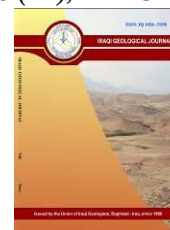




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Validity of Smectite Clays as a Binding Material for the Production of Refractory Bricks

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Abstract

This study aims to evaluate the suitability of local clays rich in smectite for the manufacture of refractory bricks by characterizing their chemical, mineralogical, physical, and thermal properties at high temperatures. Two raw materials were used: local bauxite as the main source of alumina, and local smectite clay as the main source of silica and as a binding agent, added at proportions of 10%, 15%, and 20%. The bricks were formed using a semidry pressing method with 15% water content by weight, and pressed at 10 tons. The bricks were pressed to the required dimensions according to standard specifications. After complete drying, the brick samples were fired at 1400°C for 2 h. The investigation included chemical analyses to determine major oxide proportions, as well as physical, mechanical, and thermal tests to evaluate industrial performance, in addition to X-ray diffraction analysis and scanning electron microscopy. The results showed a relative decrease in Al₂O₃ content when smectite content increased, which negatively reflects on the melting resistance. X-ray diffraction analysis confirmed the formation of the mullite phase after firing, accompanied by tridymite, corundum, and occasionally sillimanite. Scanning Electron Microscope observations revealed a microstructure characterized by an interlocking network of mullite fibers of varying lengths, which enhances thermal and mechanical stability. In addition, a silica phase and distributed angular corundum grains were identified. Increasing the smectite content lead to a decrease the bulk density and a relative decrease in thermal shock resistance and refractoriness, while improving the mechanical resistance. In contrast, high smectite content resulted in increased volumetric and linear shrinkage due to crystal rearrangement during heating. The results indicate the possibility of using local clays rich in smectite at rates 10% and 15% with local bauxite to produce refractory bricks that conform to Iraqi standard specifications, thereby supporting the economic viability of the local industry.

Keywords: Bauxite; Smectite; Refractory Bricks; Mullite; Refractoriness

1. Introduction

Refractories are non-metallic materials designed to withstand high temperatures above 1000 °C while maintaining their structural properties (Ibrahim et al., 2019; ASTM C71, 2024), characterized by high melting resistance and the ability to withstand high temperatures, both physically and chemically (Khanmohammadi and Mohtadinia, 2024). These heat-resistant materials are essential for practical temperature insulation and control, and for reducing heat loss (Schacht, 2004). To reduce heat leakage to the outside environment and energy use, refractory materials are utilized in the metallurgical industry. They serve as the inner lining of furnaces for melting and heating, in melting vessels for storing and

transporting molten metals, and in chimneys through which hot gases pass (Ewais, 2004). Refractory materials (refractory bricks) are made from natural and industrial materials, such as fireclay, bauxite, alumina, magnesite, dolomite, chromite, zirconia, and silicon carbide (Jadeja and Saradava, 2014). Another group is usually non-metallic, or a mixture of other components and minerals (Sadik et al., 2014).

The criteria for selecting the materials mentioned above are their ease of processing, abundance, and stability in manufacture of refractory materials (Sengupta, 2020). Due to the presence of large quantities of local Iraqi clay raw materials suitable for the production of refractories, many researchers have produced refractory bricks from local raw materials to provide alternatives to imported bricks. Jodi (2005) conducted laboratory experiments to evaluate Iraqi flint and kaolin clays for the production of refractories. Laboratory results have shown the suitability of flint clays for the production of refractories and the improvement of the efficiency of the thermal product after firing due to the increased proportion of mullite crystals. Hadi (2017) prepared refractories from Iraqi flint ore by adding different weight percentages 25, 30, 35, 40, and 45% of Iraqi white kaolin as a binding material.

The results showed a decrease in apparent porosity and water absorption rate, and an increase in the linear shrinkage rate with increasing kaolin content added, due to the increase in the glass phase in the thermal product. Therefore, this study investigates the potential use of Iraqi smectite-rich clays as binding materials in refractory brick production and evaluates the resulting products through mineralogical, physical, mechanical, and thermal analyses, comparing them with Iraqi standard specifications. However, previous studies had mainly focused on kaolin and flint clay as binding materials, while limited attention has been given to Iraqi smectite-rich clays despite their abundance and favorable binding characteristics. Therefore, the industrial suitability of local smectite clays for refractory brick production must be evaluated.

