

EVALUATION OF THE CLAYSTONES FOR CERAMIC INDUSTRY FROM BUSSIYA AREA, MUTHANNA GOVERNORATE

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Type of the Paper (Article)

Received: 01/ 08/ 2023

Accepted: 28/ 01/ 2025

Available online: 27/ 06/ 2025

Abstract

Clay is an important and basic raw material in the manufacture of ceramic products for building construction. Therefore, exploring clay deposits for previously unstudied areas and determining their suitability for ceramic materials is one of our priorities for the development and creation of job opportunities for citizens of the same region. The aim of the study is to evaluate the clays of the Bussiyya area in Al-Muthanna Governorate for building materials applications, and to prepare the models, the properties of clay and its thermal behavior must be studied, to identify its suitability for the ceramic industry and its behavior during forming and firing, as well as the effect of adding sand, feldspar, and the calcined clay. The components are dry-pressed with a pressure of 10 KN and fired at different temperatures (950, 1050, and 1100) °C. With a burning rate of 5°/min and a maturation time of one and a half hours, According to the laboratory results (physical and mechanical) showed the behavior of the clays when burning and the effect of all additions on them, the use of 25% sand has a clear effect on the properties of calcined and uncalcined clay, according to the temperatures that are appropriate for each application as mentioned in American Society for Testing and Materials specifications.

Keywords: Calcined Clay; Building Material; Sand; Feldspar.

1. Introduction

The importance of using clay in ceramic products (brick or tile) is one of the most visible components of the built environment. In addition to their functional use, they provide investment opportunities for the region (Joudi and Nassrullah, 2014). Choosing the appropriate raw material has a major role in determining the ceramic product and where it is used, depending on composition and various other technical characteristics (Mousharraf et al., 2011). Ceramic is usually a very hard, brittle material with a high melting point, low electrical and thermal conductivity, and good chemical and thermal stability (Barsoum, 2019).

Clay minerals vary in their geological and physical properties, so the preparation of structural clay products requires a careful study of the properties that match the requirements. In this research, appropriate building materials were collected from the Bussiyya area in Al-Muthanna Governorate, which is located in southwestern Iraq. The region has been subordinated to many economic studies and its ability to prepare building materials. The target layers are clays belonging to the Fatha (Nfayil) Formation (lower Miocene) within upper members (Ahmed, 2020). This layer consists of a group of different minerals, clay, and non-clay minerals, so it's necessary to study well the properties, well to reach the most appropriate uses, especially, if it contains the palygorskite clay mineral, which behaves differently when used to prepare ceramic materials compared to other clay minerals (Joudi and Nassrullah, 2014).

The main clay mineral in this research was Palygorskite which is also known as Attapulgite is magnesium-rich clay having a special laminated chain structure in crystals containing uncertain quantities of Na^+ , Ca^{+2} , Fe^{+3} , and Al^{+3} , and present in the shape of needles, fibers or fibrous clusters. It is naturally minding clay of 2:1 type of clay minerals structure that is two-silica (SiO_2) tetrahedron and one layer of alumina (Al_2O_3) octahedron, with basic chemical formula of $\text{Mg}_5\text{Si}_8\text{O}_{20}(\text{OH})_2(\text{OH}_2)_4 \cdot 4\text{H}_2\text{O}$ (Al-Bassam, 2000). The uses of palygorskite as an adhesive, in asphalt, ceramics, cosmetics, etc. (Galán, 1996).

This research aims to assess the suitability of the claystone of Bussiyya to prepare building products, determine the optimal conditions for this, and investigate the changes, using different proportions of sand and different temperature effects. The results of the tests can determine the appropriate condition for the preparation of ceramic material.

1.1. Previous Work

Different materials and methods have been used to prepare ceramic building materials, depending upon the chemical compositions of the raw materials and processing conditions. Al-Kaabi et al. (2022), Studied the thermal treatments of the raw clay from the Fatha (Nfayil) Formation in the Bussiyya area and its suitability for the construction industry. It recommended studying the properties of Bussiyya clay and adding other sources to determine the appropriate industry.

There is also a previous study using attapulgite clay from the Dogma Formation in the western desert of Iraq, to prepare ceramic products, as an additive to Meta kaolin and feldspar. It has shown superior physical and mechanical properties when fired at 1150 °C (Joudi et al., 2021).

(Al-Saed, 2016), studied the effect of firing temperature of some properties for clay brick containing attapulgite, and used attapulgite as supplementary material to improve the properties of building material. The result indicated that the best proportion was 20% and showed a significant decrease in the longitudinal shrinkage, increased reached 9.1% in compression, and decreased in efflorescence when fired at 750 °C.

The great reserve of stratigraphic mineral clays (e.g., Kaolin, Attapulgite, and montmorillonite) around the Bussiyya town appeared to need further study for construction materials industries

(Ahmed, 2020). Therefore, this site was chosen to study its suitability for the construction industry.

2. Materials and Methods

2.1. Raw claystone

The raw claystone was sampled from the Fatha (Nfayil) Formation in the Bussiyya area. The coordinates were 624765 E and 3367286 N, UTM projection zone 38 (Figure 1).

Twenty kilograms of claystone were quartered and divided to obtain representative samples. Clay was crushed and ground until get particle size less than 500 μm . The sample was used in two cases, raw clay, and calcined clay at 750 $^{\circ}\text{C}$, with a heating rate of 10 $^{\circ}\text{C}/\text{min}$. The representative samples (raw clay and calcined clay) were sent to a laboratory for analysis, and 23 were done at central laboratories at the Iraq Geological Survey in Baghdad, Iraq.

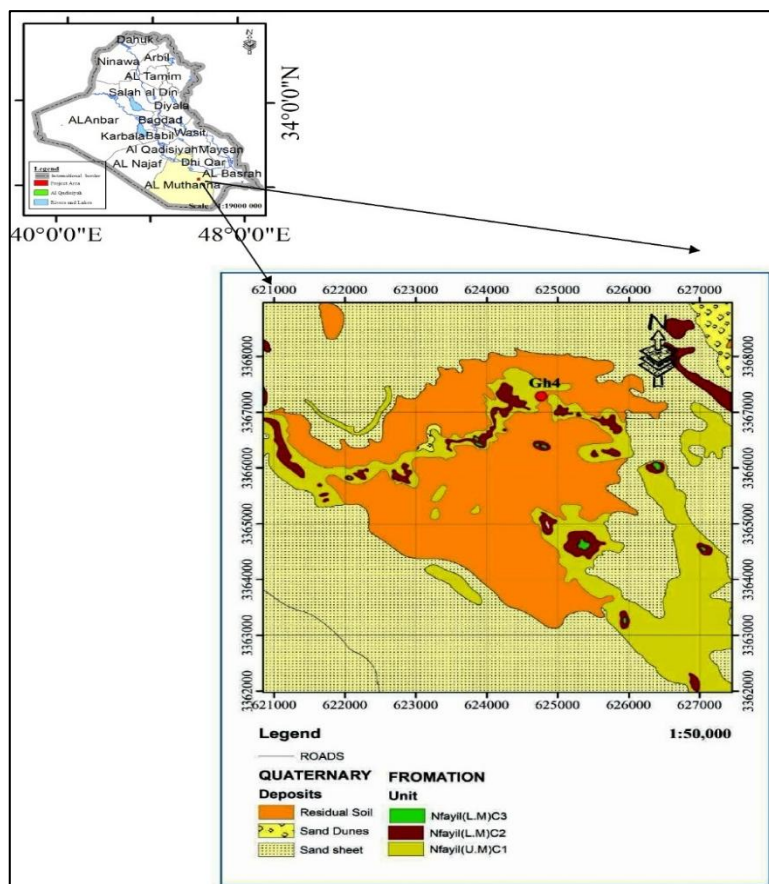


Figure 1. Location map of the project in the Bussiyya area (Zaini et al., 2014).

