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ORIGINAL

Microstructural Observations on the Durability of Stabilized Rammed Earth

Observaciones microestructurales sobre la durabilidad de la tierra apisonada estabilizada

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ABSTRACT

This study provides a qualitative microstructural investigation of fly ash geopolymers' role in rammed earth's durability against water ingress and contact erosion. A series of SEM (scanning electron microscopy) images were captured on geopolymer-stabilized as well as unstabilized rammed earth samples. These samples were fabricated from predefined amounts of sand and fine materials together with fly ash geopolymers in the laboratory by static compaction to 25 MPa inside rigid molds. Two standardized durability tests were performed, namely, the dip test and the spray (also known as erosion) test. The results of these tests reflected excellent durability properties (practically zero erosion) of the stabilized material. The microscopic investigation provided an insight into the reason behind this improvement, where geopolymer networks inhabited the macro and micro pores and served as a cementing agent interconnecting the earthen materials' grains. On the contrary, grains of the unstabilized material were weakly bonded by the clay component of the mixture, as observed with the SEM images; therefore, they were more susceptible to erosion by water.

keywords: Rammed Earth; Microstructure; Durability; Geopolymer.

RESUMEN

Este estudio proporciona una investigación microestructural cualitativa del papel de los geopolímeros de cenizas volantes en la durabilidad de la tierra apisonada frente a la entrada de agua y la erosión por contacto. Se tomaron una serie de imágenes SEM (microscopía electrónica de barrido) de muestras de tierra apisonada estabilizadas con geopolímeros y de muestras no estabilizadas. Estas muestras se fabricaron a partir de cantidades predefinidas de arena y materiales finos junto con geopolímeros de cenizas volantes en el laboratorio mediante compactación estática a 25 MPa dentro de moldes rígidos. Se realizaron dos pruebas de durabilidad estandarizadas, a saber, la prueba de inmersión y la prueba de pulverización (también conocida como prueba de erosión). Los resultados de estos ensayos reflejaron las excelentes propiedades de durabilidad (erosión prácticamente nula) del material estabilizado. La investigación microscópica proporcionó una visión de la razón detrás de esta mejora, donde las redes de geopolímeros habitaban los macro y microporos y servían como agente cementante interconectando los granos de los materiales de tierra. Por el contrario, los granos del material no estabilizado estaban débilmente unidos por el componente arcilloso de la mezcla, como se observó con las imágenes de SEM; por lo tanto, eran más susceptibles a la erosión por el agua.

Palabras Clave: Tierra Apisonada; Microestructura; Durabilidad; Geopolímero.

INTRODUCTION

Rammed earth construction is an ancient architectural method that has been extensively used for thousands of years and continues to be widely practiced today. It involves compacting soil, clay, and gravel to form solid walls, making them suitable for construction. This method of building offers environmental sustainability through its use of locally available materials with minimal energy consumption and its straightforward construction process.[1] Extensive research conducted on the traditional use of earth-based materials has led to the development of design principles for modern rammed-earth constructions. These materials have been used for thousands of years in several countries, for example, in Mesopotamia (Iraq in the present) as early as 7000 BC,[2] where the Ziggurat of Ur was built using the mud-brick technique. The Chinese used rammed earth as parts of the Great Wall of China, which still stands today.[3]

These materials align well with environmental guidelines, particularly in arid climates characterized by scorching summers and chilly winters. Earth-based constructions offer numerous advantages regarding their sustainability and resilience that are beneficial to the environment; earth materials reduce heating and cooling energy requirements by their exceptional insulating and heat-storing properties.[4] By their mechanical robustness and flexibility, these materials afford a durable, long-lasting framework with intriguing acoustic and hygrometric characteristics.

Despite the numerous benefits of this technique, it is not widely practiced in our current time. One reason beyond that is the lack of durability of the raw materials, specifically against water. These materials disintegrate with water admission by, for example, absorption from the surrounding environment, sudden immersion, or corrosion caused by rainfall.[1] Therefore, several tests have been devised to evaluate the resistance of these materials to deterioration by water admission, such as dip test, Geelong drip test, and spray test.

