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Microstructural analysis of fired clay masonry brick under aggressive environment

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Abstract: The microstructural changes in fired clay masonry brick, exposed to various aggressive environments were studied. Test samples were subjected to sodium sulphate, sodium chloride and a combination of chloride-sulphate. Beside, for the durability of specimens exposed to chloride and sulphate attack, 5, 10, and 15% solutions were used. Micrographs of the internal structure were taken at the age of 180 days. Energy-dispersive X-ray spectroscopy (EDX) analyses were performed on scanning electron microscope (SEM) images. SEM/EDX studies revealed that halite is the main deterioration product for the samples exposed to sodium chloride where thenardite formations were dominant products of deterioration in the case of sulphate attack. However, the combination of sodium sulphate-chloride produced a double compound in the specimens.

Keywords: Microstructure, Chloride, Sulphate, Fired Clay Brick

I. Introduction

Fired-clay brick is made from selected clay and is produced by shaping it to the standard shape and size. The brick is then fired for 10 to 40 hours in a kiln at a temperature of 750 to 1300 ºC. According to Lynch (1993), under-burnt brick is generally softer than its counterparts, whereas an over-burnt brick is harder, darker, and often smaller. Nevertheless, fired-clay bricks still have a similar size, shape, and function compared with other types of bricks such as cement sand bricks and calcium silicate bricks but may possess different deformation characteristics.Salt attack is one of the major causes of decay to masonry materials such as brick or mortar. This phenomenon occurs due to the presence of soluble salt in the material after the evaporation process. The process normally produces efflorescence or salt crystals.

Zsembery (2001) stated that sodium sulphate and sodium chloride causes most cases of salt attacks. Sodium sulphate is normally present in many bricks and stones, in Portland cements, and in some groundwater. Chloride comes from the salt-laden air near the sea through the mixture of water and groundwater. Ahmad (2004) found that the percentage of sulphate exceeded the safe level of 0.020%, with the highest level recorded at 6.27%. The presence of a very high level of sodium sulphate may deteriorate the surface of the brick and mortars, causing the loss of binders and cohesion. A number of previous studies have been conducted on salt attacks on masonry materials i.e. brick and mortar. However, most studies did not focus on the microstructure analysis of the degree of salt attack on the fired clay masonry brick. This issue is very important, because the understanding of the mechanism of deterioration will be useful in the production of more durable materials and determining an effective repair scheme.

II. MATERIAL AND EXPERIMENTAL PROCEDURE

Fired clay brick

Fired clay brick with five perforations were used in the tests. The nominal size of one brick is 215 x 102.5 x 65 mm. The bricks were manufactured in Selangor, Malaysia. The properties of units which have been determined are strength, water absorption, porosity and initial rate of suction. The strength of fired clay brick was 32MPa with water absorption of 13.42% for 24 hours immersion. Meanwhile the initial rate of suction is approximately 1.97×10^{-3} kg/m^2/min.

Curing condition

Three types of salt exposure conditions were prepared in this study i.e. sodium sulphate, sodium chloride and a combination of sodium chloride and sodium sulphate. Each salt solution was prepared by mixing re-agent grade with water. On the other hand, exposure conditions were considered i.e. dry and wet condition, sodium sulphate, sodium chloride and sodium sulfate-chloride with concentrations of 0%, 5%, 10% and 15% by weight of volume. All specimens were cured under polythene sheet for 14 days in a controlled environment room with 80 ± 5% relative humidity and a temperature of 25 ± 2ºC. After that, the specimens were gradually sprayed for three times with salt solution every 30, 60 and 90 min on the surface.

Scanning Electron Microscope (SEM)

Zeiss Supra 35VP Field Emission Scanning Electron Microscope (FESEM) was used to characterize the samples in this study. In order to determine the morphological characterization, the samples need to be cut into small sizes (except for powder). The sample then needs to be horizontally placed on the substrate holder (180^{o}) for the surface analysis and vertically at 90^{o} for the cross-sectional view (thickness). Magnification of 5 kX, 10 kX, 20 kX, and 50 kX were used for a detailed microstructure of the samples with the operation power of 3 kV and 5 kV.