2.2. Sand

The sand was sampled from the deposit location in Al-Najaf Governorate, 25 Km northwest of Al-Najaf city. Ten kilograms of sand were quartered and divided to obtain representative samples. The sand was milled and ground until it reached particle size – 75 μm .

2.3. Feldspar

It was crushed to a particle size of less than 1 mm, then ground by a ball mill to pass (75) μm . Feldspar deposit is located in the northeast of Al-Najaf Governorate center about 25 Km.

2.4. Preparation of Samples

Six groups of samples (B, BS, C, CS, BSF, CSF) were prepared for studying the variable percentage of different raw materials (Bussiyya clay and calcined Bussiyya clay), different ratios of sand (0, 10, 15, 20, 25 and 30) wt.% were mixed with raw materials and adding feldspar (5 wt.%) in some cases. The mixtures of materials were thoroughly homogeneous as shown in Table (1). The samples were prepared by press forming with pressure (100 Kg/cm²) using a cylindrical Mold with dimensions (3.5 \times 10) cm. The samples were dried in an oven at 110 °C after being kept at room temperature for 48 hours, then fired at different temperatures (950, 1050, and 1100) °C in a muffle furnace with a heating rate of 5 °C/min and soaking time 1.5 hour.

Table 1. Ratios of different raw materials used in preparing the tested samples.

Sample	Claystone %	Sand %	Calcined claystone %	Feldspar %
B	100		-	-
BS1	90	10	-	-
BS2	85	15	-	-
BS3	80	20	-	-
BS4	75	25	-	-
BS5	70	30	-	-
BSF	85	10	-	5
C	-	-	100	-
CS1	-	10	90	-
CS2	-	15	85	-
CS3	-	20	80	-
CS4	-	25	75	-
CS5	-	30	70	-
CSF	-	10	85	5

2.5. Measurement of physical and mechanical properties

The samples were tested for linear shrinkage, bulk density, porosity, and water absorption according to the American Society for Testing and Materials (ASTM C67-05).

2.6. Linear Shrinkage

$$L \text{ Sh. \%} = (L_d - L_f) / L_d * 100 \quad (1)$$

Where; L_d = length of the dried specimen in centimeters; L_f = length of the fired specimen in centimeters.

2.7. Bulk density

The bulk density provides a general indication of the product quality.

$$\text{Bulk density} = A/V \quad (2)$$

Where; A: The dry weight of the samples measured and V: total volume.

2.8. Water Absorption

The water absorption (A), can be calculated as follows:

$$A\% = (W_2 - W_1) / W_1 * 100 \quad (3)$$

The samples were immersed in tap water for 24 hours, after that each sample was weighed separately after immersion in cold water. Then, placed in boiling water for 5 hours, then taken out and cooled at room temperature. Each sample was weighed 5 min after removing it from the bath.

$$A\% = (W_3 - W_1) / W_1 * 100 \quad (4)$$

2.9. Saturation coefficient

The saturation coefficient for each sample can be calculated as in the equation;

$$S_t = (W_2 - W_1) / (W_3 - W_1) \quad (5)$$

Where: W_1 = Weight of the dried sample, W_2 = Immersing it for 24 hrs. In cold water, and W_3 = Weight of the sample after boiling.

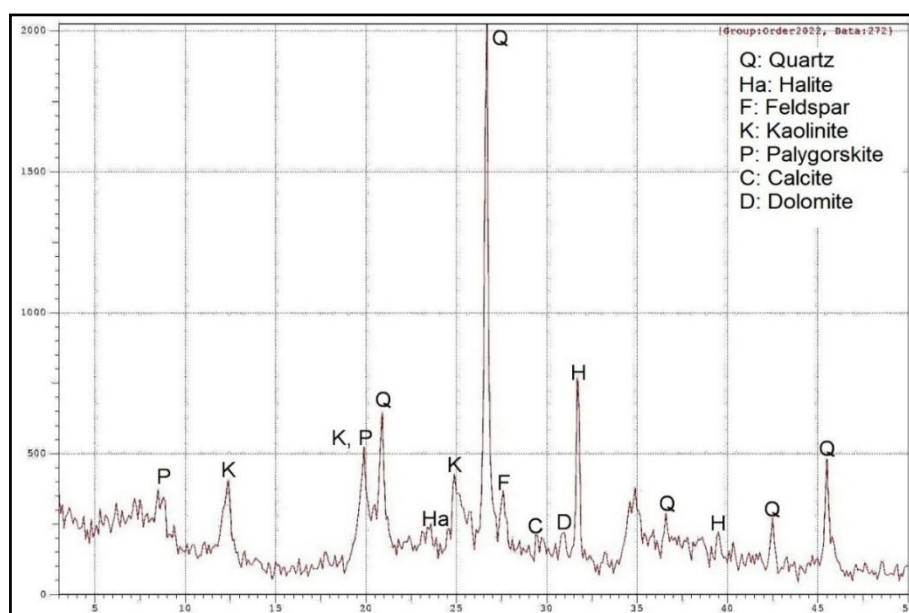
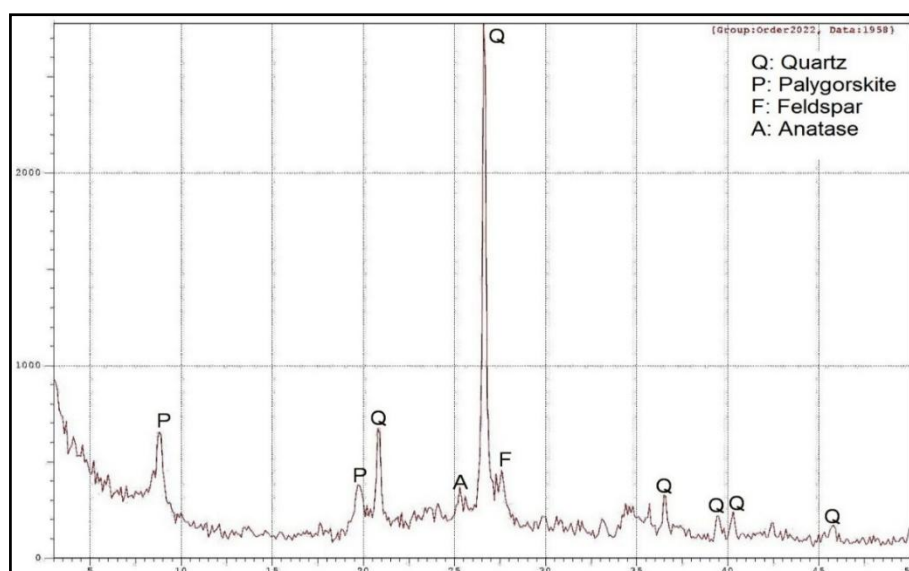
3. Results and Discussion

3.1. Characterization of raw materials

The representative samples were sent to the chemical wet analysis, were digested in concentrated acid, and then filtered. The filtrate was used to measure the concentration of each element (SiO_2 , Al_2O_3 , CaO , MgO , Fe_2O_3 , SO_3 , Na_2O , K_2O , LOI), as shown in Table (2) and mineral identification by XRD, The powder of each sample was put in sample holder and then measured in XRD 7000(ShimadzuJapan) using Cu as XRD source in the 2θ range of $5 - 50^\circ$, as shown in Figures (2, 3, 4, and 5).

Table 2. Chemical analyses of raw materials.

Raw materials	SiO ₂ %	Fe ₂ O ₃ %	Al ₂ O ₃ %	CaO %	MgO %	SO ₃ %	K ₂ O %	Na ₂ O %	Cl %	L.O.I %
Claystone	47.66	5.85	18.70	3.92	3.80	0.760	2.40	2.70	4.69	13.20
Calcined clay	55.76	7	18.99	4.88	4.50	0.08	2.64	3.18	0.14	0.73
Sand	92.08	0.30	1.39	2.24	0.27	1.33	0.36	0.36	0.04	1.54
Feldspar- rich sand	88.60	0.70	5.35	0.28	0.14	<0.07	1.28	1.05	-	0.61

**Figure 2.** XRD pattern of the investigated claystone sample.**Figure 3.** XRD pattern of the investigated calcined claystone sample at 750 °C.

