



Usefulness Study of Mineralogical Treatment Enhancing the Quality of Claystone Used in The Nineveh Governorate for The Brick Industry

Rana A. Alhialy ^{1*} , Eman Q. Al-Ojar ² , Azealdeen S. Al-Jawadi ³ , Roaa M. ALshurafi ⁴ 

¹ College of Information Technology, University of Nineveh, Mosul, Iraq.

² Department of Construction and Projects, University of Nineveh, Mosul, Iraq.

³ College of Petroleum and Mining Engineering, University of Mosul, Mosul, Iraq.

⁴ Department of Geology, College of Science, University of Mosul, Mosul, Iraq.

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ABSTRACT

The purpose of this study is to find a method of treatment to improve the properties of claystone of the geological formations that are widespread in Northern Iraq by kaolinite addition. Washing clay with different ratios of water is another treatment to find the perfect quantity of water to dissolve the more percent of salts. More improvement was found by adding 10% kaolinite and the sample was classified as grade A according to Iraqis' specifications number 24 in 1988. The water absorption of the treated sample is 16.5%, the compressive strength is 34.9 MPa. and low inflorescence. The sample characteristics are good when adding 5% kaolinite, the water absorption of 19.5%, compressive strength of 30 MPa. and low inflorescence. While the non-treated sample has a 22.2% water absorption, 20.6 MPa of compressive strength, and medium inflorescence. The treatment by water dissolution is good to some extent, where dissolution increased to 3.2% when the water quantity increased to double of sample weight, and it increased to 4.8% when the water quantity increased five times as much. Eventually, the studied rock beds have good characteristics, very large reserves, and adequate to operate eight factories of 50.000 bricks/day for 50 years and it has 7.478.258 m³ of clay.

Correspondence:

Name: Rana A. Alhialy

Email:

rana.mahmmod@unoninevah.edu.iq

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دراسة اهمية المعالجة المعدنية لتحسين جودة الاطيان المستخدمة في صناعة الطابوق في محافظة نينوى

رنا عبد الاله الحيايلى^{1*}، ايمان قاسم الاوجار²، عزالدين صالح الجوادى³، رؤى محمد الشريفي⁴

¹كلية تكنولوجيا المعلومات، جامعة نينوى، الموصل، العراق.

²قسم الاعمار والمشاريع، جامعة نينوى، الموصل، العراق.

³كلية هندسة النفط والتعدين، جامعة الموصل، الموصل، العراق.

⁴قسم علوم الارض، كلية العلوم، جامعة الموصل، الموصل، العراق.

المخلص	معلومات الارشفة
الهدف من هذه الدراسة هو إيجاد معالجة لتحسين خصائص الاطيان للتكوينات الجيولوجية المنتشرة في شمال العراق وذلك بإضافة الكاؤولين. يعد غسل الاطيان بنسب مختلفة من الماء معالجة اخرى لإيجاد الكمية المثالية من الماء لإذابة النسبة الأكبر من الأملاح. ووجد العديد من التحسينات بإضافة 10% من مادة الكاؤولين وتم تصنيف العينة على أنها درجة A حسب المواصفات العراقية رقم 24 لعام 1988. ان نسبة امتصاص الماء للعينة المعالجة هي 16.5%، وقوة الانضغاط 34.9 ميكا باسكال مع انخفاض نسب التزهر. تكون خصائص العينة جيدة عند إضافة 5% كاولين، وامتصاص الماء بنسبة 19.5%، وقوة ضغط 30 ميغا باسكال وانخفاض التزهر. بينما العينة غير المعالجة تحتوي على 22.2% من امتصاص الماء، 20.6 ميكا باسكال من قوة الانضغاط، ونسبة تزهز متوسط. ان المعالجة بإذابة الماء جيدة بشكل ما، حيث زادت نسبة الذوبان إلى 3.2% عندما زادت كمية الماء إلى ضعف وزن العينة، وزادت إلى 4.8% عندما زادت كمية الماء خمسة أضعاف. أخيرًا، تتميز الطبقات الصخرية المدروسة بخصائص جيدة، ومخزون كبير جدًا، وكافية لتشغيل ثمانية مصانع بسعة 50.000 طابوقة / يوم لمدة 50 عامًا وفيها 7.478.258 متر مكعب من الطين.	تاريخ الاستلام: 24- يوليو -2023 تاريخ المراجعة: 24- سبتمبر -2023 تاريخ القبول: 09- اكتوبر -2023 تاريخ النشر الالكتروني: 01- يناير -2024 الكلمات المفتاحية: حجر طيني طابوق تكوينات جيولوجية شمال العراق محافظة نينوى المراسلة: الاسم: رنا عبد الاله الحيايلى Email: rana.mahmmod@unoninevah.edu.iq