The primary concern for designers and consumers is the long-term durability of earthy materials.[5] Several studies suggested that the utilization of cement greatly enhances the resilience of rammed earth against erosion caused by water. Arrigoni et al.[6] conducted a study to measure the deterioration caused by spraying water and the loss of mass due to wire brushing on unstabilized rammed and stabilized rammed earth mixtures. The mixes consisted of 5 % cement + 5 % fly ash and 6 % calcium carbide residue + 25 % fly ash. The researchers observed that these mixtures successfully passed the tests and achieved adequate strengths for construction, as stated in [7]. Narloch and Woyciechowski [8] conducted erosion (by water) tests on unstabilized rammed earth and 6 % and 9 % cement-stabilized rammed earth, following the guidelines of New Zealand Standard NZS 4298.[9] The results showed that none of the cement stabilized rammed earth specimens exhibited any form of surface deterioration, whereas all the unstabilized rammed specimens developed significant cavities shortly after exposure to water. This led to the conclusion that unstabilized rammed earth is not suitable for use in a humid continental climate due to its lack of durability. Nevertheless, several studies on the long term durability (over 20 years) of rammed earth against water, implied that external protection is still needed for cement (or lime) stabilized rammed earth.[10,11]

Numerous studies have been conducted utilizing scanning electron microscopy (SEM) methodologies to shed light on the microstructural characteristics of soils treated with geopolymers. Many of these studies monitored the development of cementitious growth caused by the geopolymer to elucidate the underlying mechanism responsible for the improved properties of the treated soils at the microscopic level. According to existing literature, it has been observed that the utilization of fly-ash-based geopolymer can enhance the density of treated soils, similar to the effects observed with lime or ordinary Portland cement treatment.[12] For instance, research by [13,14] revealed that the incorporation of fly-ash-based geopolymer enhanced the uniformity of clay fabric. This improvement manifested in a greater degree of interconnectivity among clay particles and a reduction of voids. The observed improvement can be primarily attributable to the formation of artificial cementation products and the subsequent establishment of links among soil particles during the curing process.[13,14] The aforementioned observation was also corroborated by Phummiphan et al.[15], who utilized scanning electron microscopy (SEM) to analyze marginal lateritic soil. They found the presence of etched holes on the surface of partially reacted fly ash particles within the treated soil. The formation of these holes was purportedly attributed to the process of leaching silica and alumina off the surface of the activated fly ash. According to previous research by [16], it is hypothesized that the presence of partially-reacted fly ash particles and cementitious products in the treated soil act as nucleation sites, facilitating the bonding of clay plates into clusters. This modification of the soil structure is expected to result in improved mechanical properties and responsiveness.

In this study, fly ash-based geopolymer was utilized to enhance the durability of one-dimensionally compressed earthen specimen. To analyze the impact of the additive material at the microscopic level, microstructural and XRF examinations were conducted.

Experimental Work and Procedures

This work is a part of in-progress efforts on rammed earth at the University of Anbar. The soil selection, specimen preparation, and curing were detailed in a preceding paper by the authors.[17] Two different soils were employed in the study, namely, Soil 1, consisting of high plasticity fine-grains, and Soil 2, consisting of poorly graded sand, see table 1.

Table 1. Classification of Soil 1 and Soil 2[17]

	Property	Soil 1	Soil 2
Graduation	Gravel (>4,75 mm, %)	0	0
	Sand (4,75-0,075 mm, %)	0	100
	Silt (0,075-0,005 mm, %)	50	0
	Clay (<0,005 mm, %)	50	
Plasticity	Liquid limit (%)	65	Non-plastic
	Plastic limit (%)	37	
	Plasticity index (%)	28	
	Soil class (USCS)	CH	SP

The Fly ash composition was analyzed by the X-ray Fluorescence (XRF) test, which was performed at the laboratory of the Department of Geology, University of Baghdad.

Method of Testing

The Immersion Test

This test replicates sudden immersion circumstances, such as occurrences of floods. The immersion test, commonly referred to as the dip test, is a method outlined in norm DIN 18945 [18] that assesses the durability of earthen materials by subjecting them to water immersion rather than evaluating their absorption from the surrounding environment. This test aims to simulate the sudden flooding or submersion of earthen materials in water, thus subjecting them to harsh conditions. The earthen samples are first weighed before undergoing testing and thereafter immersed in water for a duration of 10 minutes. The determination of mass loss is achieved by the process of filtering the leftover particles from water. Subsequently, the specimen is subjected to a drying process at a temperature of 40 °C for a duration of 24 hours. Following this, the specimen is exposed to the atmosphere, and its weight is measured. Material loss is calculated by dividing the mass of the filtered material by the original mass of the sample. Accordingly, the normative classifies earthen building units into three classes, as shown in table 2.