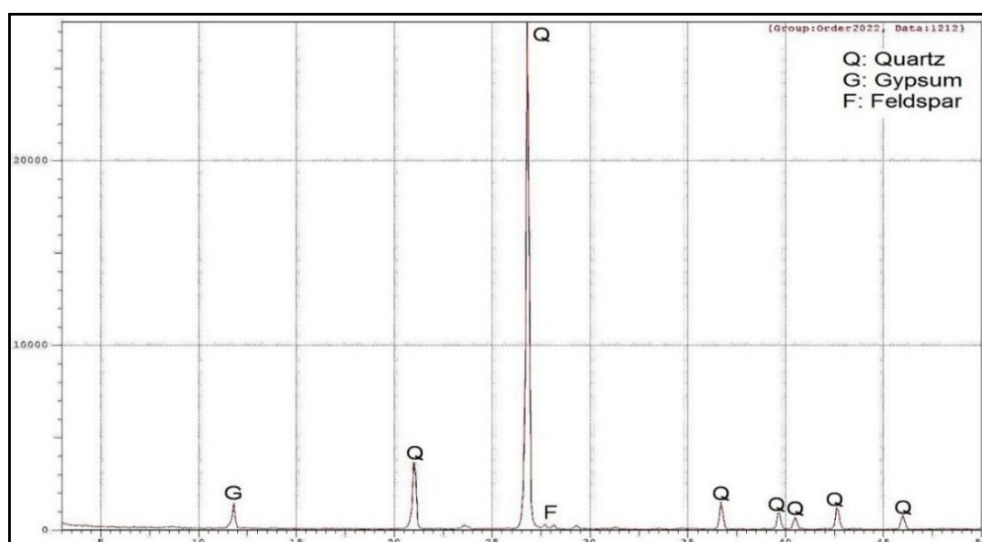


Figure 4. XRD pattern of the investigated sand sample.

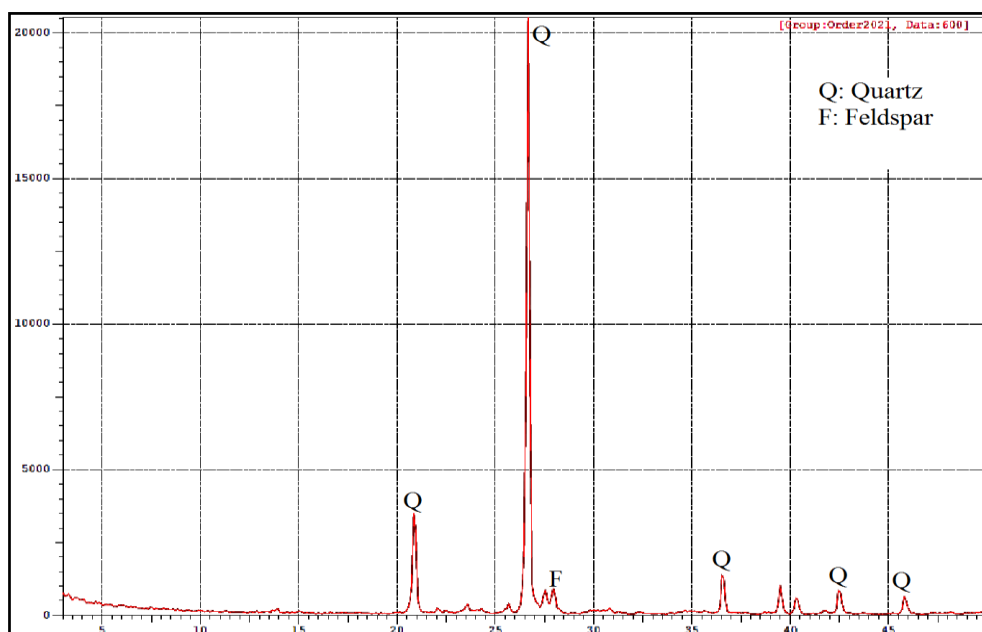


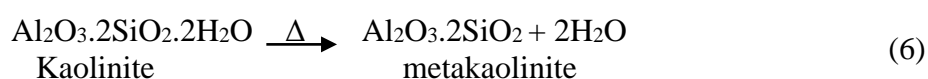
Figure 5. XRD pattern of the investigated feldspar-rich sand sample.

The results of the mineral and chemical analyses showed the presence of Palygorskite and Kaolinite as the main clay minerals, containing a remarkable amount of MgO (3.80%), part of it belongs to dolomite and the other is palygorskite clay, and it has an adherent pigment accompanying with the red kaolin clay as Fe_2O_3 (5.85%). Furthermore, the appearance of halite salt in XRD was observed also in the chemical analysis where the percentage of chlorine was 4.69%, it is a water-soluble salt, and it was shown by measuring the TDS ratio, which amounted to 8.79%.

When calcining the claystone sample at a temperature of 750 °C for 2 hours it was led to reduce the Loss On Ignition (L.O.I.) to 0.73%. Also, the major peak of Palygorskite mineral decreases

in intensity, this is imported in shrinkage percent after firing samples due to the exit of the crystallization water. The XRD diagram of calcined claystone sample at 750 °C (Figure 3) shows that the Palygorskite clay mineral remained crystallized and didn't lose all their crystallization water at this degree because the clay deposit is composed mostly of phyllosilicate minerals containing variable amounts of water trapped in the mineral structure, due to water retention in the crystalline structure of the tubular-shaped Palygorskite clay with the existence of other minerals such as quartz and feldspar (Al-Bassam, 2000, Nordmeyer, 1966).

Metakaolin as in equation (6) was used to increase the reactivity of the compound faster. Figure (2) explains the recombination of alumina and silica to form an amorphous metakaolin structure in a calcined claystone sample (Joudi et al., 2019).



Sand and Feldspar as shown in Figures (4) and (5), respectively play an important role in ceramic raw materials acting as a filler and structure material for samples to reduce shrinkage as well as, fluxing agents to reduce the melting temperatures. The chemical compositions (Na_2O , Fe_2O_3 , MgO , and CaO) are considered fluxing and can influence the densification behavior of the ceramic materials during firing and contribute to the reduction of firing temperature. During firing its fusibility and ability to form plagioclase mineral (Anorthite), makes it possible to reach a high density even at low temperatures. In fact, the great densification and high mechanical resistance shown in fired ceramic materials are due to the effect of feldspar (Escalera, 2013).

3.2. Physical and Mechanical Properties of bricks and tiles

The ceramic materials after firing produced a new material modifying in properties, and it is desirable, to maintain the stability of the product during manufacturing, as well as, the efficiency parameters (forming method, percentage of raw materials and additives, firing temperature) during the manufacture of bricks and tiles.

3.3. The effect of variables on the properties of samples

The experimental results revealed the technological properties of manufactured products. After firing, the properties of the produced ceramics were determined by controlling several variables.

It observed, the hydraulic press forming method with the pressure of 100 kg/cm^2 , some surface cracks appeared in the samples due to the high plasticity of the clay and the difficulty getting out of the crystallization water, it was observed from the properties shown in the Table (3), an increase in the density and decrease in the water absorption values, especially when the temperature reaches to 1050 °C and 1100 °C.

The properties of brick and tiles prepared are strongly influenced when adding sand and feldspar. As shown in Figures (6), (7), and (8) decreasing in shrinkage and density, increasing in water absorption, with an improvement in the external appearance by the removal of surface cracks due to the increase of the non-plastic material. The sand increases the porosity and

facilitates the release of water of crystallization from the internal composition of the samples without the appearance of cracks. It is noted that the properties of the samples in the case of adding feldspar show a significant improvement of the properties, although the addition is relatively little (5%), the effect is clear in the results.

Table 3. Physical and mechanical properties, and general shape.

