

## XRD Investigation of Fired Clay Brick Masonry Subjected To Aggressive Environment

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**Abstract:-** In this paper, the fired clay brick under sodium sulphate and chloride salt attack were investigated. Chemical changes that occur in specimen at the microstructure level due to environmental effects are identified by X-ray diffraction (XRD) analysis at the age of 180 days. For the durability of specimens under sodium chloride and sulphate attack, 5, 10, and 15% solutions were used. The results show that the formation of halite and thenardite can strongly affect the performance of specimens. However, the combination of sodium sulphate-chloride produced a double compound in the specimens.

**Keywords:-** XRD, Fired Clay Brick, Chloride, Sulphate, Durability

### I. INTRODUCTION

Salt attack is a process that needs a combination of permeable masonry, moisture, soluble salt, and evaporation [1]. The effect of salt attack on masonry material and structures can present in different forms, i.e., efflorescence, scaling, cracking, crumbling, and softening [2, 3]. Sodium chloride is a common salt in contaminated building materials and may cause material damage especially in urban, coastal, and dry land environments [4, 5]. It usually comes from rising damp, the wind, sea spray, or even flooding. However, in most cases, the main source is sea spray. Lubelli et al. classified the effect of sodium chloride on masonry damage in two cases: direct and indirect [6]. The direct case is due to crystallization, hygroscopicity, or thermal expansion mechanisms. The indirect case is related to the catalytic role that halite plays in some chemical reactions [6]. Halite is a stable phase present in sodium chloride–water system under room conditions. It is normally carried by sea spray and penetrates the solution in the masonry material especially in the porous bricks. When the masonry dries out, the salt crystallizes and generates pressures that lead to the powdering of the material. Sodium sulphate is one of the most common salts involved in salt crystallization decay. Crystallization of sulphate within the material's pore structure has been considered the likely cause of damage. In a study of crystallization of porous stone using sodium sulphate solution, Benevente et al. found that mirabilite and thenardite crystal grow inside the stone surface, producing sub florescence that strongly contribute to stone damage [7]. These findings verify that crystallization pressure is more effective when crystallization is produced within the mass. It is well known that chloride and sulphate attack on fired clay brick is related to the formation of halite and thenardite, but the research about halite and thenardite is still few, especially its mineralogical analysis. For this purpose, X-ray diffraction (XRD) investigations were carried out to demonstrate the effect of fired clay brick under aggressive environment.

### II. EXPERIMENTAL STUDY

#### Materials

Fired-clay bricks with five perforations were used in the study. The nominal size of one brick is 215 x 102.5 x 65 mm. Table 1 shows the chemical characteristics of fired-clay brick used through the investigation. The fired-clay brick is mainly composed of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>. The presence of SiO<sub>2</sub> content is associated with the quartz particles, and Al<sub>2</sub>O<sub>3</sub> is associated with gibbsite [8].

Table 1. Chemical compositions of fired-clay brick

CO <sub>2</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	TiO <sub>2</sub>	MgO	CaO	Na <sub>2</sub> O	SO <sub>3</sub>	Zr
0.1	63.4	25.2	5.84	2.68	1.34	0.753	0.345	0.148	Nil	0 < LLD

### Sample Preparation

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 weight volume with the concentrations of 5, 10, and 15%. The exposure solutions were prepared by mixing re-agent grade sodium sulphate or sodium chloride with water. The combination of salt solution, sodium chloride and sodium sulphate were prepared based on the level of minimum concentrations of salt that could affect the masonry material i.e. 5% of sodium sulphate and 15% of sodium chloride.

### X-Ray Diffraction (XRD) Test

XRD is the most common technique used to determine the mineralogical composition of masonry material that exists in a crystalline state. This technique is also attractive and efficient because it requires only a small amount of material or sample which is about 2 gram. Samples were gently ground and passed through a 100 $\mu$ m sieve. The samples were taken from the bonded brick bed face after 180 days of exposure to different concentrations of soluble salts.

## III. RESULTS AND DISCUSSION

### X-Ray Diffraction of Fired Clay Brick under Normal Conditions

XRD results garnered from a limited number of samples taken from the fired-clay brick interface show that for the control specimens, all clay samples exhibited the presence of quartz as the most predominant non-clay mineral constituent, as presented in Fig. 1. Based on figure, it can be detected that the highest peak of quartz mineral was found at 27° 2 $\theta$ . However, the major phase found in the specimen was quartz.