2. Study Area

The study area is located in Wadi Bashirah within the Western Desert of Iraq, approximately 85 km southwest of Rutba City in the Anbar Governorate (Fig. 1).

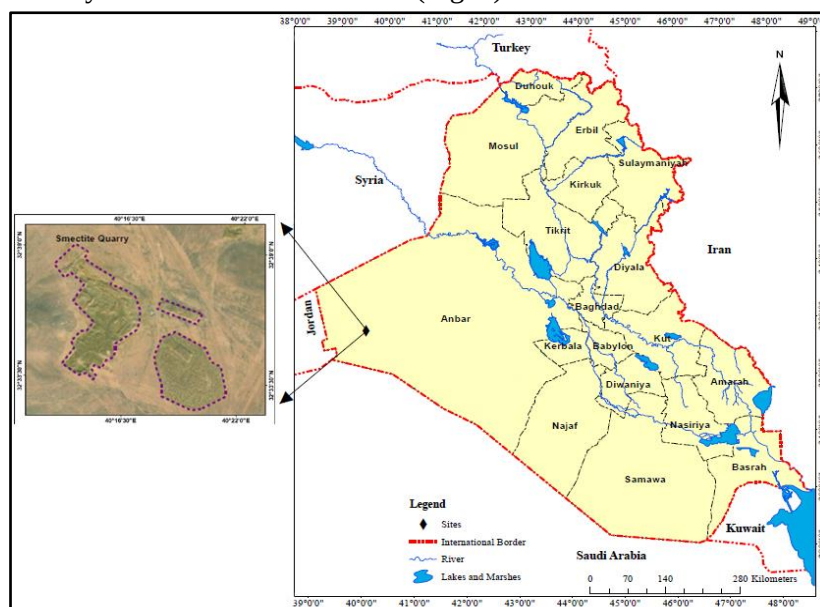


Fig. 1. Location map of the study area

Geologically, the investigated clay deposits belong to the Digma Formation, which consists predominantly of claystone and marl units with varying carbonate content. The area was selected due to

the abundance of smectite-rich clay deposits with potential industrial applications in refractory production.

3. Materials and Methods

3.1. Raw Materials

3.1.1. Bauxite

The bauxite that used is locally sourced, and supplied by the General Company for Glass and Thermal Materials, which is affiliated with the Ministry of Industry and Minerals. It was taken from the bauxite site in the western desert of the Anbar Governorate. Bauxite mainly consists of hydrated aluminum oxides and impurities such as SiO_2 , Fe_2O_3 , TiO_2 , CaO , MgO , Na_2O , and K_2O (Kirk-Othmer, 1992). It is one of the primary raw materials for the production of aluminum and refractory bricks (Al-Amer and Al-Kadhemy, 2015). The higher the percentage of aluminum oxide, the better the production of refractories (Hadi, 2017).

3.1.2. Clay

Smectite clay is rich in calcium montmorillonite (Al-Bassam et al., 2011). This deposit is characterized by its low purity, as its average montmorillonite content is about 68%, and is associated with clayey and non-clayey impurities. Calcite is the main impurity, constituting approximately 15% of the sediment weight (Al-Ajeel and Abdullah, 2010). Calcium montmorillonite has a strong bond and lower swelling capacity than sodium montmorillonite (Murray, 2007).

3.2. Chemical Analysis of Raw Materials

Chemical analysis was performed using X-ray fluorescence (XRF) to identify the major elements present in the raw material (Table 1).