Table 2. Classification of earthen building units and walls based on the dip test results [18]

Class	Immersion test Mass loss (%)	Scope of application
Ia	≤ 5 %	Ia: Plastered, weather-exposed exterior masonry of exposed half-timbered walls
Ib	≤ 5 %	Ib: Consistently plastered exterior masonry exposed to weathering
II	≤ 15 %	II: Clad or otherwise constructively weather-protected exterior masonry, interior masonry
III	No requirement	III: Dry applications (e.g., ceiling fillings, stack walls)

The Spray Test

This test measures the contact erosion caused by rainfall. This test is usually performed according to the New Zealand standard NZS 4298 (1998).[19] In this test, the specimen's face is continuously sprayed with water for 1 hour under 50 kPa gauge pressure, as shown in figure 1. A flat-end rod of 10 mm diameter is used to frequently measure the depth of the deepest hole made by the water. This depth of erosion, which is usually measured in millimeters, is then rated on a 1 to 5 scale, where one corresponds to penetration of ≤ 20 mm, whereas five corresponds to penetration of ≥ 120 mm.

Preparation of Rammed Earth Specimens

Four geopolymer-soil mixtures were investigated. These mixtures contained 20 % or 25 % fly ash, activated with (2M) or (4M) NaOH solution of 98 % purity. In addition, a reference mixture containing; 15 % silt,15 % clay, and 70 % sand, find out the mix design in Table 3. It is worth mentioning that fly ash contents of up to 15 % were found to be insufficient to provide enough binding against deterioration by water immersion.

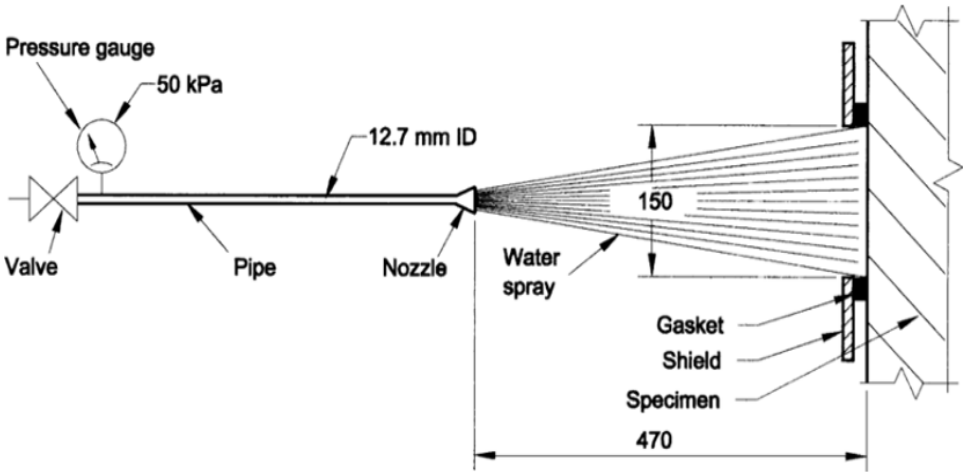

Figure 1. Spray test setup (NZS4298-1998)[19]

Table 3. Mix design of the work[17]						
	Mix	**(0:100)**	**(20:80)2M**	**(20:80)4M**	**(25:75)2M**	**(25:75)4M**
	FA (%)	0	20 %	20 %	25 %	25 %
	NaOH (M)	0	2	4	2	4
Soil 1	Silt (%)	15 %	12	12	11,25	11,25
	Clay (%)	15 %	12	12	11,25	11,25
Soil 2	Sand (%)	70 %	56	56	52,50	52,50

For the work presented in this paper, two types of specimens were produced by static compaction to 25 MPa inside stiff walled molds by using a high capacity loading machine (more details can be found in [17]. The compaction pressure of 25 MPa was selected based on a previous work by Hasan and Al-Sharrad[20]; who showed that at this pressure the earthen material possessed a suitable compressive strength, and thermal and moisture exchange with the atmosphere. Cylindrical specimens with 50 mm in diameter and 100 mm in height were prepared for the dip test, whereas prismatic specimens with 150*150*50 mm^3 were prepared for the spray test. The initial properties of these specimens are given in Table 4. For the mixtures with geopolymer, the mixing water content (optimum) was replaced by the NaOH solution of a given molarity. The compacted specimens were then cured in an oven at the constant temperature of 35 ^oC until the ages of 7, 30, or 60 days.

A curing temperature in the order of 60-70 ^oC is usually recommended for an ideal geopolymerization reaction.[21,22,23] In this work, the threshold temperature of 35 ^oC was selected to reduce energy consumption and construction cost. Actually, it can be provided naturally by the sun during the summer season. In addition, this moderate temperature is preferable to mitigate possible shrinkage of the earthen material.