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Introduction

Northern Iraq suffers from a scarcity of raw materials for brick. Clay is the primary raw material used in brick manufacturing (Ramakrishnan *et al.*, 2023). and it's a common natural mineral that has been utilized in a wide range of applications in different industries since the ancient era (Danish *et al.*, 2022). According to Mirza and Faraj (2017), many clays, including kaolinite and red clays, are used in construction because of their favorable firing behavior or because they produce favorable properties in burning utensils. Samples were taken from the beds of the Gercus Formation, Fatha Formation, and most beds of the Injana Formation.

Clays are used in the ceramic industry primarily because they contribute to the molding and drying properties of the wares being produced. And to find alternatives to the use of concrete blocks in construction and reconstruction, given the many and most significant disadvantages of these concrete blocks, such as being heavyweight, high cost, and that's after high cement prices, inadequate thermal insulation, etc. Several researchers work on

lightweight, inexpensive, and well-insulated alternatives, with a focus on the clays available in the region. Bricks are preferred for construction in Iraq because of their thermal insulation to resist extreme weather conditions in summer and winter and their energy savings. Bricks are commonly used in central and southern Iraq. Because of a lack of industry-friendly ores and the abundance of gravel and sand needed to make concrete blocks, their use in northern Iraq is constrained.

The Kurdistan Region and northern Iraq generally lack brick-making because of the scarcity of ore materials, such as clay in river deposits in the governorates of Nineveh and Duhok. There has been a need in recent years to identify valuable areas for this industry. Accordingly, the current study aims to improve the specification of clays found in geological formations around Mosul, such as the Fatha and Injana formations, because they contain a high percentage of carbonate, which makes them unsuitable for the brick industry.

In the Al-Rashidiya Area, the sediments from the Tigris River were used in the not-too-distant past, north of the city of Mosul, in limited manufacturing of bricks due to the lack of clay reserves available at that time. Also, the construction of the Mosul Dam prevented floods, thus preventing the sedimentation of mud on the narrow flood plains around the Tigris River and washing away the remains of the existing ancient mud. The idea of searching for the mud of the river's flood plains was excluded, and an alternative concept for beginning this study.

Around the validity of some clays in the geological formations exposed in the areas surrounding the city of Mosul, there are attempts made by some researchers. Clays from some beds of the Fatha, Injana, and Muqdadiya formations have been used as alternative sources in the manufacture of bricks (Al-Hakim, 1998; Maal'h *et al.*, 2007; Al-Jawadi *et al.*, 2021). Other research on clay minerals has received considerable attention in the past decades because of their attractive and distinctive properties such as natural abundance, high reactivity, and low cost (Wang, 2023).

The areas south of Mosul, such as Al Qusour, Qaraqosh, and Muhayurat, as well as Al-Nouran, were studied by Al-Jawadi *et al.* (2021), and it was discovered that they are not suitable for manufacturing bricks. New areas to the north of the city and within the Injana Formation were explored, with a focus on some clay beds that provided relatively good specifications (Al-Jawadi and Al-Naqib, 2017).

Geological Setting

Fatha Formation is exposed in the core of Qand Anticline, which consists of an equivalent sequence of carbonate, evaporate, marl, and clay stone. The Injana Formation constitutes the largest part of the rock discoveries in the Qand Anticline and reaches a thickness of 374 meters near the village of Dughata in the southern limb of the anticline. The age of the formation is the Late Miocene (Bellen *et al.*, 1959). The Injana Formation generally consists of beds of sandstone of gray, brown, and red color, which have a very fine to coarse grain size, and beds of claystone with red, gray, and green colors.

