P- ISSN 1991-8941 E-ISSN 2706-6703
2024,(18), (02):271 – 282
Journal of University of Anbar for Pure Science (JUAPS) Open Access

Chemical Treatment of Efflorescence in Clay Bricks During the Manufacturing Process

Mahmoud H. Al-Salmany[1]*, Mohammed A. Al-Nuaimy[1]

Department of Applied Geology, College of Science, University of Anbar, Ramadi, Iraq

ARTICLE INFO

Received: 17 / 04 /2024
Accepted: 30/ 06 /2024
Available online: 30/ 12 /2024

10.37652/juaps.2024.148903.1234

Keywords:
Bricks,Efflorescence,Barium Carbonate, clay, Nahrawan.

ABSTRACT

Efflorescence refers to the white deposit of salts that are deposited on the faces of buildings constructed using bricks, resulting from the evaporation of water loaded with dissolved salts. The source of these salts is usually from the raw material itself. It is an aesthetic issue that affects the durability of building units. This study aimed to treat this phenomenon chemically during the manufacturing process by adding inhibitors. Barium carbonate ($BaCO_3$) was used as an inhibitor in proportions ranging from 2% to 20% added to Al-Nahrawan and Al-Angor clay, respectively. The samples were burned at 950 °C. Efflorescence test was performed according to ASTM C67-08. Apparent porosity, water absorption, and linear shrinkage tests were performed. The results of the tests showed that the efflorescence rate decreased as the inhibition rate increased, because barium carbonate led to the conversion of water-soluble salts into insoluble salts. The tests showed a slight increase in the percentage of pores and water absorption and an increase in the length of the samples as the barium carbonate percentage increased.

1. Introduction:

Efflorescence is the emergence of dissolved salt crystals on the surface of brick walls. The evaporation of salt-saturated water induces this phenomenon. This defect is commonly observed on brick, mortar, and concrete facades. It primarily consists of whitish deposits of water-soluble salts, like alkali sulfates or sodium chloride, which generally appear soon after the construction of the facade. The high solubility of salts facilitates their transport to the facade's surface via moisture flow, and they can also be easily washed away from the surface by natural weathering [1]. This phenomenon is one of the most important problems facing the clay brick industry. This is due to its negative impact on the durability and strength of the walls when efflorescence progresses to advanced stages [2][3]. It also affects the aesthetics of the facades and walls built from clay bricks [4]. Efflorescence affects the beauty of building facades, especially in historic buildings such as the ancient city of Babylon. It was first studied by Merrigan in 1903, followed by many studies by Grogan, Conway, Robinson, Grimm, Hurd, and London [5].

*Corresponding author at : Department of Applied Geology, College of Science, University of Anbar, Ramadi, Iraq
ORCID:https:// https://orcid.org/0000-0000-0000-0000 ,
Tel: +964 7805868636
Email: mah22s4004@uoanbar.edu.iq

Sulfate salts are one of the most common salts of efflorescence on clay bricks. Many types of sulfates are usually found on the surface of clay bricks after contact with water. The primary source of these salts often originates from the clay brick itself; however, other types of salts from outside contamination often appear as efflorescence. The mechanism underlying the formation ofefflorescence of these sulfate salts on the surface of clay brick or other types of building units is not fully understood. The literature presents conflicting views on the source and development of efflorescing sulfate salts. According to Butterworth, there are two main sources of soluble salts in brick that inherently contribute to efflorescence formation. Sulfates are developed during firing by the action of sulfur gases and sulfate salts present in the raw clay [6].

2. Causes of Efflorescence Phenomenon

Several factors, if combined, will induce the precipitation of salts on the surfaces of building materials, resulting in efflorescence. The absence of any of these factors precludes the precipitation of salts on the surfaces and facades of buildings constructed from clay bricks or concrete blocks. These factors are as follows [7], [8]: 1) Soluble salts must be present either inside the building materials such as bricks, concrete blocks, or mortar or be in direct contact with these materials. 2) A source of water must be available and in contact with these salts for a sufficient period to dissolve them. 3) The structure must contain pores that allow water-carrying dissolved salts to move to the surface or other places where evaporation can occur [9]. According to [10], four conditions must be met for efflorescence to occur: the presence of water, a source of soluble salts, a wall structure with pores to allow water passage, and weather conditions.

3. Source of the Salts Causes Efflorescence

The direct cause of the phenomenon of efflorescence in bricks and ceramic materials is the migration of an aqueous solution loaded with dissolved salts through the pores in bricks or ceramic materials, where they are deposited on the external surfaces due to evaporation. These salts may be from multiple sources, including through the combustion products of the fuel used in burning bricks, or from the place where the product is stored or may come from the mortar used in construction, or present in the raw material that is developed during the drying or firing process of bricks [8]. Bricks may contain calcium sulfate in the form of anhydrite ($CaSO_4$), which can be one of the most important sources of salt causing efflorescence [11]. The salts responsible for efflorescence may originate from water used in the brick industry, which can include well water, rivers, or other water sources that contain different types of dissolved salts, including calcium carbonate, magnesium, sodium sulfate, other chlorides, nitrates, and phosphates [12]. Sulfates are the primary cause of efflorescence in bricks, originating from various sources, including mortar, soil, air, or rain. Within bricks, sulfates develop through the firing process and can persist based on the peak firing temperature. In mortar, sulfates are commonly derived from calcium sulfates (such as gypsum, anhydrite, or hemihydrate) in raw material or from any other source [13]. This study aimed to produce free-efflorescence clay bricks by treating efflorescence chemically during the manufacturing stages, incorporating chemical materials as inhibitors to the raw materials from different two sites to convert soluble salts to insoluble ones, and performing evaluative tests on them, including the efflorescence test.

4. Previous works

Many studies have been conducted to investigate all aspects of this issue, including the causes of efflorescence, the types of salts that cause efflorescence, methods of examining efflorescence, and methods of treatment. Some previous research that analyzed the subject of efflorescence in clay bricks are summarized below.