### X-Ray Diffraction of Fired Clay Brick under Sodium Sulphate Solutions

The XRD analysis for the samples exposed to the sodium sulphate solution is illustrated in Fig. 2. The main component is identified as thenardite. Benevente et al. also found that thenardite was one of the stable phases developed underneath the brick when exposed to the sodium sulphate-water system [7]. It can be said that the presence of thenardite is due to the crystallization of sodium sulphate underneath the surface. However, the peaks of thenardite for all concentrations were small and could not be easily identified, indicating that the thenardite is poorly crystallized within the fired-clay brick bodies. Still, the formation of thenardite increased with the increase in sodium sulphate concentrations. For instance, at 5% sodium sulphate, the number of peaks developed was lower than that of 10 and 15% sulphate solution.

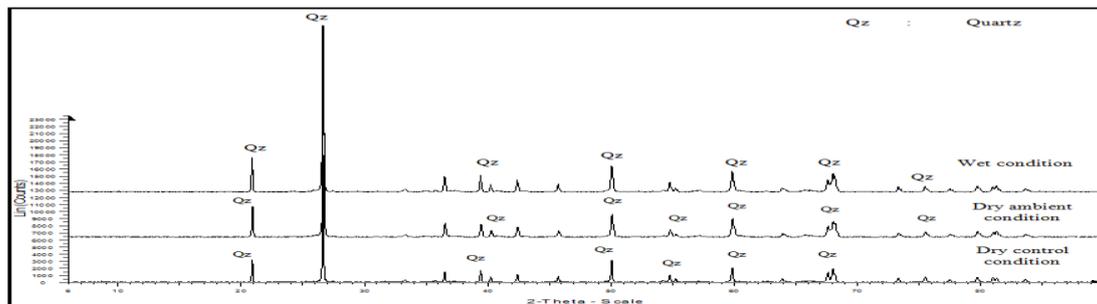


Fig. 1. X-ray diffraction analysis of fired-clay brick under control conditions

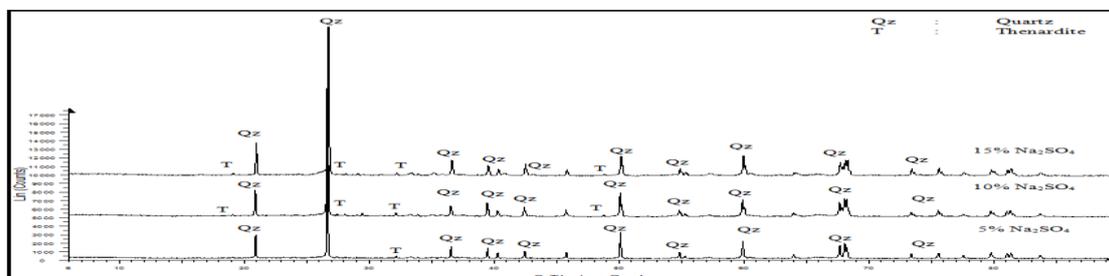


Fig. 2. X-ray diffraction analysis of fired-clay brick under sulphate solutions

As seen in Table 2, the peak of thenardite for these samples was around at 32° and 34° 2 $\theta$ . Meanwhile, the samples affected with 10% and 15% concentrations of sodium sulphate showed a possible increase in the thenardite peaks. The peaks developed at 19° 2 $\theta$  and between 27° and 34° 2 $\theta$ , as well as at 49° 2 $\theta$ . It can be concluded that the presence of this compound led to the expansion of the brick units. The expansion was

approximately 1.5 times the expansion of the control samples. Finally, the sodium sulphate could have a very damaging effect on fired-clay brick masonry.

Table 2. The peak of thenardite for fired-clay brick under sulphate solutions

Soluble salt concentration	Compound Existed	2-Theta-Scale
5% sodium sulphate	Thenardite	32° and 34°
10% sodium sulphate	Thenardite	19°, 27 - 34° and 49°
15% sodium sulphate	Thernadite	19°, 27 - 34° and 49°

**X-Ray Diffraction of Fired Clay Brick under Sodium Chloride Solutions**

The formation of halite occurred due to the crystallization of sodium chloride as illustrated in Fig. 2. Halite is a stable phase present in sodium chloride-water system. Lubelli et al. classified the formation of halite as indirect case which relate with catalytic role [6]. Fig. 3 shows that, the halite peaks were higher than those of thenardite and could easily be identified. The height of the peak also increased when the concentration increased. However, the presence of halite was not enough to deteriorate the fired-clay brick unit, where no large expansion was observed due to the presence of halite in all samples affected with sodium chloride. For instance, the samples affected with 5% concentration of sodium chloride showed the lowest peak at 31.5° and 45.5° 2θ (Table 3). The samples affected with 10% and 15% concentrations of sodium chloride showed a possible increase in the halite peak at the same 2-theta scale (see in Fig. 3).