Sample	Temp. (°C)	Density (gm/cm ³)	Liner Shrinkage%	Water Absorption %	Compressive Resistance (MPa)	Saturation Coefficient	External Shape
B	950	1.60	4.25	28.33	---	---	Smooth texture, light brown color with crack
	1050	1.73	5.42	18.50	---	---	Smooth texture, medium brown color with crack
BS1	950	1.58	0.30	20.70	---	---	Smooth texture, light brown color with crack
	1050	1.856	6.25	11.24	---	---	Smooth texture, medium brown color with crack
	1100	2.2	12.2	0.32	---	---	Smooth texture, dark brown color, and without crack
BS2	950	1.57	0.15	22.64	---	---	Smooth texture, light brown color with crack
	1050	1.73	4.73	13.9	---	---	Smooth texture, medium brown color, with crack
	1100	2.18	12.1	1.11	---	---	Smooth texture, dark brown color, without crack
BS3	950	1.59	0.13	23.3	---	---	Smooth texture, light brown color with crack
	1050	1.69	4.51	14.60	---	---	Smooth texture, medium brown color, with crack
	1100	1.98	11.90	1.86	---	---	Smooth texture, dark brown color, without crack
BS	1050	1.65	2.68	16.31	5.96	0.834	Smooth texture, light brown color, without crack
	1100	2.05	11.76	2.75	43.04	0.577	Smooth texture, dark brown color, without crack
BS5	1050	1.63	2.38	17.63	5.92	0.922	Smooth texture, light brown color, without crack
	1100	2.03	9.47	4.6	26.53	0.669	Smooth texture, dark brown color, without crack
BSF	1050	1.857	5.92	12.06	18.42	0.805	Smooth texture, light brown color, without crack
	1100	2.22	13.01	0.71	46.89	0.811	Smooth texture, dark brown color, without crack
CS2	1050	1.782	10.65	15.98	14.06	0.97	Smooth texture, light brown color, without crack
	1100	2.35	18.4	1.2	44.32	1.29	Smooth texture, dark brown color, without crack
CS3	1050	1.66	8.68	17.46	12.03	0.90	Smooth texture, light brown color, without crack
	1100	2.02	15.73	1.90	46.45	0.75	Smooth texture, dark brown color, without crack
CS4	1050	1.61	7.72	19.27	9.52	0.97	Smooth texture, light brown color, without crack
	1100	1.89	14.42	2.66	50.93	0.73	Smooth texture, dark brown color, without crack
CS5	1050	1.61	6.55	21.27	9.28	1.03	Smooth texture, light brown color, without crack
	1100	1.997	13.39	5.96	48.95	0.77	Smooth texture, dark brown color, without crack
CSF	1050	1.828	10.65	13.24	17.22	0.94	Smooth texture, light brown color, without crack
	1100	2.385	19	0.52	54.2	1.5	Smooth texture, dark brown color, without crack

There is a mutation by changing the properties. The increase in firing temperature from 950 °C to 1100 °C caused a dramatic change in physical and mechanical properties, as a result of consistency the liquid phases penetrated the open pores, thus closing them and isolating the

neighboring pores, and the color started to be dark from light brown to dark brown. Shrinkage increases as the firing temperature increases is the result of vitrification, which occurs as the temperature rises.

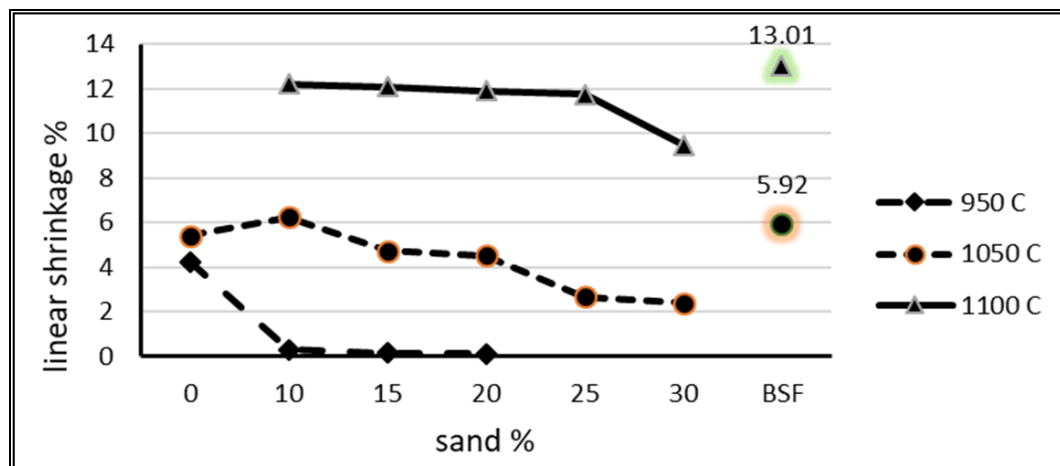


Figure 6. The effect of increasing sand on liner shrinkage for claystone samples.

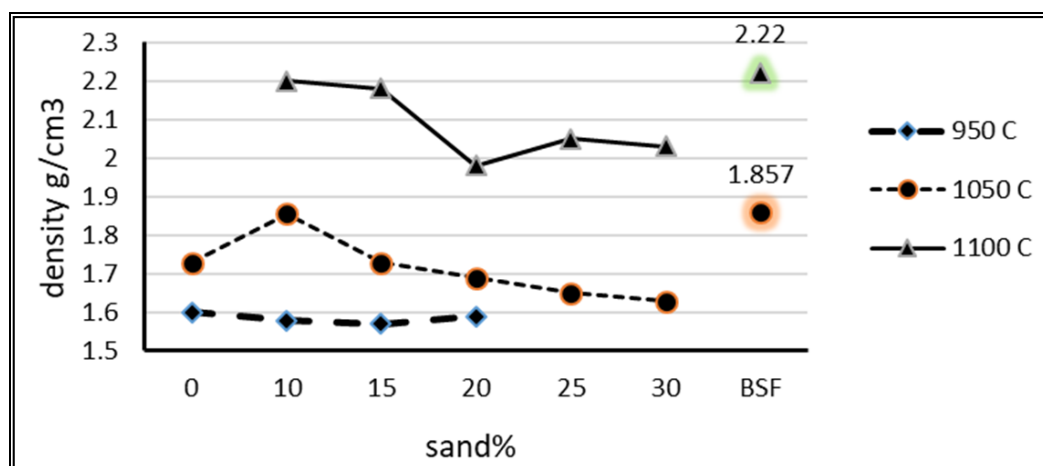


Figure 7. The effect of increasing sand on bulk density for claystone samples.

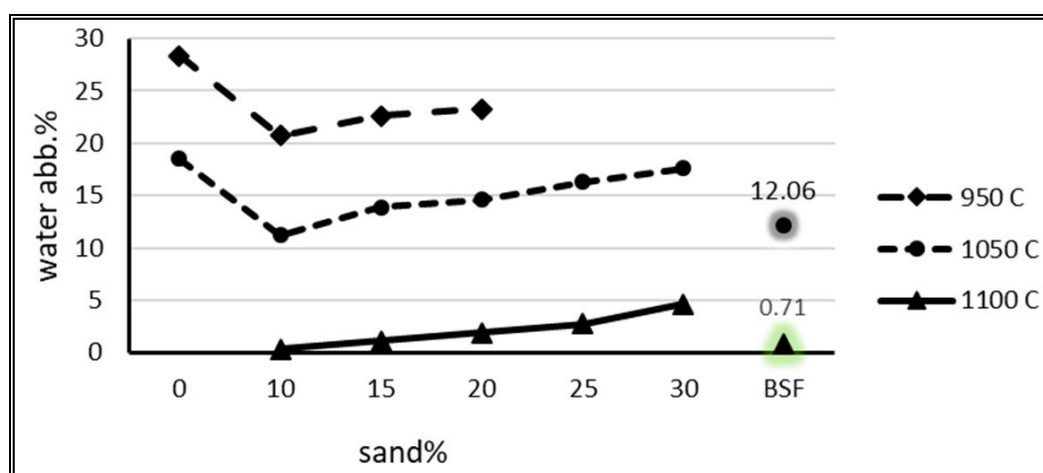


Figure 8. The effect of increasing sand on water absorption for claystone samples.