[14] studied the possibility of applying some techniques to solve the efflorescence problem. He studied shale rocks and found that the main cause of efflorescence is sodium sulfate (Na_2SO_4), and this salt is formed during burning by sulfur gases resulting from the oxidation of iron sulfide mineral (FeS_2) with sodium minerals. He found that the addition of silica is effective in reducing efflorescence by reacting with sodium sulfate, and the addition of ammonium chloride (NH_4CI) destroys iron sulfide at low temperatures, preventing the formation of sodium sulfate. A low content of free silica allows sodium sulfate to appear in the final product. Moreover, increasing the firing temperature leads to a reduction in efflorescence. These additives and techniques can be used to solve similar efflorescence issues. In the same context, [15] studied methods to produce clay bricks and tiles by treating clay and shale or other raw materials used in the ceramic industry containing (pyrite) metal where raw materials can be ground and mixed with an oxidizing agent in the pre-forming step. The oxidizing substance is dispersed inside the clay until the largest surface area of clay is exposed to the oxidizing substance. One of the most important oxidants that can be used is an aqueous solution of hydrogen peroxide. After brick samples form, it is heated to high temperatures, and the pyrite inside the mud is oxidized, thereby removing sulfur-containing compounds such as sulfur dioxide from the mud. This method can help prevent the occurrence of an efflorescence phenomenon by completely or almost completely removing pyrite. [16] performed an experimental work assessing the effect of adding(H_2O_2 and $BaCO_3$ as supplementary substances that reduce efflorescence. They also conducted some experiments and used different concentrations of hydrogen peroxide aqueous solutions. The addition of barium carbonate ($BaCO_3$)to clay during the grinding process also led to

the conversion of soluble sulfates into an insoluble form of $BaSO_4$ according to Equation 1.

$Na_2SO_4 + BaCO_3 \rightarrow Na_2CO_3 + BaSO_4$…(1).

The supplementary substances were added at rates of 1% and 3%. The results indicated the presence of $BaSO_4$ deposits in the water extract. The test showed that the amount of supplementary substances added ($BaCO_3$ or H_2O_2) was not sufficient to eliminate the efflorescence because the high percentage of salts in the soil required the addition of supplementary substances in large proportions. [17] studied the effect of inhibitors and firing temperature on reducing the appearance of efflorescence in clay products. He studied the removal of sulfate from the clay materials. In addition, sulfate ions were stabilized by forming inactive compounds insoluble in water. To reduce the solubility of sulfate, three inhibitors (barium chloride $BaCl_2$, barium hydroxide Ba $(OH)_2$, and barium carbonate $BaCO_3$) were used at different concentrations of 0.5%, 1.0%, 1.5%, and 2% were mixed homogeneously with distilled water to prepare the samples using firing temperatures of 800 °C, 900 °C, and 1000 °C. The addition of $BaCO_3$ at 2.0% and 900 °C firing temperature produced the lowest sulfate filtration due to the formation of water-insoluble $BaSO_4$. Samples without the addition of barium carbonate were examined, and efflorescence was observed on these samples, whereas samples with the addition of barium carbonate did not develop efflorescence; these results confirmed the effectiveness of $BaCO_3$ in eliminating efflorescence. Thus [18], the treatment of efflorescence in clay bricks by using sodium hydroxide and sodium silicate as an environmentally friendly binder could improve the properties of brick materials; this type of treatment is known as geopolymer technology, where sodium hydroxide is also called caustic soda. Sometimes it is used instead of acid to clean blocks or masonry. It also serves as an alkaline activator that neutralizes chloride and sulfate ions, reducing efflorescence. Sodium silicate works to reduce the permeability of the walls, which reduces or prevents the penetration of water and the formation of efflorescence. [9] tested a new method for analyzing efflorescence instead of the standard ASTM C67 method. Samples with dimensions of 2 cm×2 cm×20 cm were used and submerged in 5 cm of water for 5 days instead of 7 days in containers covered with a rubber membrane. In this method, he used smaller samples, less time, and more water than the standard method. He compared the results he obtained with the results of the standard method, in which the samples were prone to efflorescence, whereas the method that was used showed that efflorescence was not found, perhaps due to the small size of the samples and a large amount of water used, or the amount of salts was small and insufficient to detect them. [5] conducted an experimental study to address the phenomenon of efflorescence in clay bricks. The main variables considered in this study were as follows: the effect of using artesian well water; the amounts of salts in the soil, silica fumes, date vinegar, kaolin, and glass powder residues; and the burning temperature of clay bricks. The second part of this work deals with how to remove efflorescence after the completion of construction or during construction. From the results of this work, the researchers concluded that the average efflorescence was significantly reduced by about 92.02% when silica fume was added at 15% of the soil weight. The best treatment technique was found to be the use of date vinegar because this technique significantly reduced the average efflorescence (about 89.69%), making it a simple and cost-effective technique.

In the current study, for the first time in Iraq, barium carbonate ($BaCO_3$) was used as an inhibitor material additive to local clays from two different locations. The first one was brought from the Al-Nahrawan area, and the second was from the Al-Angor area to eliminate or minimize the phenomenon of efflorescence. The weight ratios that must be added to each type of clay were determined based on trial and error.

5. Material and Methods

5.1. Material:

Laboratory brick samples were produced for efflorescence treatment using materials that included clay from two different locations. The first comprised deposits from the Al-Nahrawan region, which date back to the Quaternary deposits. The Al-Nahrawan area is located to the northeast of Baghdad Governorate, about 65 km, according to the coordinates shown in Table 1. The clay of the Al-Nahrawan region was brought from a brick factory and are ready to be manufactured. The clay of the other site dates to the deposits of the Fatha Formation middle Miocene, which is Al-Angor region clays in Anbar Governorate. Al-Angor area clay deposits are located about 12 km southeast of the city of Ramadi, according to the coordinates shown in Table1, and they consist of a layer about 3 m-thick of cohesive, dark red-colored, clay interspersed with silt and containing veins of secondary gypsum and a 1.2 m-thick layer of sandy gravel. Fig. 1 illustrates the location map of clay used in this study. As for the supplementary material, barium carbonate ($BaCO_3$) with 98% purity was used to produce free-efflorescence clay bricks. Barium carbonate was previously used for treating efflorescence, but for the first time in Iraq, barium carbonate was added to the local clay mentioned above. It was added to the clay of the Al-Angor region because it is the best clay suitable for manufacturing bricks in Anbar governorate. Moreover, it was added to the clay of the Al-Nahrawan region to compare the results because the clay of the Al-Nahrawan region is the most used in manufacturing bricks in Iraq. Clay from this area suffer from efflorescence, but it is used in the brick, earthenware, tile, and also pottery industries. Barium carbonate was added to clay for the precipitation of soluble salts (calcium sulfate and magnesium sulfate, SO_3) that cause efflorescence [19][20] [21].