Table 4. Composition and average unit weight values of the mixtures						
		Unstabilized specimens	**Fly ash geopolymer stabilized specimens**			
Cylindrical	Mixtures	(0:100)	(20:80)2M	(20:80)4M	(25:75)2M	(25:75)4M
D=50 mm	[Fly ash: Soil] Molarity					
H=100 mm	Water content [%]	7,6	0	0	0	0
	NaOH solution content [%]	0	10	10	10	10
	Dry unit weight [kN/m^3]	21,8	20,6	21,9	20,1	21,7
Prismatic	Water content [%]	7,6	0	0	0	0
150*150*50 mm^3	NaOH solution content [%]	0	10	10	10	10
	Dry unit weight [kN/m^3]	21,1	20,3	20,1	20,0	20,1

Microstructure of Rammed Earth

The microstructure was investigated by performing SEM on specimens prepared with two fly ash contents, i.e., (20 and 25) %, and two NaOH molarity values, i.e., (2M and 4M). In addition, SEM imaging was performed on a specimen containing the raw earthen material without the additive.

Durability Tests

The Immersion Test

This test was conducted in accordance with the norm DIN 18945 [5] test specimen with 50mm in diameter and 100 mm in height after 30 days of curing. The cylindrical specimens prepared from various mixtures were first clamped, lowered, and then submerged in water for 10 minutes at a depth of 5 cm, as shown in figure 2. At the end of the immersion process, the sample was carefully taken out of the water bath. The residue inside the glass container was subsequently filtrated and dried at a temperature of 40 °C, then left at an ambient temperature of 23 °C and 50 % relative humidity until its constant mass was achieved. This mass was taken when the difference between two successive weighing trials separated by 24 hours did not exceed 0,2 % of the lesser measured mass. The calculation of mass loss by immersion involved dividing the mass of the remains from three identical samples by the initial total mass of these specimens at the start of the test.

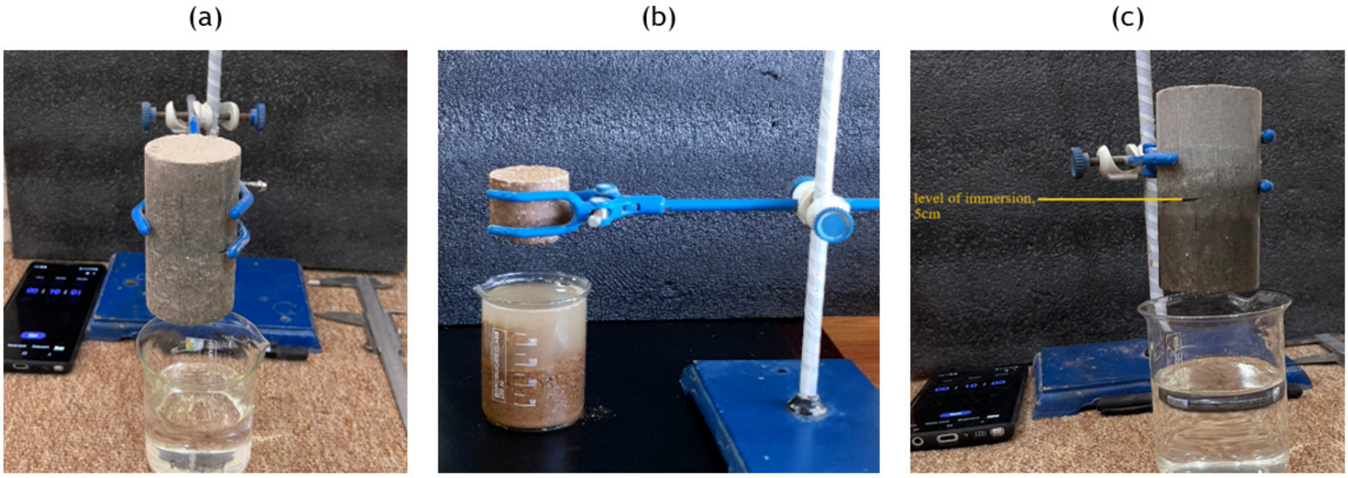

Figure 2. Dip test: (a) test setup (b) unstabilized specimen (failed) (c) stabilized specimen (passed)

The Spray Test

This test, also known as the erosion test, was performed by the New Zealand Normative NZS4298-1998 [19] on a test specimen with 150*150*50 mm^3 after 30 days of curing. The specimen's face was continuously sprayed with water for 1 hour under 50 kPa gauge pressure, as shown in figure 5.