Table 1. Chemical compositions of the raw materials

Oxides	Bauxite	Clay
Al_2O_3	50.67	17.91
SiO_2	43.7	64.26
Fe_2O_3	2.18	7.27
MgO	1.59	3.14
CaO	1.02	6.66
Na_2O	0.15	0.19
K_2O	0.01	0.41
SO_3	0.09	0.011
L.O.I	0	0
Total	99.41	99.851

3.3. Preparation of Materials

The clays were fired at a temperature of 700 °C, while the bauxite was fired at a temperature of 1000 °C. The raw materials were ground so that the particle size of the bauxite was less than 1 mm, while the particle size of the clay was less than 300 μ . The raw materials are then mixed homogeneously to produce a more chemically and physically homogeneous material for forming refractory bricks. Three

mixtures were prepared with varying proportions of raw materials to obtain the optimum raw material mixing ratio, as shown in Table 2. 15% water was added, and the samples were pressed under a pressure of 10 tons (10000 kg/cm²) to dimensions of (51×51×51 mm) to examine the physical properties and with dimensions of (50×50×50 mm) to examine the thermal and mechanical properties (Fig. 2). The samples were dried for 48 h at room temperature, followed by drying in an oven at 105 ± 5 °C to reduce the possibility of cracking during the firing process. Then, they were fired at a temperature of 1400°C in the laboratories of the Scientific Research Authority - Advanced Materials Department, according to a specific burning program (increasing the temperature by 100°C per hour) with a holding time of two hours. After the bricks cooled, physical, mechanical, and thermal tests were conducted following the ASTM standards.

Table 2. Raw material mixing ratios

No. Sample	Bauxite	Clay
1	90%	10%
2	85%	15%
3	80%	20%

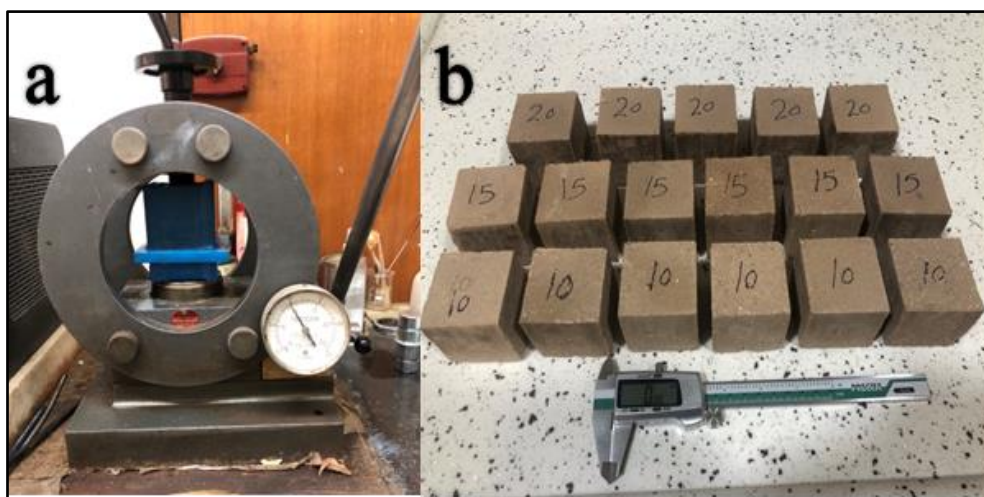


Fig. 2. a) Brick compression device; b) Brick samples before firing

3.4. Physical Properties Test

These tests are used to ensure material quality and performance in various industrial applications, contributing to improved process efficiency and reduced costs. Physical properties include apparent density, apparent porosity, absorption rate, and apparent specific gravity. Physical tests were conducted according to ASTM C 20-00.

3.4.1. Bulk density and apparent porosity

The apparent density of a porous material is defined as the ratio of the mass of the material to its apparent volume, and the apparent porosity is defined as the ratio of open pores to the apparent volume of the material (Dodd and Murfin, 1994). Bulk density can vary for the same type of refractory material (Sengupta, 2020). The higher the value of bulk density, the lower the porosity (Pountouenchi et al., 2023). Bulk density and apparent porosity are often employed in quality control tests (Schacht, 2004). These properties can influence other characteristics of the material, such as its thermal conductivity and resistance to thermal shock. Materials with low porosity can exhibit higher resistance to chemical

influences than those with high porosity (Sengupta, 2020). The following equations were used to calculate the bulk density and apparent porosity.