Samples were selected to test the compressive strength and observe the effect of changing the properties (adding sand, using calcined clay, and high burning temperature) on the mechanical properties. It was observed that the resistance decreased by increasing the added sand even by a small percentage. On the other hand, the burning at 1100 °C caused a leap in the mechanical properties, and this is evidence of the formation of hard minerals and the arrival of the sample to full maturity. As the temperature increases, sintering is governed by solid-state diffusion mechanisms, a stage that in general involves the rearrangement of the powder particles and the formation of a strong bond between particles. The amount of porosity decreases substantially and particles move closer leading to shrinkage of the material and also grain size increases during this stage, the presence of components such as low-melting clays thus the liquid phase present (Rundans et al., 2011; Escalera, 2013; Milheiro et al., 2005; Moreno et al., 2000). Higher temperature gave higher compressive strength for all samples as shown in Table (3).

3.4. The effect of using calcined clay

The objective of using calcined clay is to reduce the water of crystallization for clay and accordingly reduce cracks in the fired samples; the smooth surface has been reached, because of using calcined clay at 750 °C. Depending on the type of clay, the dihydroxylation process takes place at a temperature from 400 to 800 °C, and this has a significant effect on reducing shrinkage and increasing the reactivity of the compound (Dutra et al., 2019). However, it was noticed that the shrinkage remained high when using calcined clay and this was due to the increase in the surface area and the decrease in the particle size of calcined clay (Joudi and Nassrullah, 2014).

As well as the use of sand and feldspar as an additive to the samples to reduce shrinkage of the samples as shown in Figure (9). The greatest effect is shown in the change of firing temperature, when burnt at 1100 °C, there is a noticeable improvement in the physical properties, except for high shrinkage as shown in Figures (9, 10, and 11). Therefore, the temperature of 1050 °C is within some acceptable ASTM requirements specifications (C56, C57, C34, C1167).

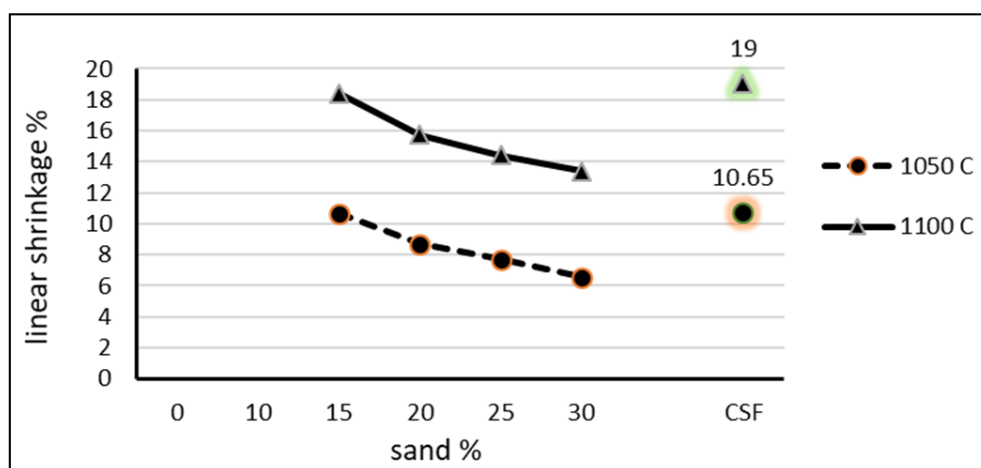


Figure 9. The effect of increasing sand on liner shrinkage for calcined clay samples.

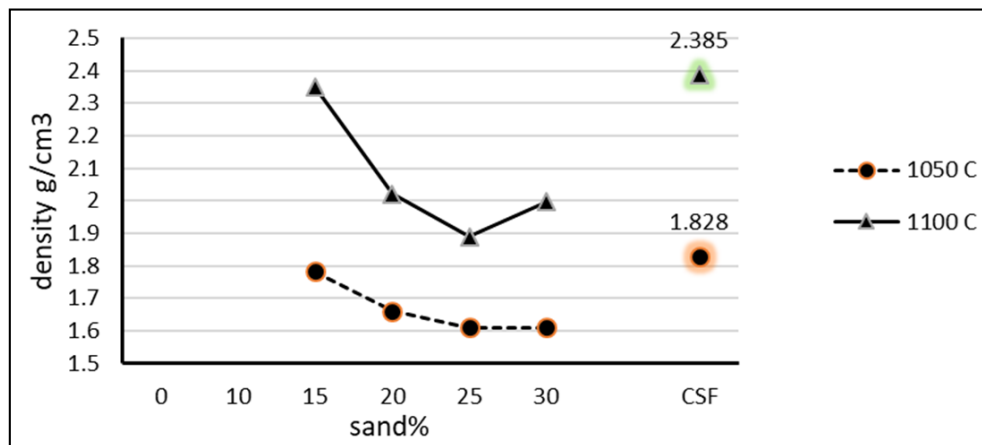


Figure 10. The effect of increasing sand on bulk density for calcined clay samples.

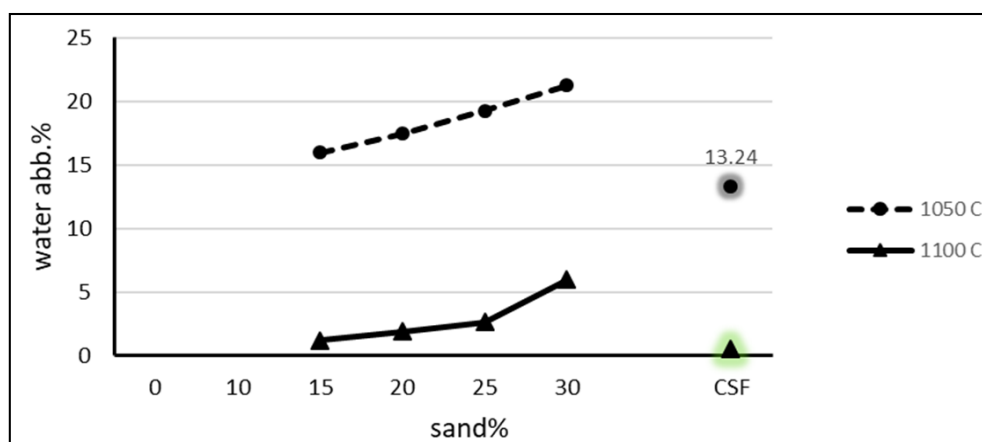


Figure 11. The effect of increasing sand on water absorption for calcined clay samples.

3.5. The color and appearance of the samples

According to the presence of iron oxide in the samples and the degree of burning, it was noted from a photo of samples (Figures 12–17) that the color of samples varied from light brown to dark brown. When burning the samples at 950, 1050, and 1100 °C, light brown, medium brown, and dark brown colors are obtained, respectively. The use of calcined clay is useful to get rid of cracks and makes the surface smooth due to the release of crystallization water. Due to the plasticity of the tested clay sample, surface cracks appear on the samples that were prepared with sand less than 20%, as shown in Figure (11; Joudi et al., 2019).

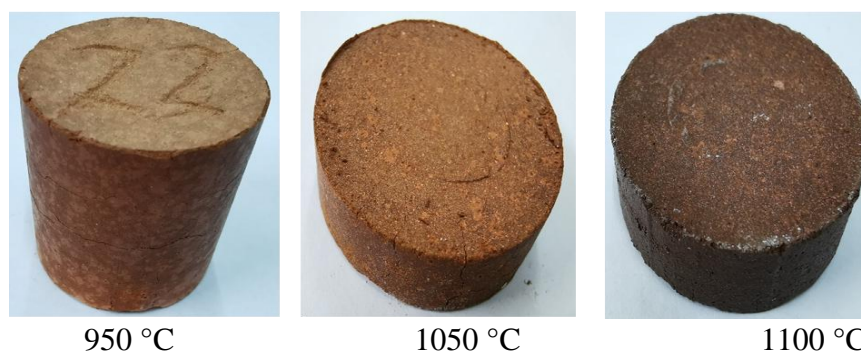


Figure 12. Samples of clay with 15% sand.