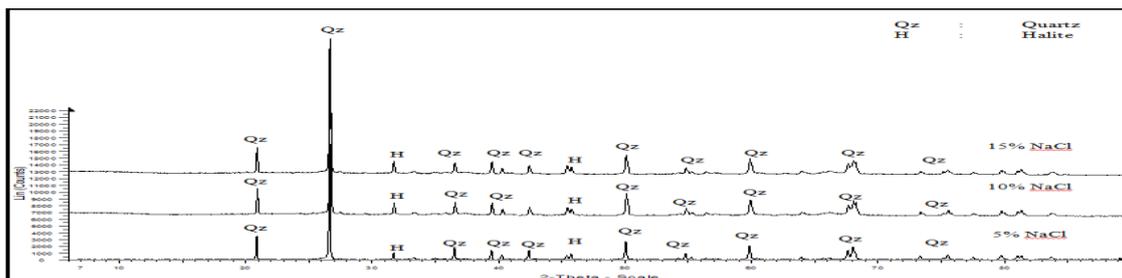


Fig. 3. X-ray diffraction analysis of fired-clay brick under chloride solutions

Table 3. The peak of halite for fired-clay brick under chloride solutions

Soluble salt concentration	Compound Existed	2-Theta-Scale
5% sodium chloride	Halite	31.5° and 45.5°
10% sodium chloride	Halite	31.5° and 45.5°
15% sodium chloride	Halite	31.5° and 45.5°

**X-Ray Diffraction of Fired Clay Brick under Sodium Sulphate-Chloride Solutions**

Fig. 4 shows test results of the XRD due to sulphate-chloride attack of the fired clay brick. From the results of sodium sulphate-chloride attack in Fig. 4, the presence of thenardite and halite in the microstructure due to the crystallization of these salts. As shows in Table 4, the peaks of thenardite were present at 32°, 40.5°, and 51° 2θ. For halite, the peaks were observed at 27.5°, 32°, and 45.5° 2θ. The number of peaks that could be observed was the same for both types of compounds, although the height of thenardite peaks was lower than the halite peaks (Fig. 3). However, the peaks of thenardite and halite for all concentrations were small and could not be easily identified. It was found that the sulphate-chloride ions are poorly crystallized within the fired-clay

brick bodies. This situation may be due to the reaction between chloride ions and sulphate ions, where the chloride ions reduces the attack by sulphate ions.

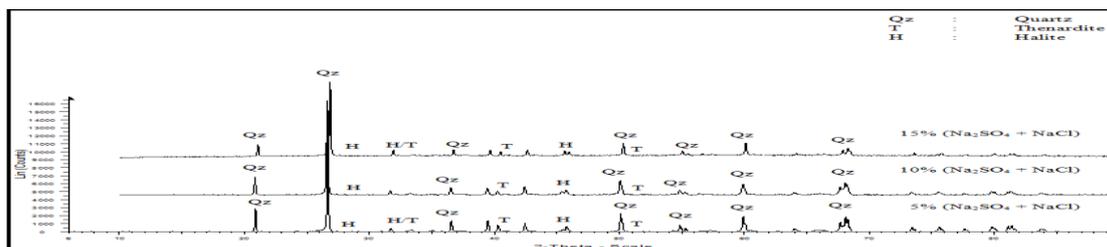


Fig. 4. X-ray diffraction analysis of fired-clay brick under sulphate-chloride solutions

Table 4. The peak of halite and thenardite for fired-clay brick under sulphate-chloride solutions

Soluble salt concentration	Compound Existed	2-Theta-Scale
5% sodium sulphate-chloride	Halite Thenardite	27.5°, 32° and 45.5° 32°, 40.5° and 51.5°
10% sodium sulphate-chloride	Halite Thenardite	27.5°, 32° and 45.5° 32°, 40.5° and 51.5°
15% sodium sulphate-chloride	Halite Thenardite	27.5°, 32° and 45.5° 32°, 40.5° and 51.5°

#### IV. CONCLUSIONS

The presence of thenardite was due to the crystallization of sodium sulphate in the samples, whereas the presence of halite was due to the crystallization of sodium chloride. However, the presence of these compounds was sometimes very small and difficult to identify. Furthermore, some of the other chemical compounds in the samples could not be easily detected by XRD. The reason is that the XRD has higher detection limits, such that minor phase changes may not be easily seen in the pattern.

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