Table 1: Raw material locations.

the region	Longitude	Latitude
Al-Nahrawan	44° 50'10.00" E	33°24'46.00" N
Al-Angor	43° 11' 49.00" E	33°29'38.08" N

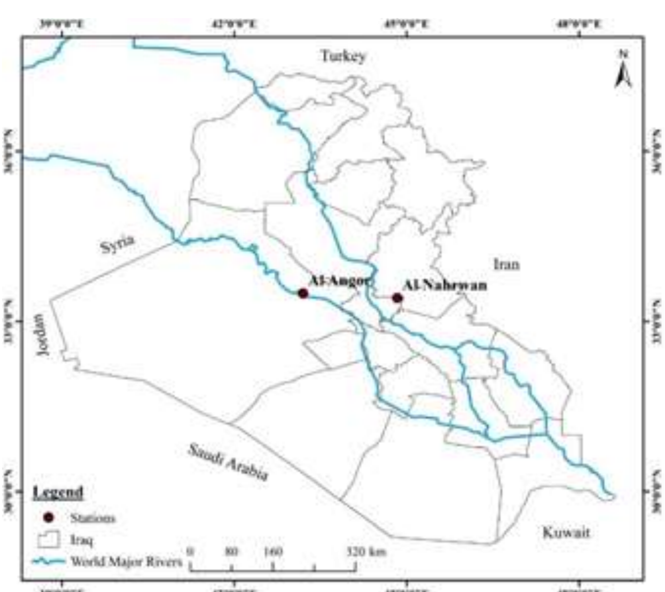


Figure.1 Location map of used raw materials.

5.1.1 XRF analysis of raw materials

The clay chemical composition was measured by XRF, and the data are tabulated in Table 2. The raw materials were suitable for the manufacture of bricks in terms of the main oxide content.

Table 2: Chemical components of used clays.

Oxides	Al-Nahrwan	Al-Angor
Na_2O	3.92	0.89
MgO	4.27	6.72
Al_2O_3	8.42	11.25
SiO_2	37.65	48.04
P_2O_5	0.12	0.16
SO_3	1.03	0.44
Cl	1.44	0.08
K_2O	1.29	2.55
CaO	23.5	20.42
TiO_2	0.72	0.88
Fe_2O_3	4.73	8.06
V_2O_5	0.02	0.02
Cr_2O_3	0.04	0.04
MnO	0.08	0.09

5.1.2 Physical properties of clays

Atterberg limits

The Atterberg limits of clays were determined according to [22], and the results of the Atterberg analysis are shown in Table 3. The results of Atterberg boundary analysis show that the used clays had sufficient plasticity to allow them to be shaped. The clays of the Al-Angor region were plastic with a plasticity index (PI) of 7.2, whereas the clays of the Nahrawan area had a higher plasticity with a PI of 19. On the basis of the Das classification [23], the clays could be categorized into high plasticity, medium plasticity, medium plasticity, and low plasticity.

Table 3: Atterberg limits.

clay	L. L	P. L	P.I %
Al-Nahrwan	40	21	19
Al-Angour	39.5	32.3	7.2

Grain size analysis

Grain size analysis used wet sieving to separate sand from clay and silt and the pipette method to separate silt from clay [24]. Table 4 shows the results of the volumetric analysis of the samples used in this study، which predominantly consisted of clay, with a low percentage of silt and sand. The results of the volumetric gradient test indicated that the clay was suitable for bricks production.

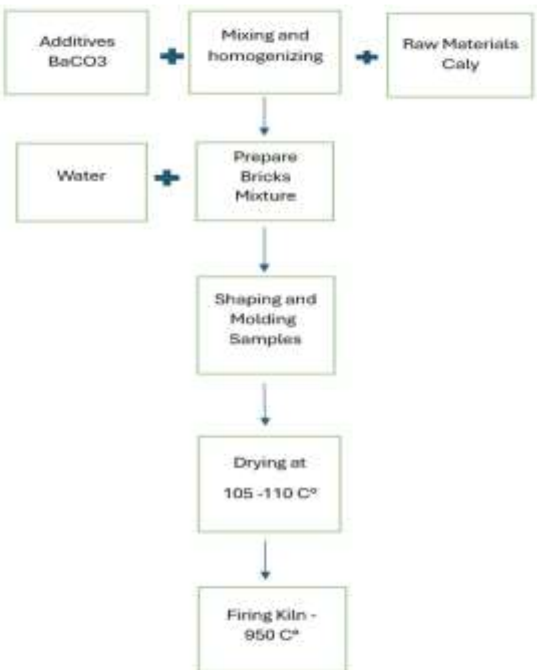
Table 4: Grain size analysis.

Clay	Clay %	Silt %	Sand %
Al-Nahrwan	84.4	12.7	2.9
Al-Angour	83.1	14.4	2.5

5.2 Manufacturing of bricks:

The manufacture of fired clay bricks can be divided into four stages according to the basic principles used for thousands of years: selection and preparation of clay, molding, drying, and firing [25].

However, when supplementary materials are added, secondary phases are added to the four main stages, as shown in Fig. 2.



The figure shows the stages of clay brick production, which goes through several stages, starting with the preparation of materials and mixing with inhibitors, wetting, shaping, drying, and firing.

Figure. 2. Brick manufacturing stages

Preparation of mixtures

The initial phase following clay selection is the process of drying and grinding the clay by using a Tema mill grinding machine to a fine size of about 300 microns because the semi-dry pressing method requires the material to be very fine in size. Subsequently, the clay was mixed with supplementary materials, namely, barium carbonate ($BaCO_3$), in different weight ratios with clay selected from the Al-Nahrawan and Al-Angor sites (Table 5). After choosing the $BaCO_3$ ratios, the mixture was blended well under dry conditions to ensure that the supplementary materials were distributed to all parts of the mixture within a plastic bag. The mixture was then moistened with 8% distilled water and allowed to ferment and homogenize for 24 h in airtight plastic bags to maintain moisture for future molding. Different proportions of barium carbonate were used to determine the lowest proportion that would yield the best results. The optimal ratio of ($BaCO_3$) is determined based on the results of the efflorescence test.