III. RESULTS AND DISCUSSION

Microstructure of Fired-Clay Masonry Brick Subjected To Dry and Wet Condition

Fig. 1 shows the SEM image of the fired-clay bricks exposed to dry and wet conditions (controlled samples). To determine the percentage elements in the sample, EDX analysis were performed and the results are tabulated in Table 1. Based on the figure, the nature of the microstructure is not significantly different. The unit pores were connected and dendritic in nature. From Table 1, the elements considered common, such as carbon, sodium, aluminium, silica, and calcium. However, these elements, except for carbon and sodium, decreased when exposed to wet conditions. A high percentage of carbon existed in the sample during the coating process of the samples. This could be used to prevent the build-up of an electrical charge on the surface of the sample. Furthermore, the incomplete burning of the fired-clay brick during manufacturing could have also influenced the percentage of carbon within the fired-clay brick bodies. The existence of carbon during manufacturing could cause the expansion and variation in the behavior of the fired-clay brick units.

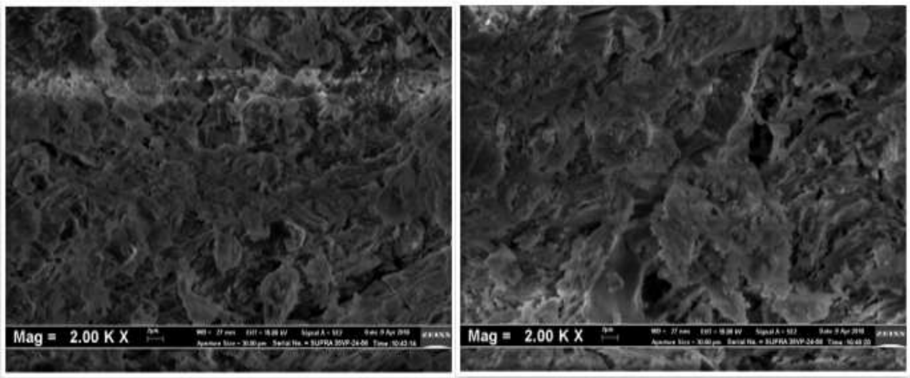

Fig. 1: SEM image of fired-clay brick exposed to dry condition (left); wet condition (right)

Table 1: Percentage of atom for elements in fired-clay brick

Exposure condition	Percentage of atom (%)									Total
	C	Na	Mg	Al	Si	S	Cl	K	Ca	
Dry	12.77	0.04	0.66	0.89	6.44	Nil	Nil	0.31	78.9	100
Wet	63.79	0.65	2.00	1.76	9.04	Nil	Nil	0.29	22.48	100

Microstructure of Fired-Clay Masonry Brick Subjected To Sodium Chloride Solution

The microstructure of the fired-clay brick samples exposed to 5%, 10%, and 15% concentrations of sodium chloride are illustrated in Fig. 2. The presence of halite was observed in the fired-clay bricks when subjected to sodium chloride. No other crystals were found. Furthermore, the shape of halite was different when the concentration of sodium chloride increased. For the specimens exposed to 5% and 10% concentration of sodium chloride, the shape of halite was difficult to interpret. In contrast, in the specimens affected with 15% concentration of sodium chloride, the shape of halite looked like a flaky mushroom. The presence of the chloride atom was observed in the elemental analysis, as presented in Table 2. Generally, the percentage of chloride atom increased when the concentration of sodium chloride increased. However, the increase of the percentage of chloride atom did not cause any defects on the fired-clay bricks, despite the occurrence of expansion.