$$B \text{ g/cm}^3 = W/V \quad (1)$$

$$P \% = [(W_1 - W)/V] \times 100 \quad (2)$$

Where: B = Bulk Density, P% = Apparent Porosity, W = Dry Weight, W_1 = Saturated weight, Volume of open pores, $\text{cm}^3 = W_1 - W$, Volume of impervious portion, $\text{cm}^3 = W - W_2$, W_2 = Suspended Weight

3.4.2. Water absorption

The water absorption ratio is defined as the relationship between the weight of the absorbed water and that of the dry sample (ASTM, 2000). The water absorption ratio is influenced by two significant factors. The first factor is the influence of the sintering process, whereas the second factor is the development of channels in the brick as a result of the release of gases (Al-Amer and Al-Kadhemy, 2015). The water absorption ratio was expressed as a percentage and calculated as follows:

$$A \% = 100 \times [(W - D)/D] \quad (3)$$

where: A% = Water absorption, W = Saturated weight, D = Dry weight

3.5. Mechanical Properties Test

Mechanical tests are necessary for evaluating the refractory bricks. These properties provide an idea of the mechanical strength of refractory products on-site (Sengupta, 2020).

3.5.1. Cold crushing strength (CCS)

The cold crush strength of a given material is defined as the maximum load at failure per unit cross-sectional area when it is compressed at room temperature. The cold crush strength test evaluated the maximum load that a given material can withstand. Higher values indicate a suitable firing temperature for formed refractory products, which is required for sintering as well as for developing a certain microstructure in the refractory materials, and also protects against mechanical damage during operation. Cold crushing strength testing focuses on values below the specified minimum, as they may reflect a lower degree of bonding. The test was conducted according to ASTM C-133-94. Standard cubic specimens (50 x 50 x 50 mm) were used to ensure good contact between the bearing surfaces. The specimens were loaded until failure at a specified rate. The cold crush value was calculated using the following equation:

$$\text{Cold Crushing Strength (CCS)} = \text{Load (N)} / \text{Area (mm}^2\text{)} \quad (4)$$

Results are expressed in N/mm² (MPa).

3.6. Thermal Properties Test

These are the properties most relevant to the performance of a refractory material in service. Measuring these properties provides a more accurate indication the performance of the material in a particular application (Sengupta, 2020).

Permanent volume, and linear change (PVC)(PLC) is a measure of the permanent change in the dimensions of the refractory brick, whether expansion (positive values) or contraction (negative values), as a result of heating, preferably to the expected hot surface service temperature. It indicates the maximum expected dimensional change and is expressed as a percentage (Schacht, 2004). During the manufacturing process, refractory bricks change dimensions when fired at high temperatures. They either shrink or expand in size. This occurs as a result of solid-state reactions, which form liquid phases,

or due to a phase transition (Silva et al., 2021). The kinetics of such reactions are very slow and do not attain equilibrium during the burning process employed in manufacturing. Hence, when refractory bricks are exposed to heat over a period of service, their dimensions may change (Sengupta, 2020). Low linear and volumetric changes are usually desirable as they imply high stability in the material, as well as low shrinkage to prevent the loosening of the refractory lining in kilns. They also prevent the penetration of corrosive materials (Schacht, 2004). The permanent linear change was calculated according to the American standard ASTM-C1407 using the equations 5 and 6.

$$\text{Permanent Volume Change (PVC) \%} = (VB - VA/VA) \times 100 \quad (5)$$

$$\text{Volume}(V) = W/B \quad (6)$$

Where: VA = original volume (cm³), VB = final reheat volume (cm³), W = Weight (g), B = Bulk density (g/cm³).

Conversion of Permanent Volume Change% to Permanent Linear Change% of Reheat Specimens: If volume change is negative (shrinkage), then.

$$\text{Permanent Linear Change (PLC) \%} = -[1 - (1 + PVC/100)^{1/3}] \times 100 \quad (7)$$

If the volume change is positive (expansion), then.

$$\text{Permanent Linear Change (PLC) \%} = [(1 + PVC/100)^{1/3}] \times 100 \quad (8)$$

3.7. Thermomechanical Properties Tests

The thermomechanical properties of the refractory material provide an idea of its potential to withstand the stresses when refractory materials are subjected to different types of compressive, shear, and tensile stresses at high temperatures (Sengupta, 2020). In the following section, the thermomechanical properties and tests carried out on the refractory materials are discussed.