A clay that needs a high percentage of barium carbonate does not necessarily mean that we must use this percentage; we can use a low percentage to partially address the efflorescence problem or minimize its effect.

Table 5: Additive ratios of $BaCO_3$.

No. Sample	Additive ratios (%)	
	Clay	$BaCO_3$
AB-2	98	2
AB-5	95	5
AB-10	90	10
AB-20	80	20
DB-2	98	2
DB-5	95	5
DB-10	90	10
DB-20	80	20

Shaping of brick samples

There are many brick-shaping methods. The method of forming and molding clay bricks depends on the nature of the clay used, especially the plasticity, moisture content, and the type of bricks required [26]. Bricks can be molded by hand, which is the oldest used method in brick shaping, and this process gives a required shape to the bricks. Molding may be carried out by hand or by machine [27]. Several other methods can be used in the clay brick molding process. Bricks can be molded by soft mud process, extrusion process, stiff plastic, and semi-dry process [26]. However, the two most commonly used methods of forming clay bricks are extrusion and semi-dry pressing [28]. Extrusion is the most modern process that can produce all types of bricks such as solid and hollow bricks. The clay is mixed with about 10%–15% water, and this percentage is not fixed and may increase depending on the plasticity of the clay. Another method that is used is dry pressing. The semi-dry pressing process is also used for clays with low plasticity. In this method, water is added in small proportions of around 7–8 wt.%. The advantages associated with the semi-dry pressing technique are the high production rate and short drying time [29]. The semi-dry pressing method was used in this study. After the percentages of the supplemental additives were determined, they were mixed thoroughly and fermented with the additives used. Two types of molds were used, namely, semi-cubic and cylindrical. The samples were pressed by a hydraulic press with pressure of 2.5 kn/cm^2. Each sample was then given a unique code before the drying process.

Drying

The process of drying brick samples before firing is a very important stage because newly made bricks contain water. This water must be completely removed before the firing process to prevent cracks in the brick body resulting from the rapid evaporation of moisture during the firing process [30]. The samples were dried using an electric oven at 105 °C–110 °C.

Firing

Firing is an essential and critical stage in the manufacturing process of bricks and ceramic products because it is responsible for most of the properties of the final product. These properties include mechanical strength, dimensional stability, corrosion resistance, water and chemical resistance, and fire resistance [30]. The firing of clay bricks results in a series of mineralogical, textural, and physical changes that depend on many factors that affect several properties of the bricks, including porosity, compressive strength, and color [31]. Brick samples manufactured in the laboratory were fired in an electric kiln according to a specific firing program. After drying the samples, they were placed in the kiln and heated at a rate of 10 °C/min until the temperature reached 600 °C. Thereafter, the heat rate was changed to 5 °C/min to reach the desired temperature of 950°C with a soaking time of 2 h. The kiln was then turned off, and the samples were allowed to cool to perform the required tests later.

6. Result and Discussion

The results of the analyses conducted on brick samples manufactured in the laboratory showed the effect of adding different percentages of barium carbonate for the purpose of efflorescence treatment to the clays of Al-Nahrawan and Al-Angor, respectively, on porosity, water absorption, linear shrinkage, and efflorescence.

Apparent porosity

An apparent porosity test was performed according to [32] for the samples after burning, where the samples were completely dried in an electric oven. The dry weight w1 was recorded. Thereafter, the samples were immersed in water for several hours until the sample was completely saturated with water. The sample was then wiped with a piece of cloth, and the saturated weight w2 was recorded. Subsequently, the samples were suspended with a string and immersed in water without touching the bottom. The submerged weight w3 was recorded, and the following equation was applied to calculate the apparent porosity. Table 6 shows the result of the apparent porosity test.

$$A.P = \frac{w2-w1}{w2-w3} x100 \ldots. (1)$$

The results of the porosity ratio test showed a slight increase in the size of the pores associated with an increase in the barium carbonate ratio, and this increase in the porosity ratio was associated with an increase in the size of the burnt brick samples.

Water absorption

The percentage of water absorption is one of the important factors affecting the resistance of bricks; the lower the percentage of water absorption, the more durable and resistant to environmental conditions such as freezing and thawing [33]. The water absorption rate was calculated [32] by applying the following equation. Table 6 shows the results of the test.

$$W. A= \frac{w2-w1}{w1} x100 \ldots (2)$$

The increase in the percentage of pores in the brick body is accompanied with a slight increase in water absorption, which is associated with an increase in the percentage of barium carbonate. Table 6 shows the results of the water absorption ratio test.

Linear shrinkage

The longitudinal shrinkage of the samples was measured according to [34] by recording the length of the samples before burning Lo, taking measurements after burning the samples L1, recording them, and calculating the linear shrinkage by applying the following formula. Table 6 shows the linear shrinkage test results.

$$L.S=\frac{L0-L1}{Lo}x100 \ldots(3)$$

The results of the linear shrinkage test of the samples after firing showed that increasing the percentage of barium carbonate ($BaCO_3$) added to the mixture led to a decrease in the percentage of linear shrinkage in brick samples made using Al-Angor clay and an increase in the percentage of expansion of brick samples made using Al-Nahrawan clay due to the release of carbon dioxide gas resulting from the decomposition of the barium carbonate.

$$BaCO3 + SO3 \rightarrow BaSO4 + CO2\uparrow \ldots.(2)$$

Note: The positive numbers indicate shrinkage, and the negative numbers indicate an increase in the length of the samples.

