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The Effect of Using Thermally Activated Metakaolin as an Additive on the Properties of Ordinary Portland Cement

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Abstract

Supplementary Cementitious Materials (SCMs) have become essential in the development of high-strength and high-performance concrete. These materials include naturally occurring pozzolans, industrial by-products, and low-energy materials that contribute to sustainable construction practices. This study examines the impact of incorporating thermally activated Iraqi metakaolin (MK) produced by calcining kaolinite clay at 600°C for 3 hours as a partial replacement for Ordinary Portland Cement (OPC) in mortars and concretes. The metakaolin cools rapidly to preserve its amorphous and reactive structure. Replacement ratios of 10%, 15%, 20%, and 25% by weight of cement are tested and compared with a control mix containing no MK. A comprehensive set of physical and chemical tests, including Blaine fineness, setting times, compressive strength, and chemical composition. The results indicate a notable enhancement in the mechanical and durability properties, especially at 10% replacement, which demonstrates optimal performance. Beyond technical improvements, the use of metakaolin also offers environmental advantages such as reduced clinker content, lower CO_2 emissions, and the utilization of abundant local resources. These findings position the Iraqi metakaolin as a promising SCM that aligns with global trends in green and sustainable construction, making it a practical and eco-friendly solution for modern cement-based systems.

Keywords:

Kaolin, Metakaolin, Cement, Ga'ara Formation, Compressive strength.

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1. Introduction

Increased demand for environmental sustainability and climate change has led the construction sector to find alternatives in order to minimize its carbon footprint. The cement sector alone contributes 7-8% of CO2 emissions (IEA, 2023), and hence there is ongoing research on Supplementary Cementitious Materials (SCMs) like metakaolin as a partial replacement of Ordinary Portland Cement (OPC). Among the numerous SCMs, metakaolin (MK), a thermally activated Iraqi kaolinite clay product of Iraq's Western Desert (Ga'ara Formation), is utilized because it is very fine in particle size and pure (Tammar-agha and Al-Hazaa, 2023). MK reacts with Calcium Hydroxide (CH) and forms cementitious products such as Calcium Silicate Hydrate (C-S-H) and Calcium Silicate Aluminate Hydrate (C-S-A-H) that improve strength and microstructure organization. Tests by Parande et al. (2008) and Akasha et al. (2008) measurements enhanced strength of MK cement pastes and mortars up to 20% replacement. More recent research (El-Dakroury and Gasser, 2020; Ghafoori et al., 2022) also attests to the benefits of MK for early strength and durability, particularly under severe exposure conditions.

The main goal of this study is to investigate the influence of adding thermally activated Iraqi metakaolin as an additive on the physical and mechanical performance of OPC-based mortar.

The study targets improving compressive strength, reducing the overall permeability of the concrete and making the mix design economical by reducing the amount of cement used. Also, it will assess how metakaolin enhances microstructural growth via pozzolanic reaction and overall contributes to the durability and performance of the end product.

2. Location and the Geology of the study area

Raw kaolin clay samples used for the current research were collected from Dweikhla mine (Ga'ara Formation), about 80 km north of Al-Rutba City, Al-Anbar Governorate, Iraqi Western Desert. The study area is located in the Ga'ara depression between longitudes (40° 20′ 00″ and 40° 22′ 00″) east and latitudes (33° 31′ 00″ and 33° 33′ 00″) north (Fig. 1).

The Ga'ara Formation is one of the oldest exposed geological units in the Iraqi Western Desert. It is located within an oval-shaped geological depression extending approximately 60 km east-west and 30 km north-south (Fig. 2), and is characterized by a well-defined succession of fluvial sedimentary units (Tamar-Agha and Al-Hazaa, 2023). This depression has been formed by deep erosional processes affecting the highest parts of the Rutba Uplift.

Tectonically, the area lies within the Stable Shelf of the Arabian Plate (Arabian Platform). During the Late Carboniferous to Early Permian period, this passive margin was influenced by Hercynian orogenic movements, which caused the uplift of the eastern and western margins of the Arabian Plate. These tectonic events led to the development of the Rutba Basin, which was subsequently filled with sediments attributed to the Ga'ara Formation (Jassim and Goff, 2006). During the Late Permian to Early Triassic, further uplift of the Rutba Basin resulted in the exposure of Permian rocks at the surface (Sissakian and Mohammed, 2007).

Geologically, the Ga'ara Formation comprises continental sediments deposited in a meandering fluvial system. It includes fining-upward sequences of quartz sandstone and kaolinitic clay beginning with coarse-grained channel-fill sandstones and transitioning upwards into finer clay-rich deposits that represent floodplain environments (Tamar-Agha and Al-Hazaa, 2023).

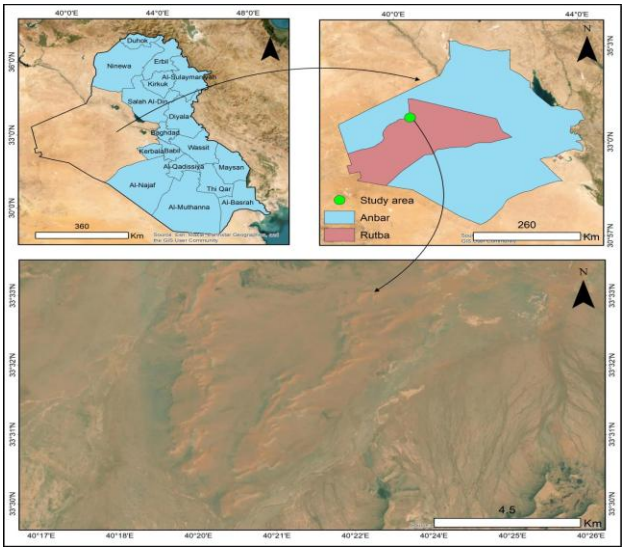

Fig.1: Location map of the study area, Dweikhla kaolin mine within the Ga'ara Formation.