3.7.1. Thermal shock resistance

Thermal shock resistance is the ability of a material to withstand changes in temperature without cracking. This condition can occur when the material is subjected to a sudden change in temperature or is exposed to a sharp temperature gradient during use (Schacht, 2004). Rapid temperature variations induce thermomechanical stresses in the refractory lining, such as insulation linings in high-temperature industrial units, which are subjected to periodic thermal shocks (Andreev et al., 2019). Refractory bricks are assumed to withstand multiple heating and cooling cycles without cracking or disintegration (Odewale et al., 2020). Various methods are used to measure thermal shock. These involve measuring the level of cracking observed and the number of cycles until failure. The test was carried out according to the standard specification ASTM C1171-05. Standard-sized brick samples were heated to 900°C according to ASTM C1171-05 in a kiln for 15 min. The hot bricks were then removed from the kiln and immersed in water at room temperature for 15 min. This process was repeated until observed cracks were in the bricks.

3.7.2. Refractoriness

The heat resistance of bricks was estimated using the Schoen formula (Agbo et al., 2022; Odewale et al., 2023) using the equation 9.

$$R = (360 + Al_2O_3 - SO)/0.228 \quad (9)$$

Where: R = Refractoriness (°C), Al₂O₃ = Alumina Content in refractory bricks, SO = Sum of all the Oxides (Fe₂O₃+CaO+MgO+SO₃) besides SiO₂ in refractory bricks 360 and 0.228 are constants

3.8. X-Ray Diffraction (XRD)

The mineralogical analysis of the burnt brick samples was done using the X-ray Diffraction (XRD) to identify the crystalline phases present in the samples.

3.9. Scanning Electron Microscope (SEM)

A Scanning electron microscope (SEM) was used to examine the microstructure and surface morphology of the samples.

4. Results

Table 3 shows the properties of the produced refractory bricks and compares them with the limits of the Iraqi Standard Specification No. 2023/1996. Chemical analysis was performed using XRF to identify the major elements present in the refractory bricks. The chemical composition results indicate that silica (SiO_2) and alumina (Al_2O_3) are the dominant oxides in all samples. The SiO_2 content increases from 45.75% to 47.81% with increasing addition ratio, while Al_2O_3 decreases from 47.38% to 44.11%. Iron oxide (Fe_2O_3) shows a slight increase from 2.68% to 3.19%, and magnesium oxide (MgO) also increases from 1.74% to 1.90%. Similarly, calcium oxide (CaO) increases from 1.58% to 2.58%. SO_3 remains low at 0.07-0.08% and the LOI is 0% in all samples due to firing at 1400 °C. The total oxide content is 99.24% for all samples (Table 4).

Table 3. Properties of the produced refractory bricks and comparing them with the limits of the Iraqi Standard Specification

Propertis	Limits	Sample		
		10%	15%	20%
Bulk density (gm/cm^3)	> 2.1	2.17	2.12	2.03
Apparent porosity (%)	< 27.5	19.24	15.03	17.32
Absorption rate (%)	-	8.83	7.21	8.53
Cold crushing strength MPa	> 24.5	25.7	25.8	26.2
Permanent Volume Change (PVC) % on re- heating	-	-0.888	-1.029	-1.141
Permanent Linear Change (PLC) % on re- heating	(+0.2 - 0.6)% at 1400C	-0.296	-0.344	-0.382
Thermal shock	> 15 Cycle	16	16	12
Refractoriness	1750° C	1760.1	1750.2	1740.3
Al_2O_3 (%)	> 45	47.38	45.74	44.11