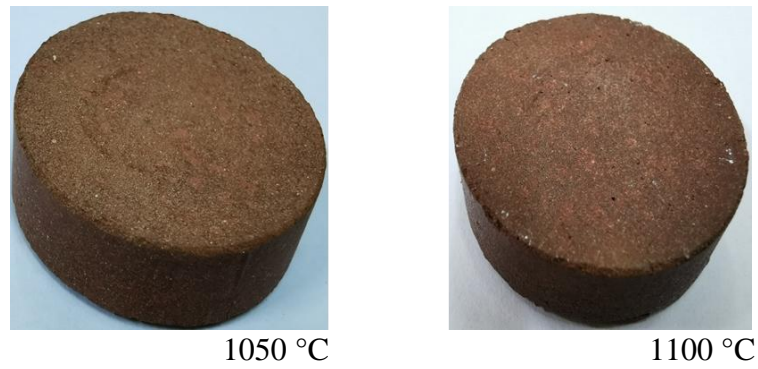


Figure 13. Samples of clay with 25% sand.

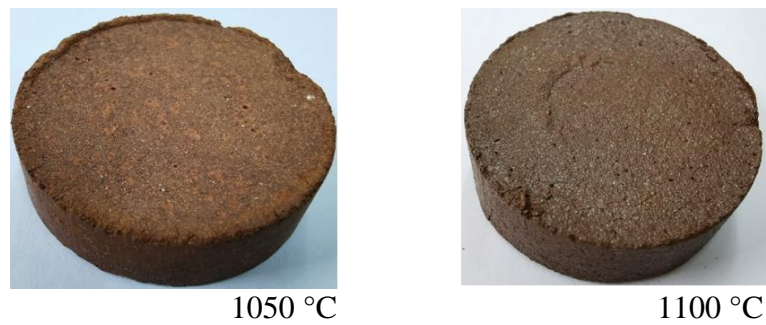


Figure 14. Samples of clay with 10% sand and 5% feldspar.

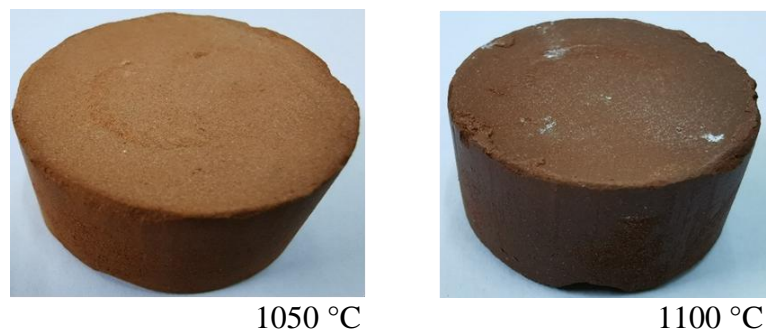


Figure 15. Samples of calcined clay with 15% sand.

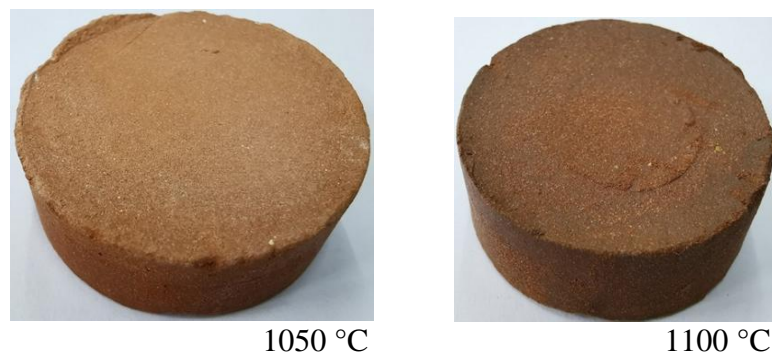


Figure 16. Samples of calcined clay with 25% sand.

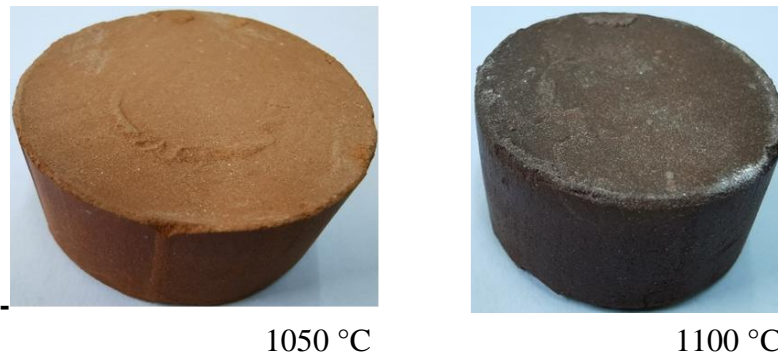


Figure 17. Samples of calcined clay with 10% sand and 5% feldspar.

3.6. Comparing results with ASTM standard specification

The successfully prepared ceramic building material samples were prepared by using the best ratio of clay (calcined or not) with sand, and successful sintering temperature, in according with ASTM specifications (structural clay facing brick or tile C216 and C212, floor tile C57, roofing tile C1167, structural clay load and non-load bearing wall tile C34 and C56) shown in Table (4).

Table 4. Comparing successful samples with ASTM standard specifications. ASTM: American Society for Testing and Materials.

Forming	Samples	Temperature (°C)	*ASTM Standard Specification
By presser 10 KN	BS	1050	C34, C56, C57, C1167
	BS	1100	C216, C212, C34, C56, C57, C1167
	CS	1050	C34, C56, C57, C1167
	CS	1100	C216, C212, C34, C56, C57, C1167

Water absorption is a major factor affecting the durability of ceramic products, larger than 10% for wall tiles and less than 10% for floor and roofing tiles. The characteristic of the water absorption of the ceramic tiles provides information on the mechanical properties of the tiles. The lower water absorption values give higher mechanical resistance, as shown in Table (3) (Mousharraf et al., 2011).

3.7. Phase composition and microstructure

The phase composition of the fired samples is shown in Figures (18–21). The samples look similar in mineral composition (quartz, feldspar, hematite), although they contain slight changes in the crystallization rate when the firing temperature rises to 1100 °C, this is attributed to the fact that the crystalline phases are at the beginning of growth of minerals, and it contains molten phases because of its increasing distance from the base of the drawing of XRD. The appearance of Augite in some samples fired at 1100 °C as in (BSF) could be attributed to the low percentage of sand (10%) and the presence of feldspar, small peaks of the augite mineral

are observed as in Figures (17, 18, and 19). Augite (Ca, Na) (Mg, Fe, Al, Ti) (Si, Al)₂O₆ crystallizes in a continuous series of reactions due to the formation of magma at low temperatures of 851 °C (Escalera, 2013). Knowledge of the mineral phase composition of prepared samples is very important for understanding the technological properties of clay products (Joudi et al., 2021).