Efflorescence test

The most important test that was conducted on the laboratory brick samples was the efflorescence test, where efflorescence in the samples was examined according to [35]. The efflorescence of clay bricks was measured via the efflorescence test by partially immersing the samples in distilled water for 7 days (Fig. 3). At the end of this period, these samples were allowed to dry. Thereafter, they were examined to confirm efflorescence, compared with the samples that were not immersed, and classified as efflorescence or free efflorescence by the examiner. Shallow watertight trays, pots, or basins are made of corrosion-resistant materials or materials that do not react with soluble salts when in contact with distilled water and are at least one inch deep or 25.4 mm [2]. The test results are expressed in the following scales according to Iraqi Standard Specifications No. 25 (IQS,1988) [36]:

1- Nil efflorescence is when no efflorescence appears on the brick.
2- Light efflorescence is when the efflorescence is a light layer of salt that does not exceed 10% of the surface area of the brick.
3- Medium is when the salt layer is within 10%–50% of the surface area of the brick.
4- Dense is when the salt layer is more than 50% of the surface area of the brick. This is not accompanied with crumbling or peeling on the surface of the bricks.
5-Very dense is when the salt layer covers more than 50% of the surface area of the brick and is accompanied with crumbling or peeling of the brick surface or both [2]. The result of the efflorescence test is illustrated in Table 6.

Figs. 4 and 5 illustrate the results of efflorescence treatment on the brick samples.

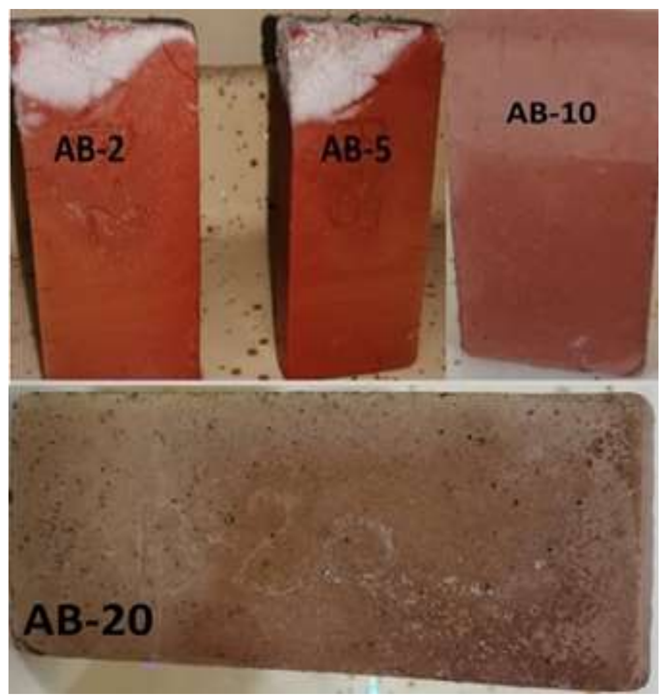


Figure. 3 Efflorescence test.

The results of the efflorescence test in Table 6 and Figs. 4 and 5 showed that the use of barium carbonate with the Al-Angor clay gave better results than the clay of the Al-Nahrawan region, which needs larger proportions of barium carbonate because the salt levels in the clay of the Al-Nahrawan region are higher than the salt levels in the clay of the Al-Angor region. The clays differ in the percentage of inhibitory materials they need for the purpose of efflorescence treatment because they contain different ratios of salt.

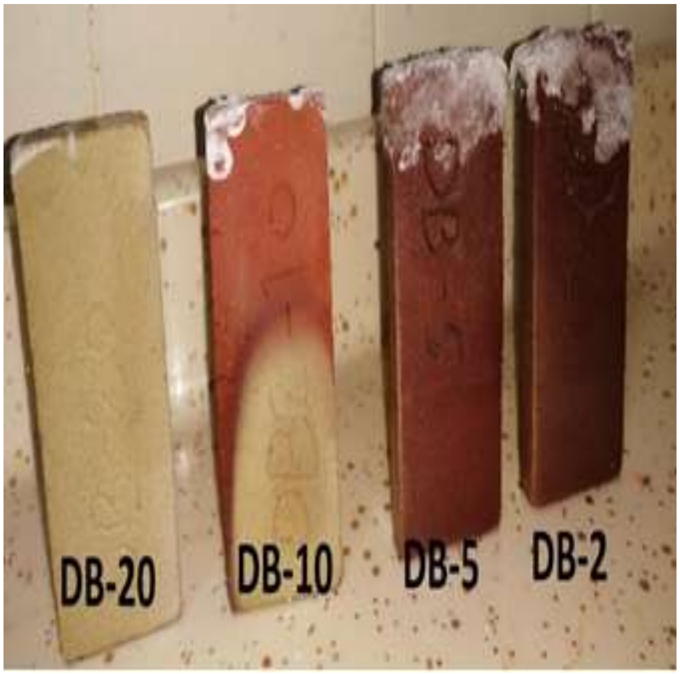


Figure. 4. Brick samples of Al-Nahrawan with different ratios of $BaCO_3$.

Figure. 5. Brick samples of Al-Angor with different ratios of $BaCO_3$.

Table 6: Results of the test.

No. Sample	Test results			
	A.P%	W.A%	L.S%	Efflorescence
AB-0	34.3	22.2	1.6	Medium
AB-2	34.6	22.3	1.6	Medium
AB-5	35.5	22.8	1.4	Medium
AB-10	35.8	22.8	1.1	Nil
AB-20	36.6	23.2	0.9	Nil
DB-0	25.7	14.18	-0.8	Medium
DB-2	26	14.4	-0.8	Medium
DB-5	26.7	15.4	-0.9	Medium
DB-10	30.3	16.4	-1	Light
DB-20	39.0	24.1	-1.6	Nil

7. XRD

XRD analysis was performed on samples of burned bricks after grinding, and the results were obtained using the MATCH phases analysis program. The results in Figs. 6–9 show the mineral phases that were formed after burning the bricks. XRD analysis was carried out on the samples of bricks made from the clay of the Al-Nahrawan area without adding $BaCO_3$ (Fig. 6) and with 20% of $BaCO_3$ (Fig. 7). Moreover, XRD analysis was carried out on the samples of bricks made from the clay of the Al-Angor area without adding $BaCO_3$ (Fig. 8) and with 10% of $BaCO_3$ (Fig. 9). A comparison of Fig .6 with Fig. 7 and of Fig. 8 with Fig. 9 revealed that the formation of $BaSO_4$, an insoluble inert compound, resulted from adding $BaCO_3$. The reaction of barium carbonate with SO_3 is expressed as follows:

$Ba^{+2} + SO_4^{-2} \rightarrow BaSO_4$ ……(4) [17]

Or

$Na_2SO_4 + BaCO_3 \rightarrow Na_2CO3 + BaSO_4$ …....(5) [16].