The thickness of the beds studied is about 25 meters, tilted by 20–30 degrees towards the north for the northern limb of the anticline and by 30–35 degrees towards the south for the southern limb of the anticline. The clays of this bed are described as light brown, slightly reddish, with a greasy texture, hard, and very compact, and there is no indication of the presence of coarse carbonate rocks in them.

For the present research, Dwekhla Kaolinite of the Ga'ara Formation was used as a treatment material for the improvement of the claystone characteristics of the Injana Formation. The kaolin clay deposits are present at the top of the Ga'ara Formation, which is

about 80 km north of the city of Rutba in western Iraq. The Ga'ara Formation, one of the oldest formations exposed within the region, is located within the stable shelf and forms part of the Rutba area. The age of the formation is pre-carboniferous (Cytroky, 1973; Buday and Hak, 1980; Nader *et al.*, 1993). The formation consists of sandstone, mudstone, and ironstone successively (Al-Mallah, 1999). The clays of this formation are characterized by being of secondary origin, having been eroded, transported, and transformed into kaolin under acidic conditions and deposited in freshwater conditions (Zainal, 1980). Figure 1 shows the location from which the samples were taken.

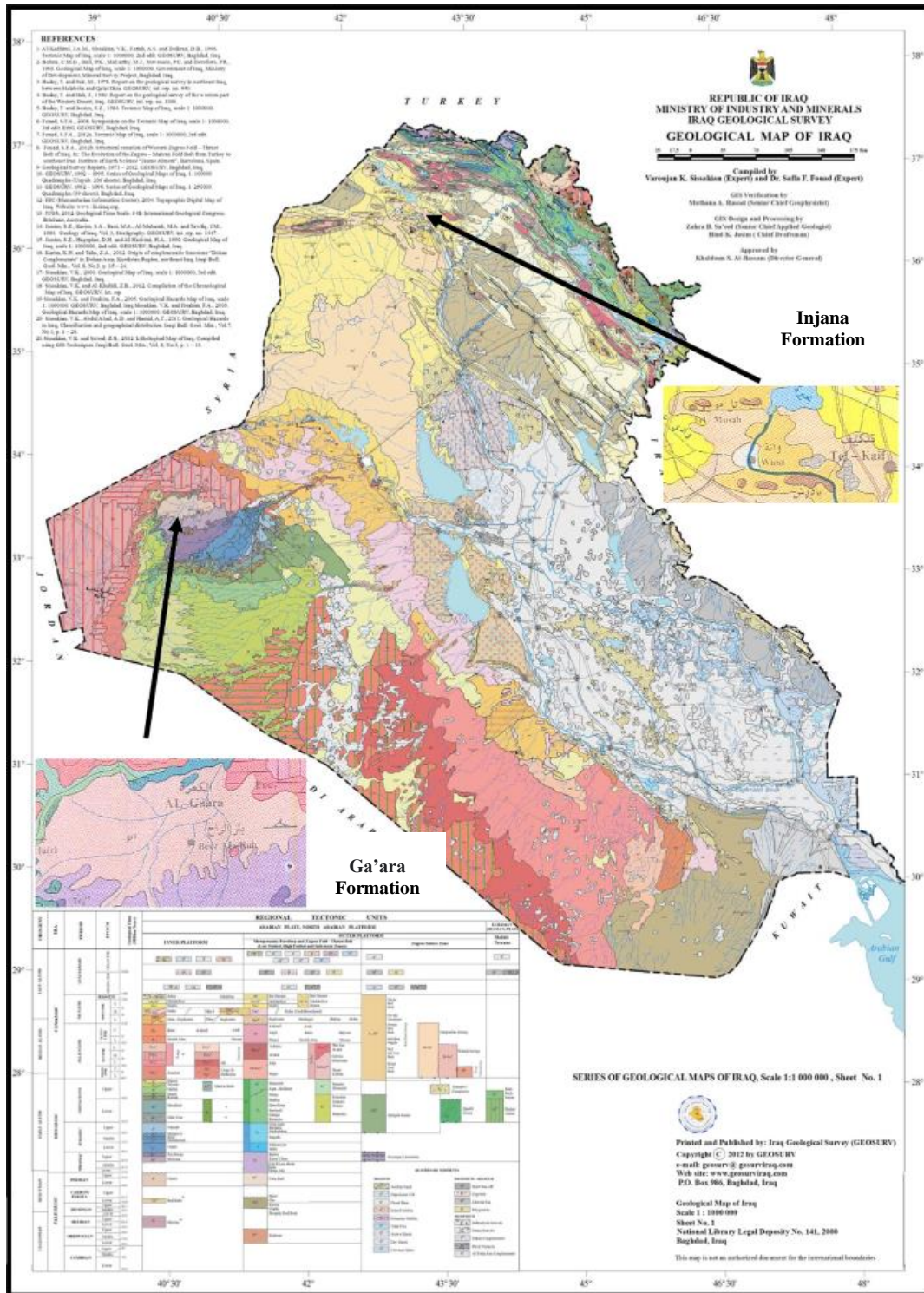


Fig. 1. Geological map of Iraq showing the Study area and location of Injana and Ga'ara formations modified after (Sissakian and Fouad, 2015).

Materials and Methods

Sampling

A quantity of Qand Clay (Q1) was extracted from geological beds with a thickness of at least 3 meters and a slight inclination to make a clay reserve suitable for industrial purposes in the manufacture of bricks. As for the Kaolinite Clays Dwekhleh sample, it was obtained from the General Authority for Geological Survey and Mining.

To show the distribution of particles in these clays (William, 2002), they were sieved with graduated sieves to a size limit of 0.074 mm. Then the volumetric gradient test of the finer materials was carried out using the Hydrometer Method.

The liquid and plastic limits were tested through the use of Atterberge limits experiments (Bodur and Yilmaz, 2000)., and then the plasticity index was extracted. The plastic limit was used as the moisture content needed to form the samples that were used for carrying out the engineering investigations. The clay samples were prepared by hand crushing with a hammer and finely milling with a Tema Ring Mill.