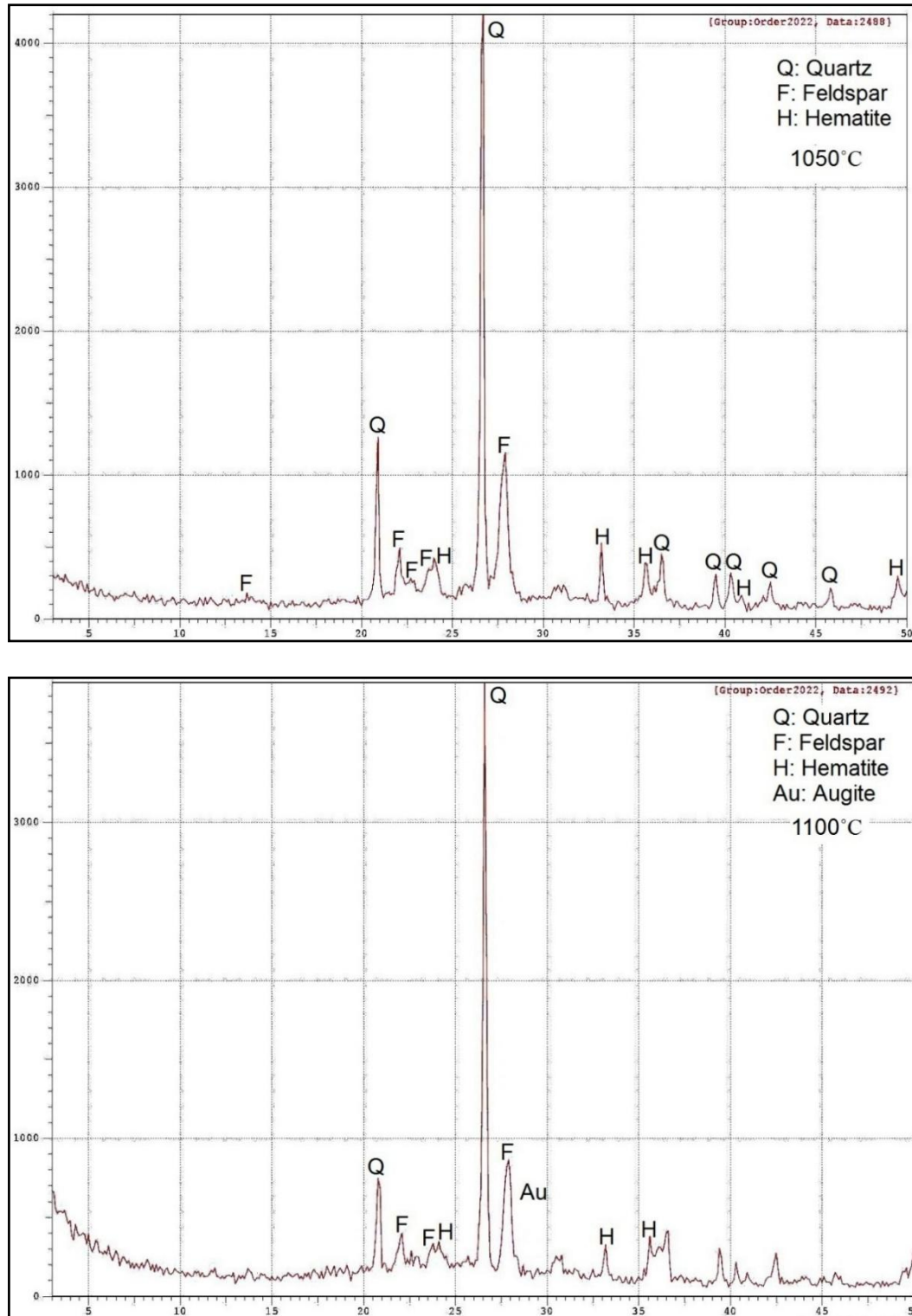


Figure 18. XRD diagram of claystone sample with 25% sand fired at 1050 and 1100 °C.

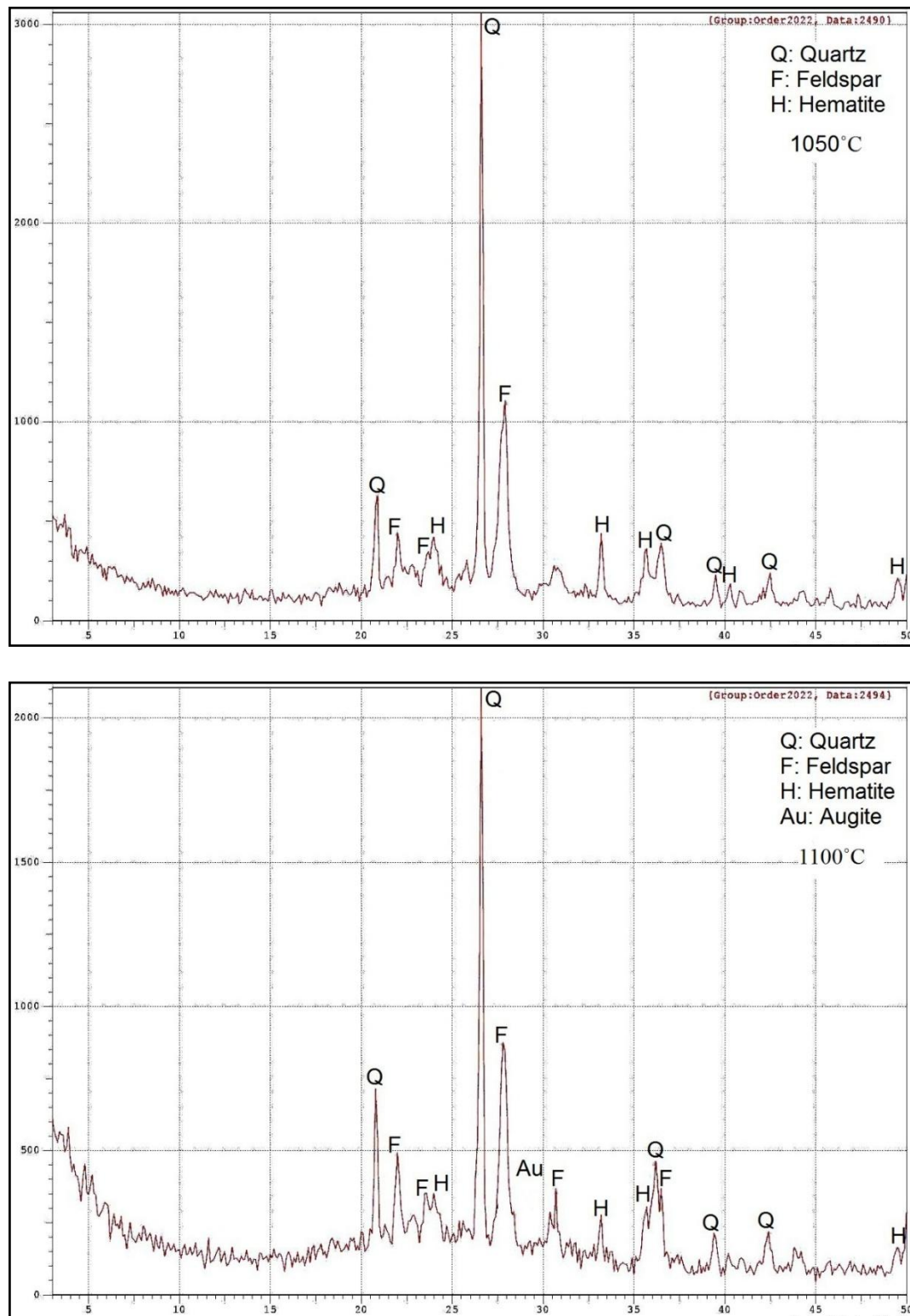


Figure 19. XRD diagram of claystone sample with 10% sand and 5% feldspar fired at 1050 and 1100 °C.