This is the desired result of adding $BaCO_3$.

The need to use high percentages of barium carbonate is due to the high percentage of salts especially in the Al-Nahrawan region clay. In the study conducted by Marku et al. in 2012, they only added 3% of barium carbonate to the clay, which was deemed insufficient due to the high percentage of salts in the clay used. XRD analysis revealed that barium sulfate in Al-Nahrawan brick samples that contained 20% of $BaCO_3$ minerals formed wollastonite, diopside, and graphite. By contrast, diopside, quartz, and lime formed in the Nahrawan clay samples that did not contain barium carbonate.

In the samples of the Al-Angor bricks that contained 10% barium carbonate, diopside and barium sulfate formed.

In the bricks of the Al-Angor area that did not contain any additives, the minerals quartz, leucite, and diopside formed.

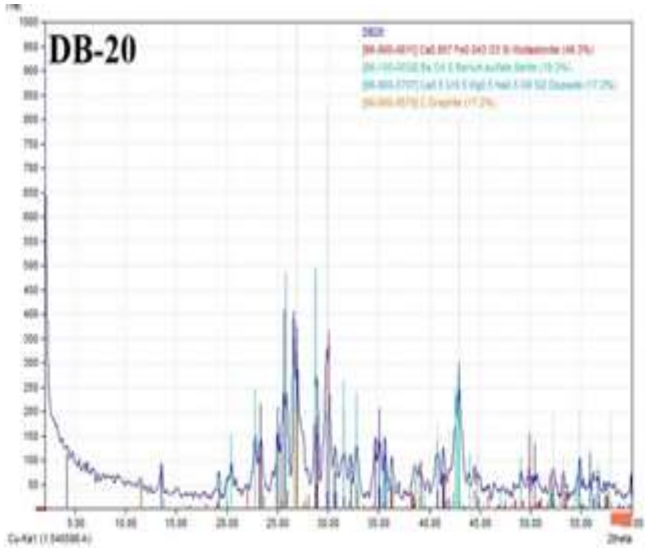


Figure .6. XRD pattern analysis of Al-Nahrawan bricks without adding $BaCO_3$.

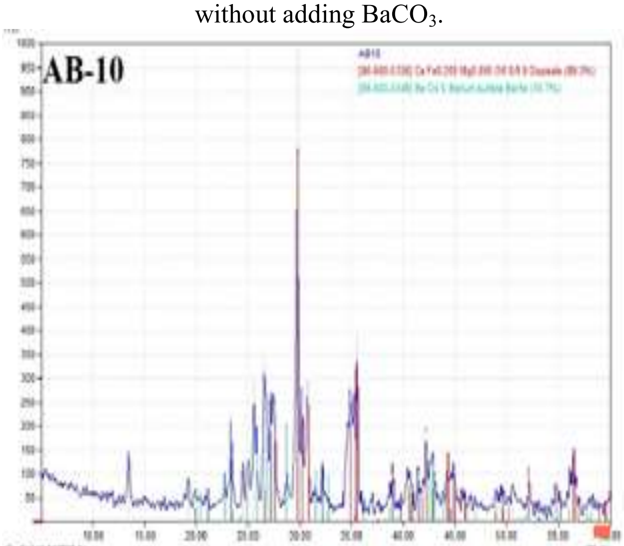


Figure. 7. XRD pattern analysis of Al-Nahrawan bricks with 20% of $BaCO_3$.

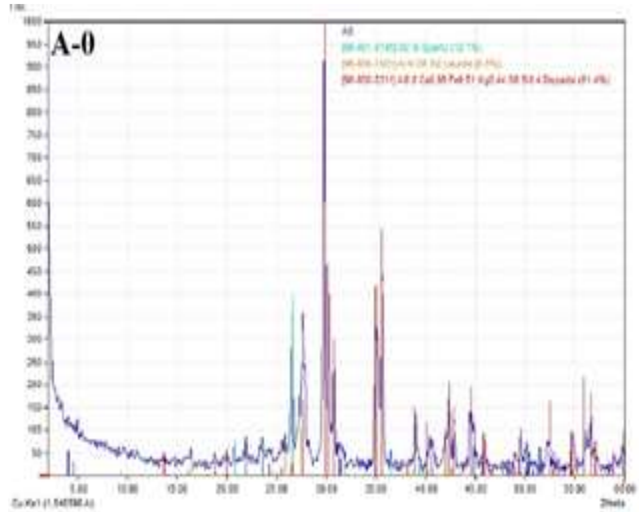


Figure. 8. XRD pattern analysis of Al-Angor bricks without adding $BaCO_3$.

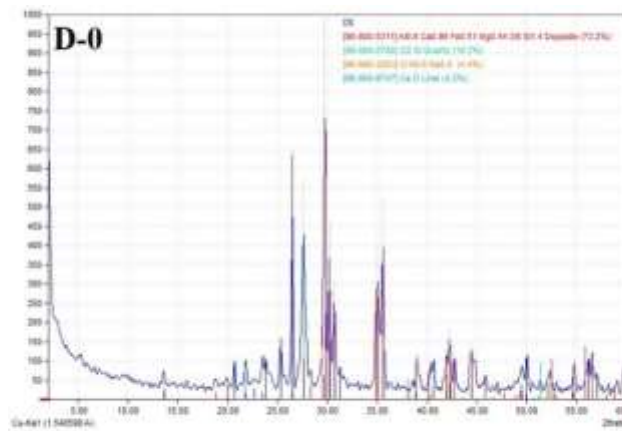


Figure. 9. XRD pattern analysis of Al-Angor bricks with 10% of $BaCO_3$8.

Conclusion

8. Conclusion

In this work, a chemical method using barium carbonate was used for the treatment of efflorescence in clay bricks to convert soluble salts into inert salts. Several tests were performed on brick samples, and barium carbonate was added in different proportions and burned at 950 °C. From these tests, the following conclusions were drawn.