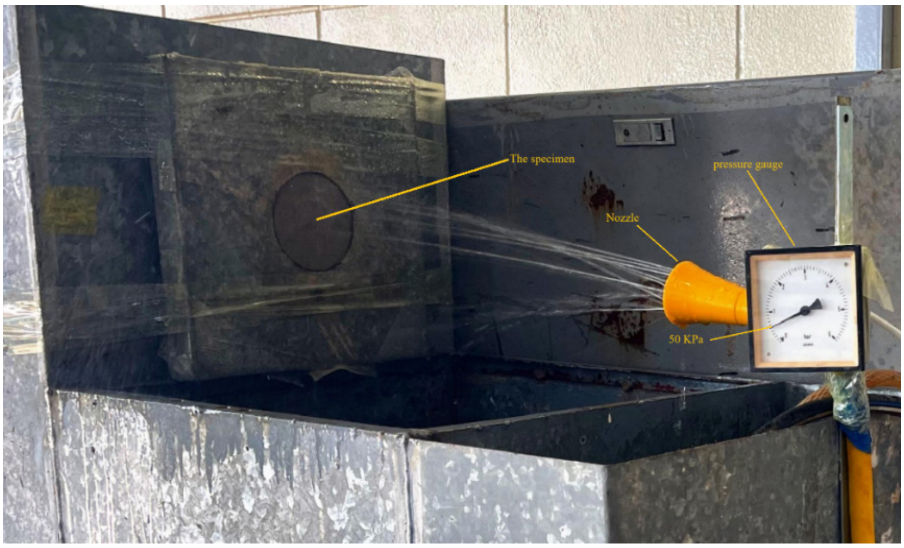

Figure 3. In-progress spray test

RESULTS AND DISCUSSION

Dip Test

The results of the dip test indicated that the unstabilized sample, marked as (0:100), exhibited a mass loss of 70 %. As anticipated, the unstabilized samples experienced extensive failure as a result of water softening of

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the fine component of the material, which serves as a binding agent. In contrast, the stabilized samples from other mixtures containing fly ash geopolymer remained largely intact, exhibiting negligible mass loss of less than 0,2 % that fell well within the limits specified by DIN 18945.[18] Thus, it is evident that fly ash geopolymer serves as an effective binder, which significantly enhances the material's resistance to water infiltration.

Spray Test

The results of the spray test are shown in figure 4 and table 5. The results show that by the end of the test, the unstabilized earthen sample expressed a considerable depth of erosion (about 30 mm). This is expected as the contact erosion resistance of the raw material is relatively low so the deterioration commenced immediately after the test started. On the contrary, the results of geopolymer-stabilized earthen specimens indicated more or less no penetration and zero erodibility. This once again confirms that fly ash geopolymer is a powerful binder to protect rammed earth against erosion by water.

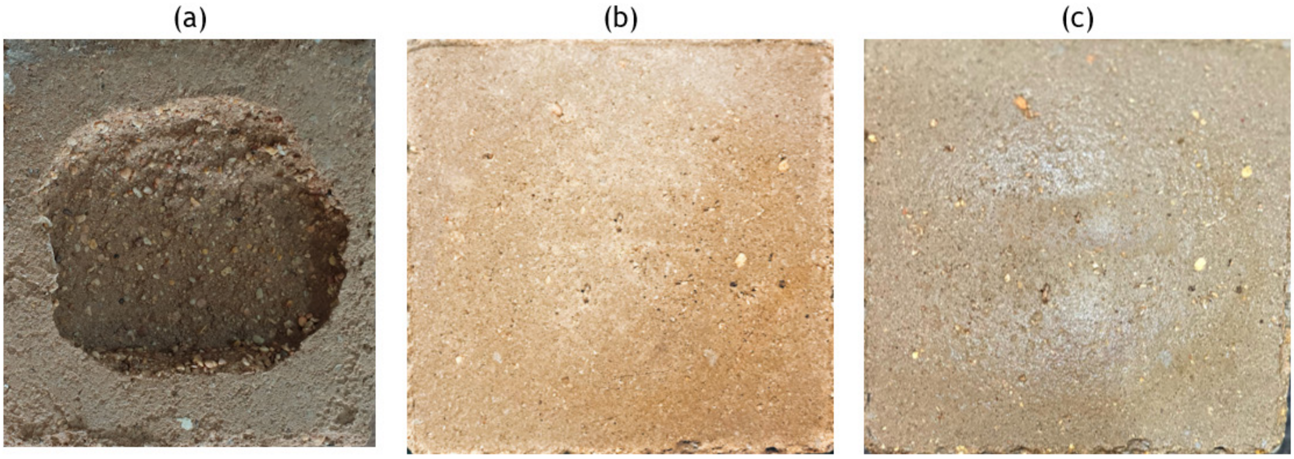

Figure 4. Typical spray test results on a) unstabilized specimen after the test, (b) stabilized specimen before the test, c) stabilized specimen after the test