Treatments under study through soil tests on the clay sample (William, 2002), by adding water in different proportions, demonstrate the effect of the washing process with water on the percentage of salts and carbonates present in clays in a ratio of mud to water (1:1, 1:2, 1:3, 1:4, and 1:5), where 10 grams of the sample were weighed finely and water was added to it according to the proportions mentioned and left for 24 hours. The sediment was filtered with filter paper and dried at a temperature of 45–50 °C because some clay minerals were destroyed at a temperature higher than 60 °C, then weighed. The precipitate is subtracted from it, then the weight of the filter paper is subtracted, and the difference between the two weights before and after addition is calculated to obtain the percentage of dissolved salts in water, and kaolin is added to the sample.

The clays are under study at a rate of 5% and 10% to compare the bricks produced from the sample without addition with the bricks treated by adding kaolinite.

The chemical and mineralogical analysis conducted for the clay Qand and the kaolin used in the study was conducted by the General Directory of Geological Survey and Mining (Al-Qattan, 1990; Al-Jawadi and Al-Naqib, 2017).

The laboratories of the Dams and Water Resources Research Center were used to prepare brick samples in the first stage and to examine the engineering properties and validity of these materials in the manufacture of bricks in the second stage. The following is a brief explanation of both stages:

First Stage

1. Fermentation of prepared clays by adding water (17–20%) and leaving it in sealed bags for 24 hours, according to the clay brick tracking guide (CBA, 2002).
2. Formation of fermented clay samples according to Reeves *et al.* (2006) in the press device in two shapes: a rectangular shape with dimensions of 7.4 X 4 X 4.5 cm and tablets with a diameter of 5 cm and a thickness of 2.5 cm.
3. Dry the samples in an electrical oven at a temperature of 110 °C for 48 hours.
4. The dried material dimensions are measured (ASTM, 2002), then placed in the electric furnace and burned at a temperature of 950 °C, according to Villeda-Munoz *et al.* (2011), at an incremental rate of 200 °C per half hour with a maturation time of two hours.

5. Keep the samples in the furnace for 24 hours after turning them off until the temperature drops.
6. The lengths of the samples are measured after burning, and the longitudinal and volumetric shrinkage ratios are calculated (Fig. 2).