- The addition of barium carbonate to clays that contained soluble salts led to a reduction in efflorescence. The percentage of reduction of efflorescence in bricks after the addition of barium carbonate was dependent on the percentage of salts in the raw material, as the bricks that contained high percentages of soluble salts needed to be added with large percentages of barium carbonate compound.

- Clays that contained a small percentage of salts could be treated by adding small percentages of barium carbonate compound, which works to inhibit these salts and prevent them from dissolving by forming inert compounds such as barium sulfate.

Therefore, the clays of the Al-Angor region need to be added with a lower percentage of barium carbonate compound for the treatment of efflorescence because they contain less salts than the clays of Al-Nahrawan.

- The addition of barium carbonate led to an increase in the percentage of pores and water absorption because it led to the formation of pores due to the release of carbon dioxide gas.
- The addition of barium carbonate reduced the percentage of linear shrinkage of the samples after firing.

ACKNOWLEDGMENT

The authors would like to thank to the Ministry of Science and Technology and to the German laboratory in the Geology department, College of Science, University of Baghdad, for their commendable contributions to chemical analysis (XRF). The authors are also grateful to the University of Technology - Iraq for their great help in conducting XRD analysis, and the authors would like to express their gratitude to all the staff of the Journal.

Reference:

[1] J. Chwast, J. Todorović, H. Janssen, and J. Elsen, "Gypsum efflorescence on clay brick masonry: Field survey and literature study," *Constr Build Mater*, vol. 85, pp. 57–64, Jun. 2015, doi: 10.1016/j.conbuildmat.2015.02.094.

[2] S. Nazhan, Y. Allbadi, Z. Hataf Naji, A. A. Abdullah Al-Karakchi, and J. Adeeb, "New Technique and Manufactured Device for Determining the Efflorescence of Clay Brick Masonry," *Adv Civ Eng Mater*, vol. 12, no. 1, pp. 114–126, Apr. 2023, doi: 10.1520/ACEM20220040.

[3] D. Marín-García, D. Bienvenido-Huertas, M. J. Carretero-Ayuso, and S. Della Torre, "Deep learning model for automated detection of efflorescence and its possible treatment in images of brick facades," *Autom Constr*, vol. 145, Jan. 2023, doi: 10.1016/j.autcon.2022.104658.

[4] H. Brocken and T. G. Nijland, "White efflorescence on brick masonry and concrete masonry blocks, with special emphasis on sulfate efflorescence on concrete blocks," *Constr Build Mater*, vol. 18, no. 5, pp. 315–323, Jun. 2004, doi: 10.1016/j.conbuildmat.2004.02.004.

[5] H. T. Nhabih, K. K. Arat, and A. S. Haidi, "Methods of Processing Efflorescence of Clay Brick," *International Journal of Scientific Engineering and Science*, vol. 3, no. 12, pp. 48–56, 2020, [Online]. Available: http://ijses.com/

[6] W. E. Brownell, "Efflorescence Resulting from Sulfates in Clay Raw Materials," *Journal of the American ceramic Society*, vol. 41, no. 8, pp. 310–314, 1958.

[7] Brick Industry Association, "Efflorescence-Causes and Prevention," 2019. Accessed: Mar. 31, 2024. [Online]. Available: www.gobrick.com

[8] -ard E Marbaker, E. Virginia Benedict, A. Edibr Vol, and B. W. E, "THE JOURNAL OF THE AMERICAN CERAMIC SOCIETY FUNDAMENTAL FACTORS INFLUENCING EFFLORESCENCE OF CLAY PRODUCTS*," 1949. doi: https://doi.org/10.1111/j.1151-2916.1949.tb18917.x.

[9] A. Baptista, A. Carneiro, G. Parsekian, and F. Fonseca, "A Proposed Test to Evaluate Efflorescence Potential of Ceramic Blocks," in *RILEM Bookseries*, vol. 18, Springer Netherlands, 2019, pp. 532–539. doi: 10.1007/978-3-319-99441-3_57.

[10] J. I. Davison, "Study of efflorescence in clay bricks," 1965, doi: 10.4224/20337960.

[11] H. Janssen, J. Chwast, and J. Elsen, "Gypsum efflorescence on clay brick masonry: Analysis of potential efflorescence origins," *J Build Phys*, vol. 44, no. 1, pp. 37–66, Jul. 2020, doi: 10.1177/1744259119896083.

[12] E. Catelli, F.-G. Bănică, and A. Bănică, "Salt efflorescence in historic wooden buildings," *Herit*

Sci, vol. 4, no. 1, p. 31, Dec. 2016, doi: 10.1186/s40494-016-0099-9.

[13] M. R. Karim, M. H. Islam, and S. Y. Morshed, *Influence of salt efflorescence on rendering mortar of brick masonry walls*. 2016.

[14] W. E. Brownell, "Application of New Techniques to the Solution of an Efflorescence Problem," *Journal of the American Ceramic Society*, vol. 33, no. 12, pp. 360–363, 1950.

[15] D. A. Brosnan, J. C. Frederic, and J. P. Sanders, "Method for processing clay ceramic materials," 2003. [Online]. Available: https://tigerprints.clemson.edu/clemson_patents/174

[16] J. Marku, K. Vaso, J. Marku, and K. Vaso, "Peroxide treatment in efflorescence-preventing," *Journal of Engineering & Processing Management*|, vol. 7, no. 1, 2012, doi: 10.7251/JEPM1204007M.

[17] S. Jindasuwan, P. Chakornnipit, and S. Supothina, "Influences of inhibitor and firing temperature on efflorescence reduction of clay products," in *Key Engineering Materials*, Trans Tech Publications Ltd, 2015, pp. 111–115. doi: 10.4028/www.scientific.net/KEM.659.111.

[18] T. Raghunathan, "A case study on Application of Geopolymer in Treatment of Efflorescence affected Masonry Walls," *Journal of Engineering Research and Application www.ijera.com*, vol. 9, pp. 38–41, 2019, doi: 10.9790/9622-0901013841.

[19] R. Kresse *et al.*, "Barium and Barium Compounds," in *Ullmann's Encyclopedia of Industrial Chemistry*, Wiley, 2007. doi: 10.1002/14356007.a03_325.pub2.