Table 5. Results of spray test on various geopolymer-soil mixtures

Mixtures (Fly ash: Soil) Molarity	Penetration, D [mm]	Criteria	Erodibility index (NZS4298-1998)
(0:100)	30	0 ≤ D < 20	1
(20:80)2M	≈ 0,0	20 ≤ D < 50	2
(25:75)2M		50 ≤ D < 90	3
(20:80)4M		90 ≤ D < 120	4
(25:75)4M		D ≥ 120	5 (Fail)

X-Ray Fluorescence (XRF)

The main oxides comprising the fly ash used in this study are displayed in Table 6. According to the ASTM C618-12,[24] the fly ash is classified as Class F. The chemical analysis indicates that silica represents a significant proportion of fly ash composition, with aluminum oxide being the next major component. The inclusion of these constituents is essential for the beginning of pozzolanic reactions and the progression of geopolymerization, thereby improving materials' mechanical characteristics.[25]

Table 6. Chemical composition of the fly ash

Oxide Composition	SiO_2	AL_2O_3	CaO	Fe_2O_3	MgO	Na_2O	K_2O	SO_3	MnO	Others
Concentration	49,11	29,98	9,497	4,129	1,314	0,637	0,7057	1,064	0,07 %	3,2 %

Microstructure of the Rammed Earth

Microstructure of the Raw Rammed Earth: figure 5 shows two microscopic images taken at 70x and 4000x magnifications on a raw earth material compacted to 25 MPa. Inspection of figure 5a shows several sand particles partly coated with fine material acting as a cementation agent. Despite the fact that rammed earth is usually made with proportions of sand in excess of 50 %, fine materials are indispensable to improve its strength, moisture exchange, and thermal responses. Aggregations of silt and clay particles are noticeable (figure 5b), essentially forming during the specimen preparation stage. Inspection of the image indicates that both micro and macro pores coexisted (dual-porosity) in the fabric, a characteristic of compacted materials.

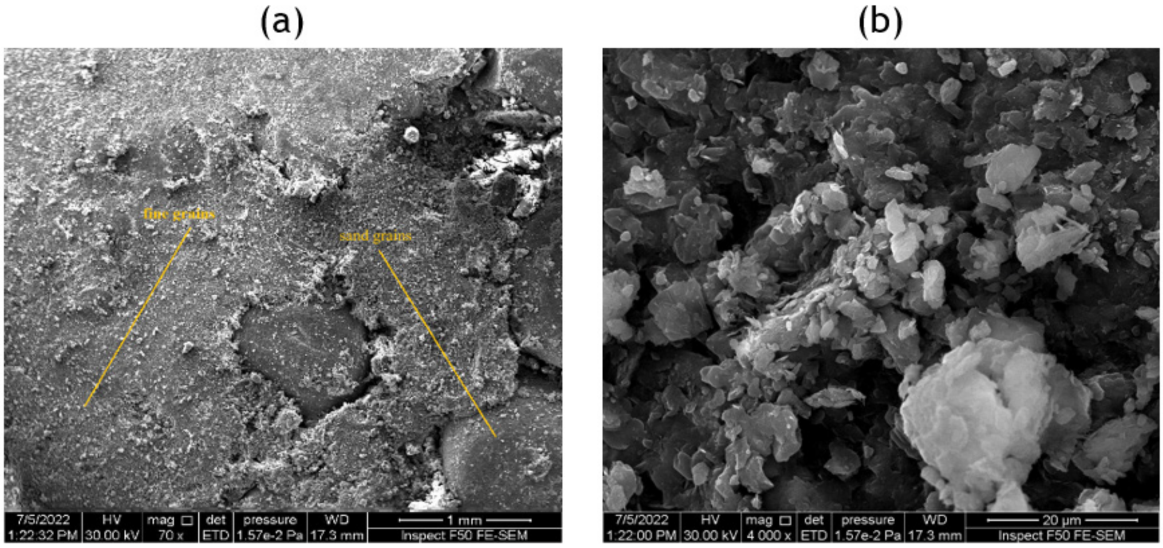

Figure 5. SEM image of a raw rammed earth a) 70x magnification b) 4000x magnification