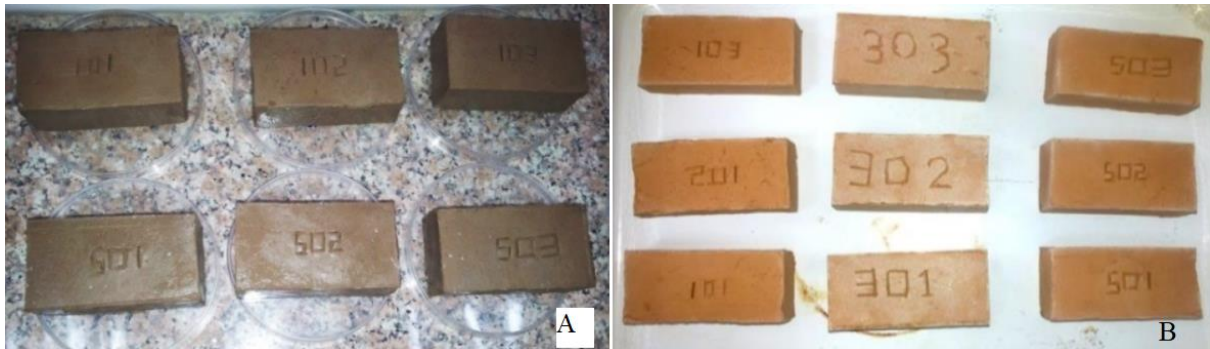


Fig. 2. Samples of formed blocks A- before drying, B- after burning.

Second Stage

1. Conducting physical tests on disk samples to find the total density as well as the percentage of water absorption according to Iraqi Standard 25 of 1988. The samples are dried in the drying oven at a temperature of 105 °C until their weight is stable, then they are cooled at room temperature in the Desiccator and weighed with a sensitive scale to calculate the dry weight immersed the sample is distilled in water at a temperature of 15–30 °C for 24 hours, then the surface is lifted and dried with a piece of cloth and weighed within three minutes of taking it out. The absorption percentage is calculated from the following equation:

$$\text{Absorption percentage after 24 hours} = [(b-a) / a] \times 100$$

Where:

A = weight of the dry form in grams.

B = the sample's weight in grams after being immersed in water for 24 hours.

2. Measurement of the compressive strength of rectangular samples and the tensile strength of disc samples in the laboratories of the College of Technology.
3. Measuring flowering, which is the percentage of salts that appear on the surface of the brick, by placing the brick vertically in a flat pot so that a quarter of its length is submerged in water and left for seven days. The water is dried in a pot and the rate of salt deposition is observed, and this percentage is theoretically classified with the naked eye into four types (none, light, medium, dense, and very dense) based on Iraqis' specifications (25) for the year 1988.

Results and Discussions

Grain Size and Atterberge Limits

The granular size of the Q1 sample in this clay bed ranges from 11% fine sand to 51% silt and then 38% clay, and this volumetric gradient is considered good in the manufacture of bricks and ceramics because it contains sandy and silty grains that are useful in forming the bonding material during the burning process, and its containment of a good percentage of the clay is useful in giving the necessary plasticity to the forming processes. When dropping it on a chart (Picard, 1971) and as shown in Fig. 3, it was found that the sample is located in the clay silty area.

Atterberge limits tests revealed that the values for the plasticity limit are 22%, the plasticity index is 12%, and the fluidity limit is 34%. As for kaolinite, its granular size is as follows: 5% fine sand, 30% silt, and 65% clay, and it is located within the silted area. The fluidity limit is 41%, as shown in Table 1, and according to the plasticity scheme (Fig. 3), the texture composition of primary materials (Picard, 1971). The values of the Atterberge limits were the plasticity limit of 21% and the plasticity index of 20% (Fig. 4).