Table 4. Chemical compositions of the resulting refractory bricks

Oxide	Sample		
	10%	15%	20%
Al_2O_3	47.38	45.74	44.11
SiO_2	45.75	46.78	47.81
Fe_2O_3	2.68	2.94	3.19
MgO	1.74	1.82	1.9
CaO	1.58	1.86	2.14
SO_3	0.08	0.07	0.07
L.O.I	0	0	0
Total	99.24	99.24	99.24

5. Discussion

The results demonstrate a strong correlation between phase composition, chemical characteristics, microstructure, and the physical performance of the produced refractory bricks. The bulk density values of the burnt samples ranged between 2.17 and 2.03 g/cm³, and an inverse relationship was observed between the bulk density and the percentage of smectite clay added, as the density decreases with increasing weight content of smectite. This decrease is attributed to the structural changes associated with the burning process, resulting in microstructure development and a relative decrease in bulk density, which was also reflected in the absorption values. Apparent porosity initially decreased from 19.24% to 15.03% due to improved particle packing and enhanced liquid phase formation. However, further smectite addition (20%) caused a slight increase to 17.32%, likely due to excessive shrinkage and pore formation during firing process.

The cold crushing strength ranged between 25.7 and 27.2 MPa, and showed a clear increase with the increasing proportion of smectite added. This is explained by the formation of larger quantities of the glassy phase at the burning temperature (1400°C), where this phase acts as a bonding agent between the grains, which enhances structural bonding and increases the resistance of the samples to mechanical stresses. Upon reheating the refractory bricks to 1400°C, the values for permanent volumetric change ranged from -0.888% to -1.141%, while the values for permanent linear change ranged from -0.296% to -0.382%. This shrinkage is attributed to partial melting and increased formation of the glassy phase, which contributes to closing the interstitial pores and reducing the voids, leading to a reduction in the overall volume of the models.

Regarding resistance to thermal shock, samples containing 10% and 15% smectite clay showed the ability to withstand more than 15 heating and cooling cycles, while it decreased to 12 cycles at 20% content. Increased porosity contributes to improve thermal shock resistance due to the pores' ability to absorb thermal stresses resulting from repeated expansion and contraction; however, an excessive increase in the glassy phase may negatively affect this behavior. Thermal shock behavior appears to be controlled by a combination of porosity and glass phase development rather than porosity alone. The refractoriness, it ranged between 1760.1 and 1740.3°C, and a gradual decrease was observed with the increase in the smectite content. This is attributed to the relative decrease in Al₂O₃ content, which leads to a reduction in the formation of the mullite phase, which is the main phase responsible for enhancing the refractoriness and thermal stability in high alumina firebricks.

The increase in SiO₂ content with increasing smectite addition can be attributed to the silica-rich nature of smectite clay. Simultaneously, Al₂O₃ decreased because the proportion of alumina-rich bauxite was reduced.

The XRD analysis of the refractory bricks reveals the presence of multiple crystalline phases, with mullite (3Al₂O₃.2SiO₂) dominating the crystal structure at a primary level, accompanied by secondary phases of sillimanite (Al₂SiO₅), corundum (Al₂O₃), cristobalite (SiO₂), tridymite (SiO₂), quartz (SiO₂), and a glassy phase. This complex crystal structure reflects the sequential thermal reactions occurring during the firing process, where bauxite (Al₂O₃.3H₂O) gradually transforms into crystalline aluminum oxide while reacting with silica liberated from the decomposition of smectite (silicate clay) to form mullite mineral.

The XRD patterns of the fired refractory brick samples (Figs. 3 and 4) indicate that mullite is the dominant crystalline phase in all specimens, accompanied by corundum, sillimanite, tridymite, and cristobalite. The predominance of mullite confirms the successful reaction between alumina-rich bauxite and silica-rich smectite during firing at 1400°C. Mullite is considered the most desirable phase in aluminosilicate refractory systems because of its excellent thermal stability and mechanical properties.

Corundum peaks indicate the presence of residual alumina that did not completely participate in mullite formation. The presence of tridymite and cristobalite is attributed to polymorphic transformations of silica at elevated temperatures. The 15% clay sample additionally exhibited quartz peaks, indicating the presence of residual silica due to the increased clay content.