Effect of Geopolymer: figure 6 shows an SEM image of a specimen representing the mix (25:75)2M. Compared to the specimen fabricated from raw material (figure 5b), the stabilized specimen in figure 6 contains additional materials. These materials are unreacted fly ash, which appears as spheres of different sizes, and geopolymer, which partly coats sand, fly ash, and fine aggregates. Despite the fact that the sodium hydroxide, as a liquid, represents only 10 % by weight of the solid constituents, the produced geopolymer provided a reasonable amount of cementation for the solid constituents of the stabilized rammed earth.

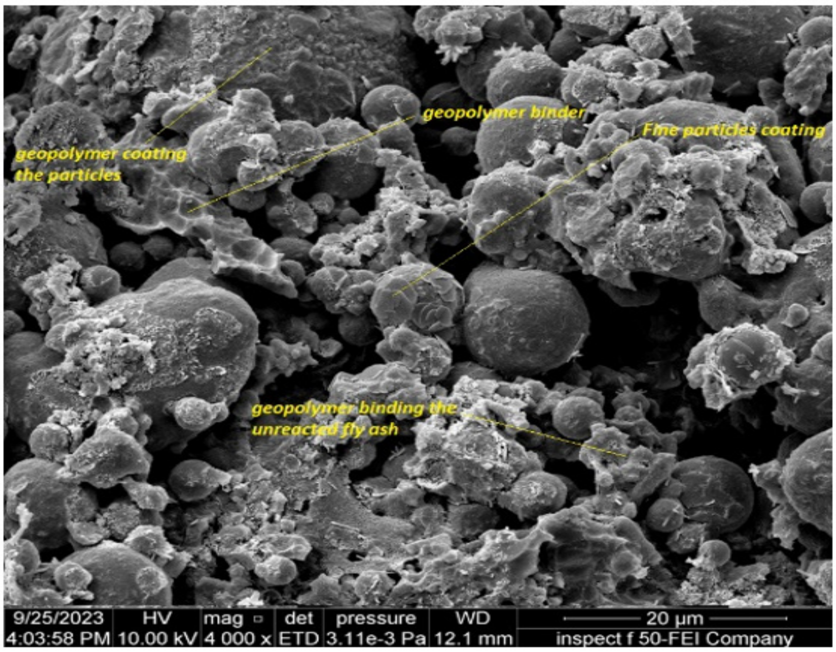

Figure 6. SEM image of a stabilized rammed earth- Mix (25:75)2M at 4000x magnification

Figure 7 compares two microscopic images from the mixtures (20:80)4M and (25:75)4M, where both mixtures were prepared with the same molarity of sodium hydroxide (i.e., 4M) but with 20 % and 25 % fly ash content, respectively. The mixture with higher fly ash content tended to show more inactivated fly ash particles. This suggests, on the one hand, that the amount of the NaOH solution was not sufficient to dissolve the fly ash. However, on the other hand, fly ash as a by-product does not usually comprise 100 % dissoluble particles. The latter statement is evidenced by the mineralogical analysis of the fly ash, shown in Table 6. As mentioned previously, fly ash contents of up to 15 % were found insufficient to provide enough binding against deterioration by water ingress. Therefore, fly ash contents of about 20 % to 25 % seem optimal from practical and economical perspectives.