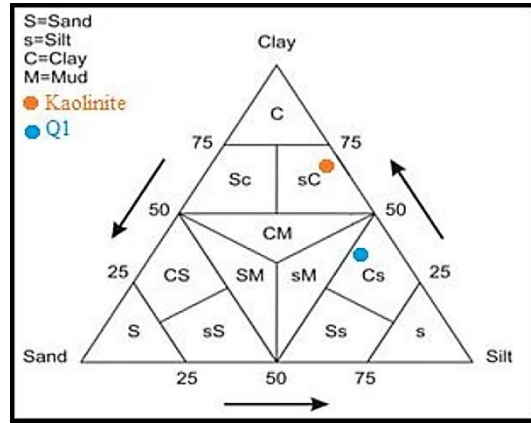


Fig. 3. A diagram showing the texture composition of primary materials (Picard, 1971)

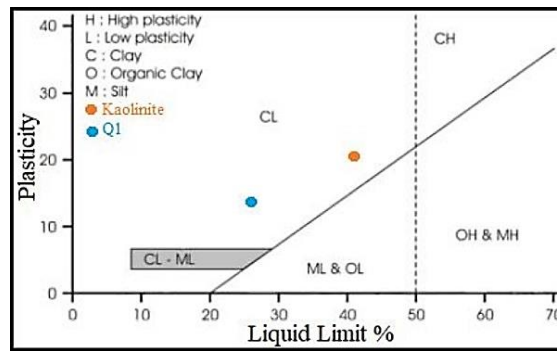


Fig. 4. The two samples fall within clays of low plasticity (Krynine and Judd, 1957).

Table 1: Atterberge Limits results with the classification of clay samples.

Samples	PL	LL	PI	Classification
Kaol.	21	41	20	Clay low plasticity
Q1	22	34	12	Clay low plasticity

Mineralogical Analysis

The mineral analysis of the clays of the region represented by sample Q1 shows that they contain the clay minerals represented by Montmorillonite, Elite, Chlorite, and Kaolinite, as well as non-clay minerals, namely Quartz, Plagioclase, and Calcite (Al-Jawadi and Al-Naqib, 2017).

Chemical Analysis

The results of the chemical analysis of the sediments are an evaluation in terms of their content of oxides and beneficial and harmful elements, which determine their suitability for the brick industry. The main analysis of sediments (Table 2) showed that silica is the main component in both clays, where it was in the Q1 form by 44.22%, and that part of this oxide is present in the form of free silica (quartz), as the results of the mineral analysis of the samples showed and that the presence of free silica in the clays It reduces plasticity and thus reduces shrinkage during drying and burning (Budnikov, 1964; Grimshaw, 1971). As for aluminum

oxide, its percentage in the Q1 form and kaolin was 10.86% and 26.5%, respectively. This oxide is considered one of the important oxides in ceramic industries, including the brick industry, due to its ability to give the product high strength and make it resistant to firing temperatures (Rayan, 1978). Kaolinite with a lower degree of defects needs higher activation energy for dehydroxylation (Wang, 2023).

It was observed that calcium oxide was present in a high percentage in the Q1 sample, which is why it was necessary to treat this percentage by adding kaolinite to reduce this percentage, as the increase of this oxide led to the predominance of calcium carbonate over other components in the samples, which appears as the mineral calcium here. CaCO_3 decomposes with heat to CaO and CO_2 , as the first reacts with water or moisture to form Ca(OH)_2 , which leads to swelling and cracking of the brick when obscured in water during the examination, and the second, the toxicity increases. As for the alkaline oxides K_2O and NaO , as well as iron oxide, their presence is considered important in accelerating the formation of the glass phase during firing at a temperature of $900\text{ }^\circ\text{C}$ (Serry *et al.*, 1985).

Table 2: Chemical analysis of clays in wt. %

Sample	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	L.O.I.
Q1	44.52	10.86	4.7	6.2	13.98	0.82	1.2	16.41
Kaolinite	56.20	26.50	3.40	0.17	1.85	0.45	0.55	0.90

The treatment of clays under study Q1 by washing them with different proportions of water gave good results, as shown in Table 3. So, washing the sample with water at a sample ratio of 2:1 led to the removal of 3.2% of the salts, and thus the percentage increased to reach 4.8% when the sample was washed with water at a ratio of 5:1. This reduced the inflorescence that occurs as a result of the precipitation of salts on the surface of bricks when exposed to humidity (Table 3).