These silica polymorphs may contribute to the slight increase in shrinkage and the reduction in refractoriness due to their lower refractoriness compared with alumina-rich phases and their influence on liquid-phase formation during firing.

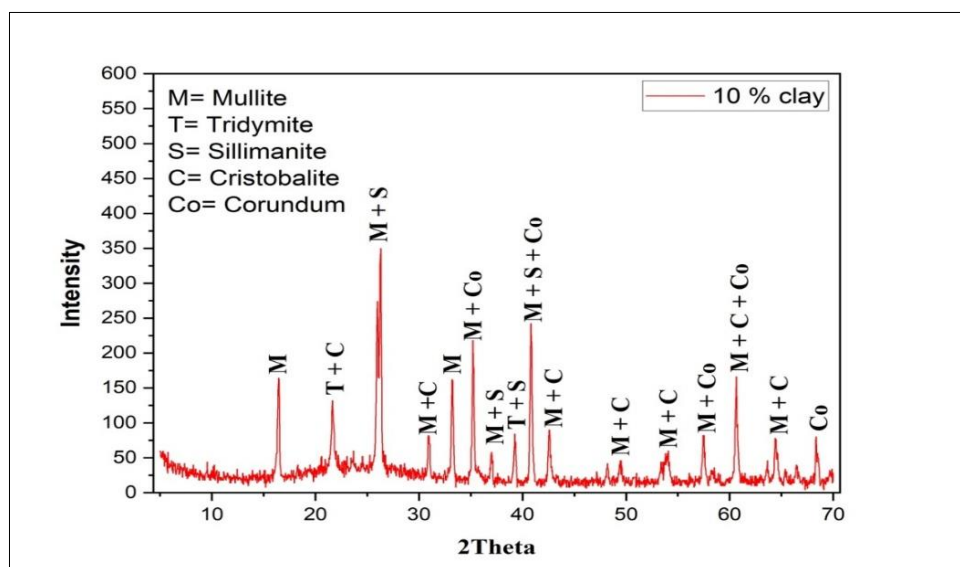


Fig. 3. X-ray diffraction pattern of a refractory brick sample composed of bauxite and 10% clay

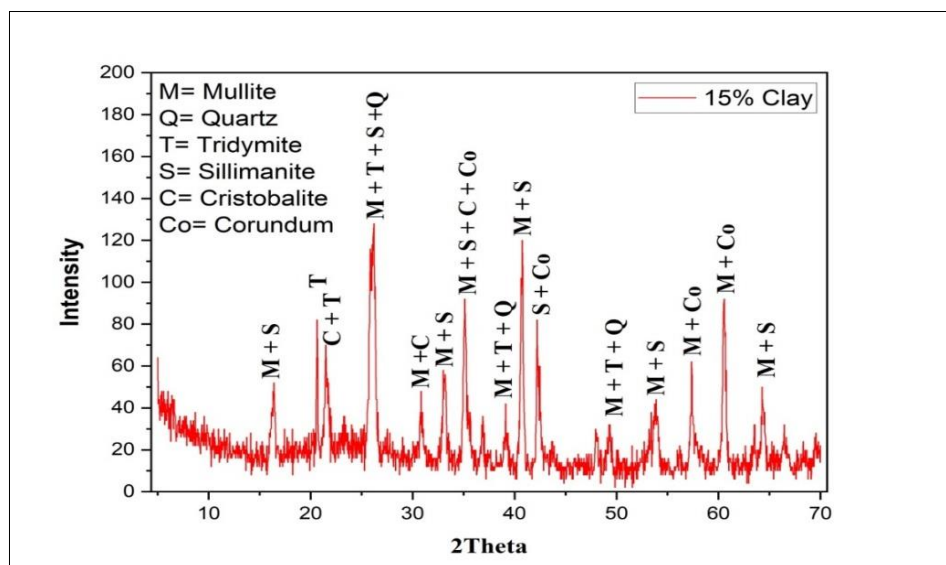


Fig. 4. X-ray diffraction pattern of a refractory brick sample composed of bauxite and 15% clay

SEM observations (Fig. 5) revealed heterogeneous microstructures characterized by elongated needle-like mullite crystals forming an interconnected network structure.

