or magnesium. The SEM-EDX used 20 m area to observe the structure and chemistry composition of the sample. The main property of enzyme is that it is capable of an ionic reaction with the clay soil. SEM micrographs of enzyme clay brick samples at high magnification could help observe several structures related to the reaction between the enzyme and the clay soil.

The clay soil acts as a substrate with the active site of the enzyme to form a good bond. This action causes hydrated clay particles to be pressed to fill the voids throughout the soil, resulting in tight and dense structure

4. CONCLUSION

This study aimed to investigate enzyme stabilization in clay-rock soil. Bricks were designed having clay/rock ratios of 70/30, 60/40, 50/50, 40/60, and 30/70 by weight with enzyme/water ratio of 1/100, 1/300, 1/500 and without enzyme stabilization. Compressive strength, flexural strength, and compaction of soil were tested. The particles size highly affected the strength of the enzyme brick. Enzyme reacted with the clay soil to improve the strength of brick. 5MPa compressive strength was obtained in this study and this value satisfies some standards, such as the Indian standards with the minimum value 3 5MPa and the British standards with the minimum value of 5MPa. The maximum compressive and flexural strengths were found for the 60/40 ratio; however, the maximum dry unit weight was found for the 50/50 clay to gravel ratio. Based on these studies, the paper concludes that in order to obtain better strength, soils should be mixed in 60/40 clay/rock ratio; however, the clay/gravel ratio of 50/50 should be used to achieve more compaction with enzyme/water, Finally, SEM observation taken from the 60/40 sample shows the structural image and identifies the chemical oxides of enzyme brick. The bond like structure and early stage of ettringite can be observed after the Matlab image processing.

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점토-골재 벽돌 경화에 있어 효소 사용의 효과

본 연구는 점토와 골재로 구성된 벽돌을 경화하는데 있어 효소 첨가제의 효과를 재료시험과 이미지 프로세싱을 통해 분석하였다. 벽돌의 강도와 밀도 시험에 사용한 벽돌 샘플의 점토/골재 중량비는 70/30, 60/40, 50/50, 40/60, 30/70이였으며, 최대 압축강도와 휨강도는 각각 5MPa와 1,25MPa으로 중량비 60/40에서 발현되었다. 또한 최대 건조단위 중량은 2,073g/cm³으로 중량비 50/50에서 발현되었다. 시험 결과 전반적으로 벽돌의 강도는 효소를 첨가함으로 약 27% 향상되었다. 강도 향상을 위해서는 점토/골재 중량비 60/40, 밀도 향상을 위해서는 점토/골재 중량비 50/50에 효소를 첨가하여 벽돌을 경화하는 것이 효과적인 것으로 확인되었다. 효소 첨가 시 점토-골재 벽돌의 결합구조가 더 치밀해짐을 SEM-EDX 분석과 Matlab을 이용한 이미지 프로세싱을 통해 확인하였으며, 이를 통해 효소가 점토와 골재 간 결합구조를 형성하여 벽돌의 강도와 밀도를 향상시키는 효과가 있는 것으로 판단된다.