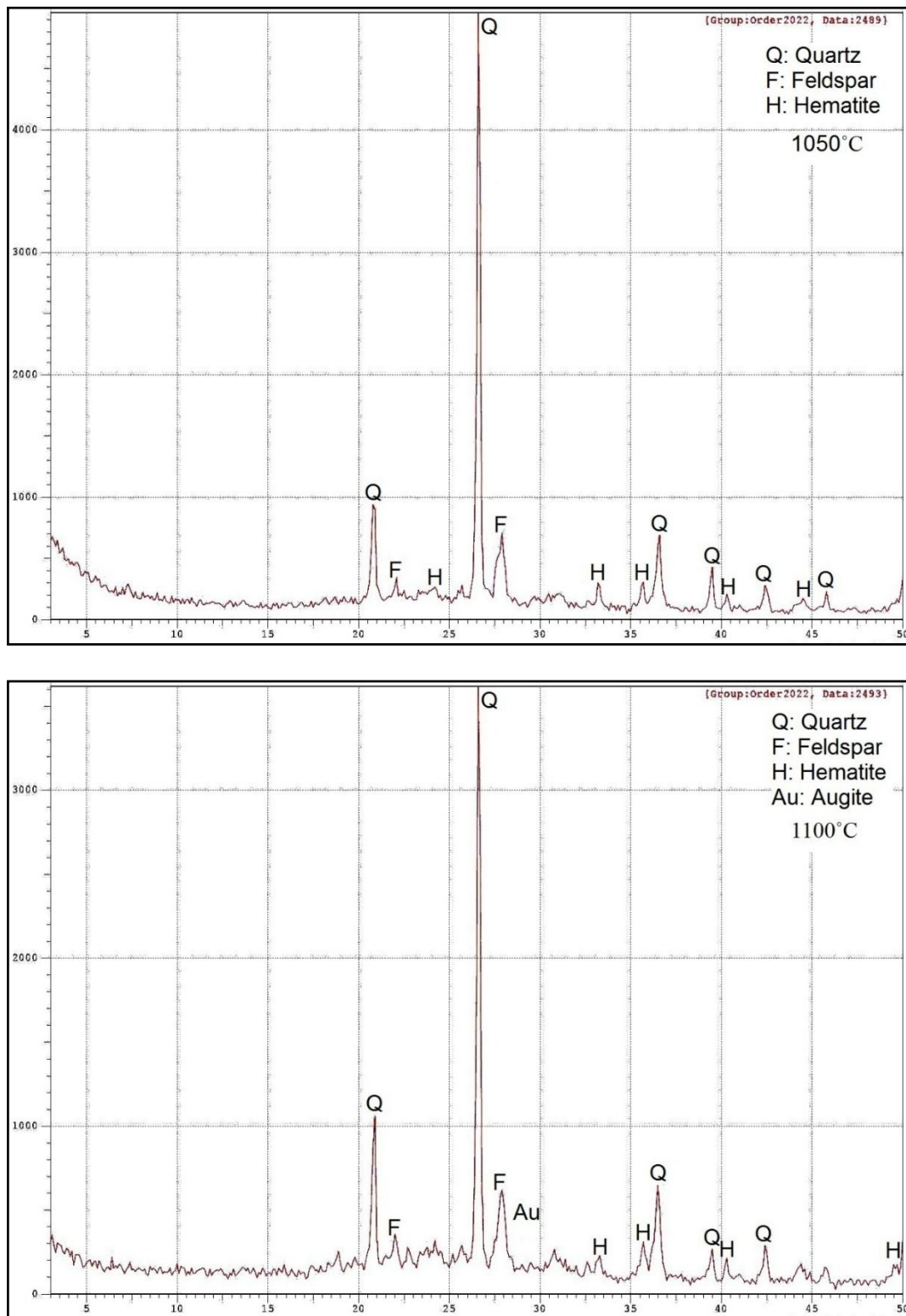


Figure 20. XRD of calcined clay sample with 25% sand fired at 1050 and 1100 °C.

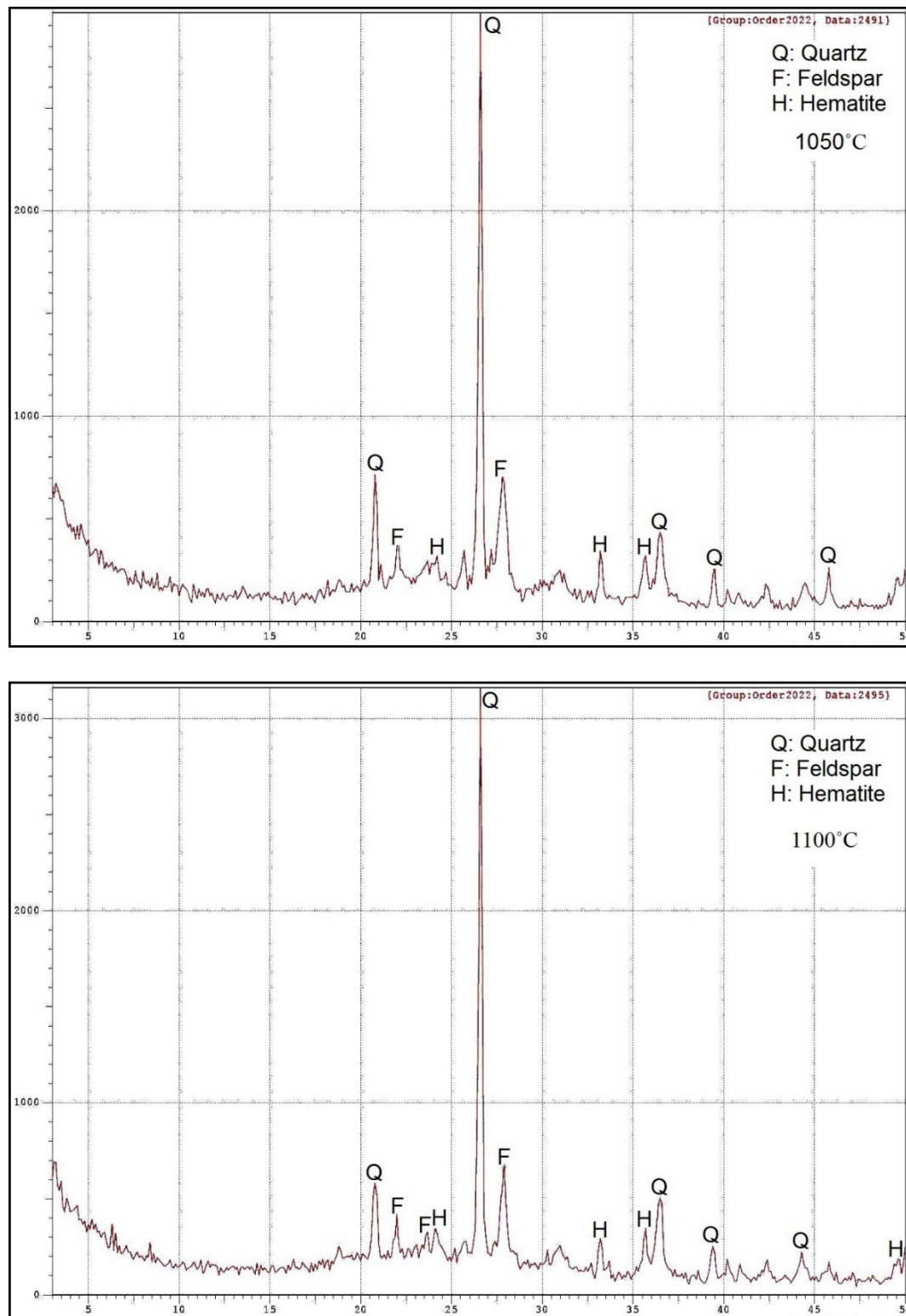


Figure 21. XRD of calcined claystone sample with 10% sand and 5% feldspar fired at 1050 and 1100 °C.

6. Conclusions

According to the ASTM requirements, the claystone of the Fatha (Nfayil) Formation in the Bussiyya area is considered suitable as a raw material for the tiles industry when fired at a temperature of 1050 °C. Using calcined clay useful in riding cracks and making the surface smooth. From the results, the two mixtures of BS4 and CS4 were recommended at a temperature of 1050 °C to maintain the shrinkage at an acceptable limit and to obtain appropriate properties according to the requirements. The addition of feldspar (5%) as a partial substitute improved the physical and mechanical properties, such as in the samples BSF and CSF, but didn't increase the percentage of feldspar to maintain not increasing the cost of the product.

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