Corundum grains and silica-rich phases were also identified in the samples. The interlocking mullite fibers contribute significantly to strengthening the refractory matrix and improving the mechanical properties, thereby improving both thermal and mechanical stability. This observation is

consistent with previously reported findings. In addition, a glassy phase was observed in the specimens with higher clay content, which may improve particle bonding and densification, contributing to a reduction in apparent porosity. However, excessive glassy phase formation may adversely affect refractoriness because of the lower softening temperature of the vitreous phase. The presence of silica polymorphs may also contribute to the observed changes in firing shrinkage and thermal behavior.

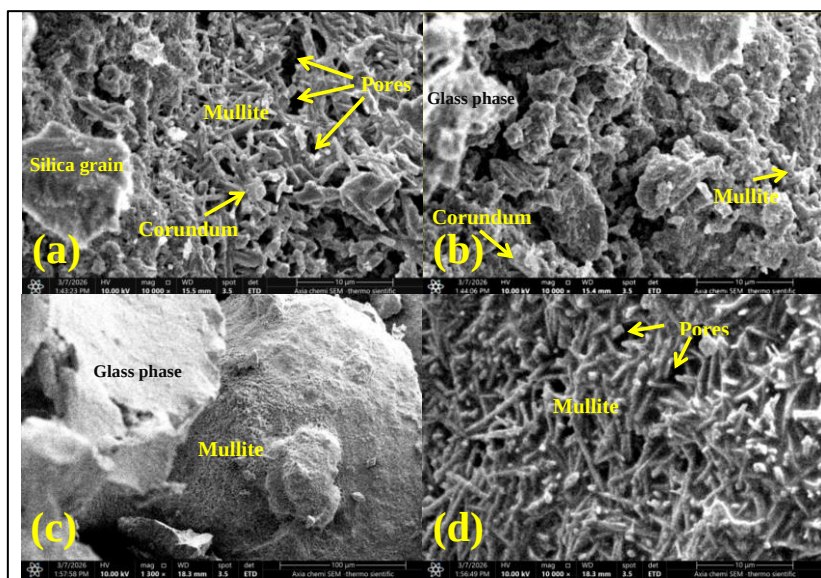


Fig. 5. SEM of a refractory brick: a,b) sample composed of bauxite and 10% clay; (c,d) sample composed of bauxite and 15% clay

6. Conclusions

This study demonstrated the potential of locally available smectite-rich clay as a binding material combined with Iraqi bauxite from the Western Desert for refractory brick production. The investigation revealed smectite-rich clay additions of 10–15 wt.% produced refractory bricks satisfying Iraqi industrial standard requirements with acceptable physical, mechanical, and thermal properties, comparable to imported products, thereby offering a viable alternative to expensive foreign materials. XRD analysis confirmed mullite as the dominant crystalline phase after firing at 1400°C, accompanied by secondary phases including corundum, tridymite, sillimanite, and minor silica phases.

SEM observations revealed an interlocking network of acicular mullite crystals embedded within a heterogeneous matrix, contributing to improved structural integrity and mechanical performance. The study demonstrated that increasing the smectite content resulted in a reduction in bulk density and refractoriness, while a slight improvement in mechanical strength was observed. While higher proportions of smectite additions (20 wt.%) caused increased shrinkage and lower thermal shock resistance, negatively affecting the overall refractory performance. Future research should focus on optimizing burning conditions and investigating long-term durability under industrial operating conditions to strengthen the commercial viability of this approach.

Author contributions: CRediT

Author 1 (Laith H. Mohammed): Data curation, Formal analysis, Investigation, Methodology, Software, Visualization, Writing – Original draft. Author 2 (Sawsan H. Al-Hazaa): Project administration, Supervision, Validation, Writing – review and editing.

Declaration of Competing Interests

The authors declare that they have no competing financial interests or personal relationships that could influence the work reported in this study.

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Data Availability

All data generated or analyzed during this study are included in this published article and/or its supplementary materials

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