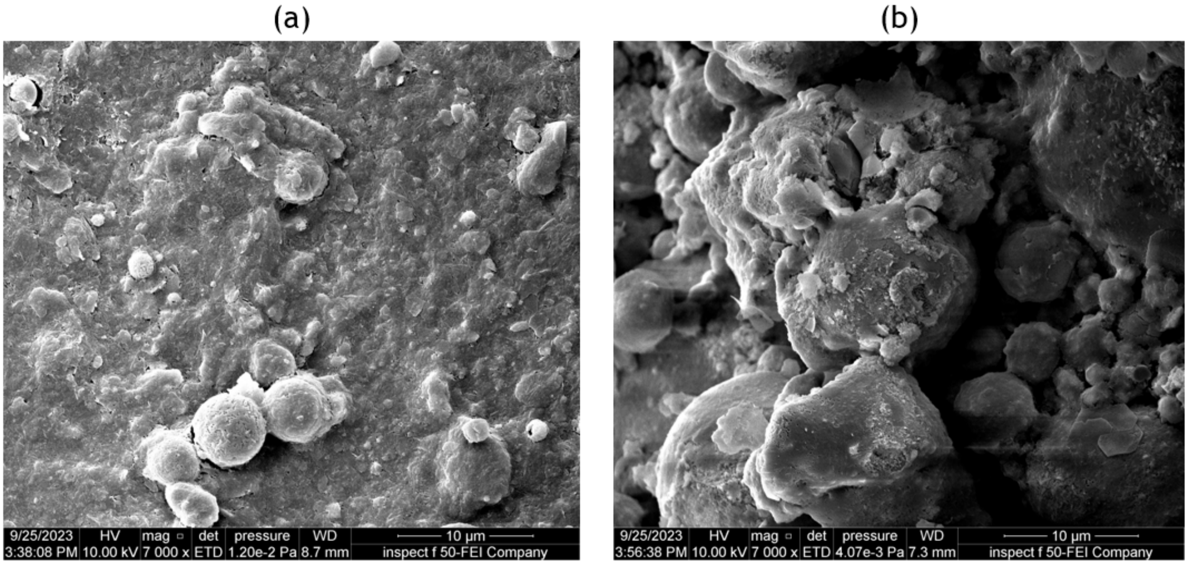

Figure 7. Effect of fly ash content of a mix with 4M NaOH and a) 20 % fly ash and b) 25 % fly ash

At the level of micropores (figure 8), geopolymer appears as a continuous network inside the pores and between the clay particles. This further confirms the effectiveness of the geopolymer of binder to rammed earth constituents. The addition of geopolymer enhances the intergranular interactions at both the macroscopic and microscopic levels. One of the consequences of this is that materials durability against water ingress has significantly improved.

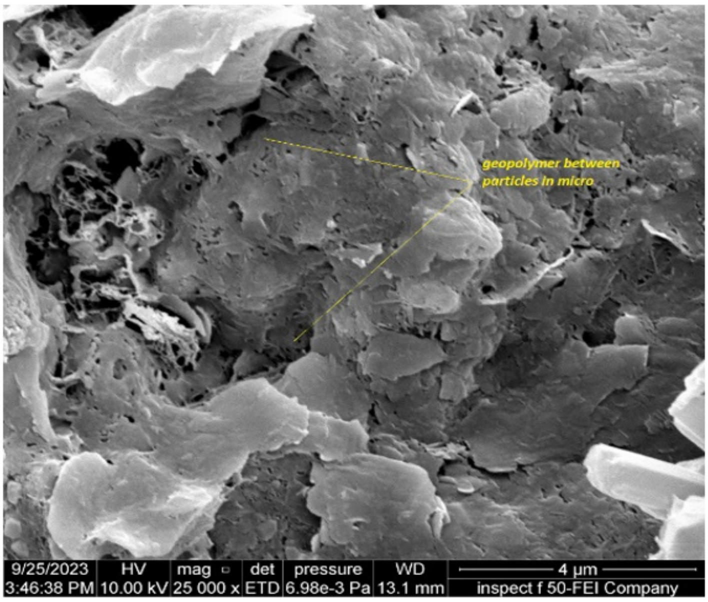

Figure 8. Geopolymer at micropores level of Mixture (20:80)2M

CONCLUSIONS

The primary objective of this study was to discover the role of fly ash geopolymer in improving rammed earth durability against erodibility caused by water by means of microscopic imaging. The results showed that the geopolymer-stabilized specimens possessed excellent durability properties compared to those made of the raw materials alone. During the dip test, the stabilized specimens demonstrated a low mass loss of less than 0,2 %. During the spray test, all the stabilized specimens showed almost no penetration and were highly resistant to erosion.

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The analysis of SEM images of stabilized and unstabilized materials brought out a number of interesting observations. The microstructure of the unstabilized material was characterized with dual porosity, for which macro and micropores coexist. Sand grains of the material were partly coated and interconnected by clay particles and aggregations, which provide weak protection against contact erosion exerted by water. For the stabilized material, the geopolymer products were available as a network at the macro and micro levels. Given the binding nature of the geopolymer, the stabilized material possessed an adequate amount of cementation to resist contact erosion. All the mixtures showed a satisfactory level of cementation despite the differences in molarity of the NaOH solution and fly ash content.

Finally, the results presented in this work confirmed previous observations that stabilizing rammed earth with additives is indispensable to ensure long-term durability. The fly ash geopolymer is one of the potential binders for stabilizing rammed earth.

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