Table 3: The percentage of dissolved salts when washing the Q1 form with different ratios of water

The ratio of the sample to the water	The ratio of dissolved salts
2:1	3.2
3:1	3.9
4:1	4.6
5:1	4.8

Assessment Tests

Shape and Color

The samples of bricks made from treating Q1 clays with kaolin were distinguished by their flat surfaces and the straightness of their edges, while a slight curvature was observed in the surfaces of the untreated sample, as well as cracks on its surface, while cracks were less in the sample added to kaolinite by 5%, and no cracks were noticed in the sample that was added to kaolinite at 10% (Fig. 5). It was also noticed that these cracks began to increase with the exposure of the sample to moisture in the air after several days of burning, and attributed This is due to the presence of quick lime CaO , which interacts with the water in the atmosphere, which leads to its transformation into Ca(OH)_2 , and this, in turn, leads to internal pressure in the structure of the bricks, which leads to the occurrence of cracks in the pattern as shown in (Fig. 5). As for the color of the samples General, it changed from reddish brown to yellowish brown after burning. Suggest (Wang *et al.*, 2023) that adding a quantity of a 20 wt.% of kaolinite, the variation in kaolinite defect proportions did not significantly change the color of the fired brick and Kaolinite added to that samples prevented strength loss at higher temperatures.

Volumetric and Longitudinal Shrinkage

Table 4 shows that the longitudinal shrinkage of the samples after drying increases in the Q1 form than in the 5% kaolin. added form and the highest percentage in the added form is 10% kaolin., and the reason for this may be due to the increase in the percentage of water added during the formation because the samples added to it Kaolin. needs a higher percentage of water because kaolin has a higher plasticity limit, and it was noticed that the longitudinal shrinkage values after burning for the untreated Q1 sample were negative, meaning that it had slight swelling, and this may be due to the release of gases resulting from the breakdown of carbonate, which led to the occurrence of swelling, while we observe in the treated samples that the longitudinal and volumetric shrinkage increases with increasing the kaolinite ratio. As well as for weight loss during burning before and after drying.