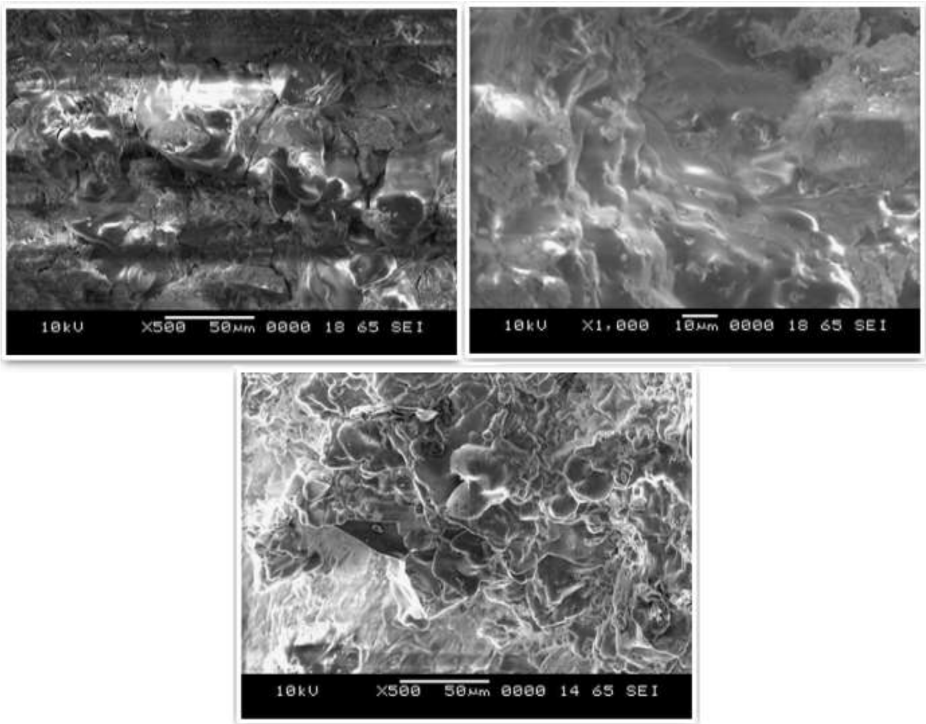

Fig. 2: SEM image of fired-clay brick subjected to 5% sodium chloride (left), 10% sodium chloride (right), and 15% sodium chloride (bottom)

Table 2: Percentage of atom for elements in fired-clay brick

Exposure condition	Percentage of atom (%)									Total
	C	Na	Mg	Al	Si	S	Cl	K	Ca	
5% Chloride	32.28	2.81	0.39	13.64	37.57	Nil	8.35	1.95	3.03	100
10% Chloride	46.82	15.04	0.00	0.76	2.69	Nil	33.51	0.11	1.09	100
15% Chloride	32.93	28.01	0.00	0.18	0.48	Nil	38.13	0.04	0.23	100

Microstructure of Fired-Clay Masonry Brick Subjected to Sodium Sulphate Solution

In the fired-clay brick zones exposed to sulphate ions, the sodium sulphate crystal was thernadite (Benevente et al., 2004). SEM confirmed the presence of thenardite, and the mineral phase was found to be heterogeneous. The shape formed was also different, depending on the concentration of sodium sulphate. For instance, from Fig. 3, at 5% sodium sulphate concentration, thenardite was observed, but the shape formed was difficult to interpret. However, at 10% and 15% of sodium sulphate, the thenardite shape was like a diamond. All the thenardite formed inside the surface and were associated with the porosity of the samples. The fired-clay bricks samples were saturated in the sodium sulphate condition and caused the specimens to expand due to the development of crystallization pressure. In materials with small pores, the crystallization pressure is higher than in materials with large pores (Benevente et al. 2006). The EDX analysis confirmed the compositions of elements present in the specimens as given in Table 3. The presence of atom sulphate was noticeable after exposure to sodium sulphate. It can be concluded from Table 3 that the percentage of atom sulphate increased when the concentrations of sodium sulphate increased. The increment of atom sulphate caused the expansion of fired-clay brick to increase.

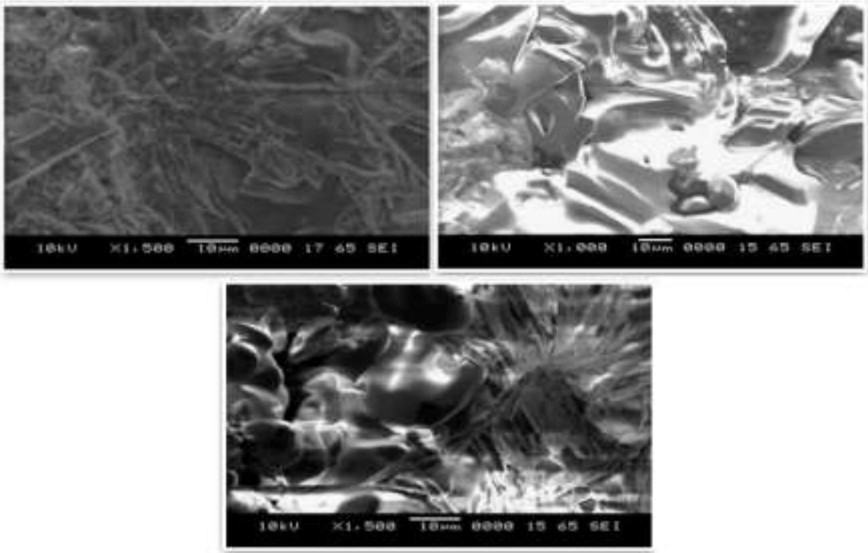

Fig. 3: SEM image of fired-clay brick subjected to 5% sodium sulphate (left), 10% sodium sulphate (right), and 15% sodium sulphate (bottom)