[20] S. Jonathan, "scumming from bricks without barium carbonate - is it possible reduce," 2020, Accessed: Apr. 03, 2024. [Online]. Available: https://blog.biokeram.com/reduce-scumming-from-bricks-without-barium-carbonate-is-it-possible

[21] A. Andrés, M. C. Díaz, A. Coz, M. J. Abellán, and J. R. Viguri, "Physico-chemical characterisation of bricks all through the manufacture process in relation to efflorescence salts," *J Eur Ceram Soc*, vol. 29, no. 10, pp. 1869–1877, Jul. 2009, doi: 10.1016/j.jeurceramsoc.2008.11.015.

[22] ASTM D4318, "Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils," 2000. doi: 10.1520/D4318-17E01.

[23] Braja. M. Das, *Advanced Soil Mechanics Fifth Edition, e-book*, Fifth Edition. published by Taylor and Francis group, London, New York, 2019.

[24] ASTM D 422-63, "Standard Test Method for Particle – Size Analysis of Soils. ASTM International, West Conshohocken, PA, USA.," 2022.

[25] F. M. Fernandes, P. B. Lourenço, and F. Castro, "Ancient Clay Bricks: Manufacture and Properties," in *Materials, Technologies and Practice in Historic Heritage Structures*, Springer Netherlands, 2010, pp. 29–48. doi: 10.1007/978-90-481-2684-2_3.

[26] J. Perold, "Ceramic parameters in the financial evaluation of brick clay deposits, with reference to two South African examples," 2006. Accessed: Apr. 05, 2024. [Online]. Available: https://www.proquest.com/openview/aeb3b8a912649fbf23a3ca8738b43522

[27] R. Pudasain, "How to Reduce Pollutant from Brickmaking Industrie," 2023, doi: DOI: 10.13140/RG.2.2.23011.22564.

[28] R. E. Hughes and B. L. Bargh, "The Weathering of Brick: Causes, Assessment and Measurement," 1982. Accessed: Apr. 05, 2024. [Online]. Available: https://pubs.usgs.gov/of/1983/0272/report.pdf

[29] E. Escalera, "Characterization and Preparation of Lightweight Silica Based Ceramics for Building Applications," 2015, pp. 12–13. Accessed: Apr. 05, 2024. [Online]. Available: https://ltu.diva-portal.org/smash/record.jsf?pid=diva2%3A999051&dswid=-8567

[30] J. O. Akinyele, U. T. Igba, T. O. Ayorinde, and P. O. Jimoh, "Structural efficiency of burnt clay bricks containing waste crushed glass and," 2020, doi: https://doi.org/10.1016/j.cscm.2020.e00404.

[31] F. M. Fernandes, P. B. Lourenço, and F. Castro, "Ancient Clay Bricks: Manufacture and

Properties," in *Materials, Technologies and Practice in Historic Heritage Structures*, Springer Netherlands, 2010, pp. 29–48. doi: 10.1007/978-90-481-2684-2_3.
[32] ASTM- C373 – 88, "Designation: C373-88 Standard Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products 1," 2006, doi: 10.1520/C0373-88R06.
[33] H. M. Ali, "Production of Lightweight Thermal Insulating Clay Bricks using Papyrus," *Polytechnic Journal*, vol. 9, no. 1, pp. 74–81, Jun. 2019, doi: 10.25156/ptj.v9n1y2019.pp74-81.
[34] ASTM C326-82, *Standard Test Method for Drying and Firing Shrinkages of Ceramic Whiteware Clays*. 2017. doi: 10.1520/C0326-82R97E01.
[35] I. R. Chin and B. Behie, "Efflorescence: Evaluation of Published Test Methods for Brick and Efforts to Develop a Masonry Assembly Test Method," in *Symposium on Masonry*, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959: ASTM International, 2010, pp. 3-3–11. doi: 10.1520/STP49100S.
[36] M. O. Khalaf and B. M. A. Issa, "Determining the physical and chemical properties of the soils in babylon and al-qadisiyyah for manufacturing clay bricks," *Iraqi Geological Journal*, vol. 54, no. 2, pp. 100–116, Sep. 2021, doi: 10.46717/IGJ.54.2C.10MS-2021-09-29.

معالجة التزهر في الطابوق الطيني كيميائيا خلال عملية التصنيع

محمود حميد السلماني[1*] محمد احمد النعيمي[1]

قسم الجيولوجيا التطبيقية، كلية العلوم، جامعة الانبار، العراق
Email: mah22s4004@uoanbar.edu.iq

الخلاصة:

التزهر عبارة عن ترسبات بيضاء من الأملاح التي تترسب على واجهات المباني المشيدة باستخدام الطابوق الطيني، وينتج التزهر عن تبخر المياه المحملة بالأملاح الذائبة. وعادةً ما يكون مصدر هذه الأملاح من المادة الخام نفسها. يعد التزهر مشكلة جمالية بالإضافة إلى تأثيرها على متانة وحدات البناء. تهدف هذه الدراسة إلى معالجة هذه الظاهرة كيميائياً أثناء عملية التصنيع بإضافة المواد المثبطة. استُخدم مركب كربونات الباريوم ($BaCO_3$) كمثبط بنسب تتراوح بين 2–20% حيث تمت اضافته إلى طيان منطقة النهروان والعنكور على التوالي. تم حرق العينات عند درجة حرارة 950 درجة مئوية. ومن خلال إجراء اختبار التزهر على نماذج الطابوق وفقا للمعايير الامريكية ASTM C67-08. بالإضافة الى فحص امتصاص الماء وفحص المسامية ونسبة التقلص الطولي للنماذج بعد الحرق. أظهرت نتائج الاختبارات أن معدل التزهر انخفض مع زيادة نسبة المواد المثبطة المستخدمة وذلك لأن مركب كربونات الباريوم $BaCO_3$يؤدي إلى تحويل الأملاح القابلة للذوبان في الماء إلى أملاح غير قابلة للذوبان عن طريق تكوين مركب $BaSO_4$ الخامل. كما بينت الفحوصات زيادة طفيفة في نسبة المسامات وامتصاص الماء وزيادة في طول النماذج مع زيادة نسبة كربونات الباريوم.

الكلمات المفتاحية: الطابوق، التزهر، كربونات الباريوم، اطيان، النهروان.