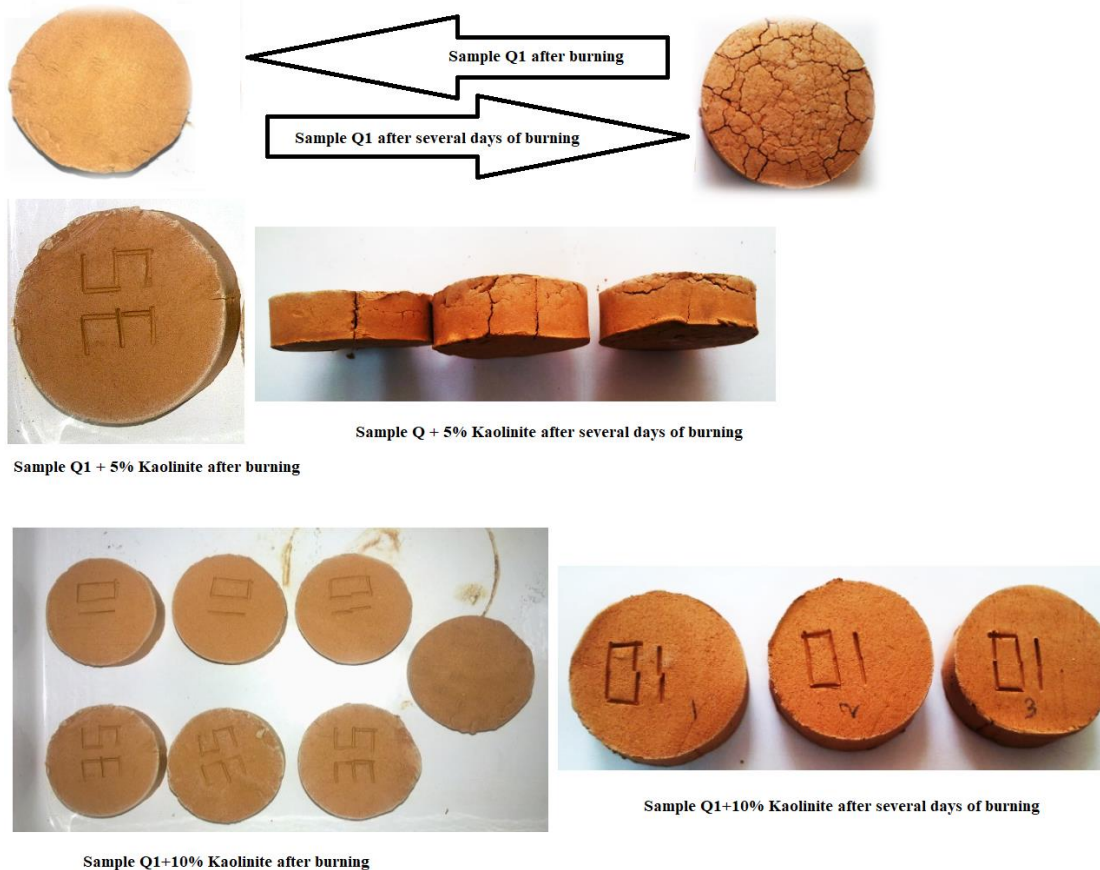


Fig. 5. Samples showing the effect of air humidity on the samples after several days of burning.

Water Absorption

The water absorption rate was 22.22% in the untreated sample, and it is higher than in the samples with kaolinite added by 5% and 10%, as the absorption values for them were 19.5%. and 16.5%, respectively (Table 4). In general, all values were within the Iraqi standard and were classified under Class A.

Table 4: The physical specifications of the produced bricks.

Sample	After drying			After burning			Absorption rate %	Inflorescence
	longitudinal shrinkage %	Size shrinkage %	Weight loss %	longitudinal shrinkage %	Size shrinkage %	Weight loss %		
Q1	3.45	3.45	16	1.7	16	28.6	22.22	Medium
Q1+5kaol.	6	23	18	7.3	16.9	30.6	19.5	Light
Q1+10kaol.	6.5	21	19.5	7.3	20.8	31.7	16.5	Light

Inflorescence

It was noticed that inflorescence appeared on average in the untreated form, i.e. it occurred within the class B according to the Iraqi standard for the classification of bricks, while the flowering was light in the samples treated with kaolinite, i.e. it fell within the category class A.

Compressive and Tensile Strength

It is noted from Table 5 that the values of compressive strength are higher than the percentage of kaolin added, and the reason may be due to less pore formation. This may be because the gases released from the sample upon burning are less, which gives a stronger structure to the sample and the same concerning the tensile strength. It is noted from Table 6, which includes the Iraqi specifications for bricks, as it can be seen that the compression strength ratio of the samples manufactured in this study falls within category class A.

Table 5: The compressive and tensile strength of bricks

Sample	Compressive strength Mpa	Tensile strength Mpa
Q1	22.22	---
Q1+5 kaol.	19.5	2.2
Q1+10 kaol.	16.5	2.5

Table 6: Iraqi Standard Specifications No. 25 of 1988 for bricks. The sample has the highest water absorption rate %, the minimum compressive strength (Mpa), and inflorescence.

Brick grade	Highest water absorption %	Min. compressive strength (Mpa)	Inflorescence
A	22	16	Light
B	26	11	Medium
C	28	7	High

Conclusions and Recommendations

1. The clays of the region contain carbonite and they are suitable for making bricks after processing them because carbonite is quick lime when burning, which hydrates when exposed to humidity in the air.
2. Kaolinite was added in various amounts to reduce the amount of carbonate in the clays, and the results were compared to made bricks without additives, it was found that the best results were obtained when adding 10% kaolinite and were within the A class according to the Iraqi specifications for the bricks.
3. When treating clays by washing with water to get rid of salts and reduce the percentage of inflorescence, it was found that the higher the percentage of washing water, the lower the salts percentage in the samples. This treatment is considered good from an economic point of view to get rid of the phenomenon of inflorescence.
4. Treated bricks by adding kaolinite are considered economically feasible compared to the prices of imported bricks. According to the type of bricks and the source of production.
5. It is recommended to conduct more comprehensive studies to improve the properties of clays in the region with other economically feasible and appropriate treatments in the brick industries that reduce the carbonate rates, as well as conducting studies on other areas to investigate clays containing fewer carbonate rates.
6. In terms of economy, the cost of one brick for treated clays by adding kaolinite is about 150 to 250 Iraqi dinars, while the selling price is 350 to 600 Iraqi dinars according to size, processing, and type of fuel.

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