Table 3: Percentage of atom for elements in fired-clay brick

Exposure condition	Percentage of atom (%)									Total
	C	Na	Mg	Al	Si	S	Cl	K	Ca	
5% Sulphate	19.16	40.77	0.42	2.85	8.45	27.75	Nil	0.58	0.04	100
10% Sulphate	33.41	37.80	0.00	0.11	0.33	28.21	Nil	0.07	0.07	100
15% Sulphate	16.23	27.29	0.00	0.08	0.34	55.98	Nil	0.10	0.00	100

Microstructure of Fired-Clay Masonry Brick Subjected to Sodium Sulphate-Chloride Solution

The combination of sodium sulphate and sodium chloride produced a double compound in the specimens as shown in Fig. 4 and was confirmed by EDX analysis, as given in Table 4. The results clearly indicate that the expansion of fired-clay brick was due to the crystallization of sulphate ions and chloride ions. For instance, at 10% and 15% concentrations of sodium sulphate-sodium chloride solutions, the crystal shape looked like a diamond, which could be interpreted as the stone at the mountain from the plan view. However, the crystal shape after subjected to 5% concentration of double salt solution was difficult to interpret.

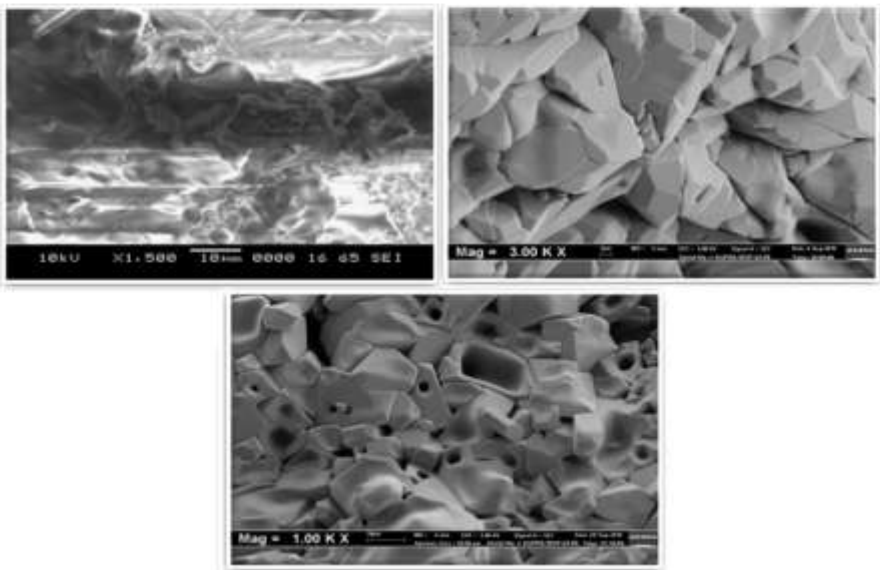

Fig. 4: SEM image of fired-clay brick subjected to 5% sodium sulphate-chloride (left), 10% sodium sulphate-chloride (right), and 15% sodium sulphate-chloride (bottom)

Table 4: Percentage of atom for elements in fired-clay brick

Exposure condition	Percentage of atom (%)									Total
	C	Na	Mg	Al	Si	S	Cl	K	Ca	
5% (Sulphate + Chloride)	37.74	27.02	0.00	0.52	1.33	Nil	33.26	0.07	0.07	100
10% (Sulphate + Chloride)	34.63	20.27	0.00	0.00	0.49	19.00	26.92	0.00	0.84	100
15% (Sulphate + Chloride)	25.24	36.62	0.00	0.34	0.66	0.77	36.22	0.00	0.12	100

IV. Conclusions

- Based on the obtained data in this study, the following conclusions can be drawn:
- The changes in microstructure caused by the ingress of sodium chloride and sulfate are clearly manifested by SEM analyses.
- The mode of deterioration was different in the presence of chloride and sulphate solutions.
- The halite formation was the main deterioration mechanism in the specimen exposed to sodium chloride and thenardite formation related to sulphate attack.
- The combination of sodium sulphate-chloride produced a double compound in the specimens such as crystallization.
- It is suggested that a further detailed investigation is needed for the evaluation of the resistance fired clay brick to chloride and sulphate attack.

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VI. References

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