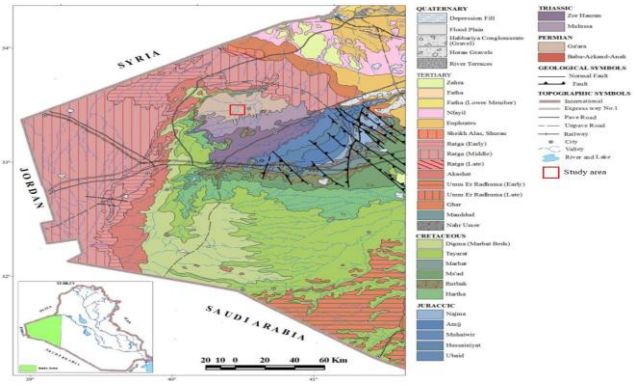

Fig. 2: Geological map of the study area, Dweikhla kaolin mine within the Ga'ara Formation, Western Desert of Iraq (modified after Sissakian, 2000).

3. Materials and Methods

A. Materials

Kaolin clay used in this study is sourced from the Dweikhla mine, part of the Ga'ara Formation in the Western Desert of Iraq. The clay is predominantly composed of kaolinite with minor constituents such as illite, quartz, iron oxides, titanium, and organic matter. A representative white kaolin sample obtained from the General Company for Geological Survey is thermally activated at 600 °C for three hours to produce metakaolin. This calcination process converted the crystalline kaolinite into an amorphous, highly reactive pozzolanic material, which is subsequently used as a partial cement replacement in the concrete mixtures.

Cement is defined in the general sense as a material with adhesive and cohesive properties that enable it to bind mineral fragments together

(Gambhir, 2004). It plays a vital role in the concrete industry due to its ability to set and harden underwater through a chemical reaction with water and is therefore classified as a hydraulic cement (Neville et al., 2012).

B. Methodology

Ordinary Portland Cement (OPC) used in this study is of type N32.5 supplied by the Rafidain Cement Factory located west of Mosul. The mixing process is conducted under controlled laboratory conditions at a room temperature of 20–25 °C and relative humidity between 50% and 65% in accordance with the Iraqi Standard Specification IQS No. 5/2019, which regulates the preparation and testing of Portland cement materials in Iraq.

The kaolinite clay used in this study is obtained from the Dweikhla mine located within the Ga'ara Formation in the Western Desert of Iraq (Al-Anbar Province). The raw material is initially crushed using a laboratory jaw crusher and subsequently ground into a fine powder. According to Vōgvölgyi et al. (2008) and Frost et al. (2004), the grinding process alters the thermal behavior of kaolin by lowering the dehydroxylation temperature, increasing surface area, and enhancing water adsorption due to the disruption of interlayer hydrogen bonds.

The ground kaolin is then thermally activated in a laboratory furnace at 600 °C for three hours and gradually cooled to room temperature, producing metakaolin (an amorphous and highly reactive pozzolanic material). This phase transformation results from the removal of hydroxyl groups and structural disordering. As noted by Kostuch et al. (2000), further calcination above 850 °C induces recrystallization leading to the formation of relatively inert ceramic phases such as spinel, silica and mullite (Bensted and Barnes, 2002). To confirm the transformation, X-ray diffraction (XRD) analysis is conducted on kaolin samples before and after calcination (Figs. 3 and 4).

The disappearance of kaolinite peaks post-calcination confirms the complete conversion into amorphous metakaolin. Five mix designs are prepared: one control mix with 100% OPC, and four mixes, in which OPC is partially replaced with metakaolin at levels of 10%, 15%, 20%, and 25% by weight (Table 1). The mixing process is conducted under controlled laboratory conditions at a room temperature of 20–25 °C and relative humidity of 50–65% following the Iraqi Standard Specification IQS No. 5/2019. Metakaolin is further ground to a particle size of 0.090 mm to ensure homogeneous blending. Each metakaolin ratio is mixed with OPC using a laboratory mixer for a total of 10 minutes, 5 minutes at high speed followed by 5 minutes at low speed. After mixing, chemical and physical tests are performed on both the control and blended cement samples.

C. Physical Examinations

Physical tests conducted in this study include Blaine fineness, initial setting time, final setting time, and compressive strength. A physical examination is conducted for the reference cement as given in Table (2). The setting times are determined using the Vicat apparatus according to ASTM C191 based on the standard consistency of the cement paste. The amount of mixing water is adjusted to meet the standard penetration requirements as detailed in Table (5). The compressive strength test is carried out in accordance with ASTM C109 using a mortar mix ratio of (1:3:0.5 of cement: sand: water) corresponding to 450 g of cement, 1350 g of standard silica sand, and 225 ml of water. Specimens are cast in molds and cured in a moist environment for 24 hours, then demolded and submerged in water for continued curing periods of 2, 28, and 60 days. These time points represent early strength, standard strength, and long-term reactivity, respectively. The results are shown in Table 6. All physical tests are conducted in accordance with the Iraqi Standard Specification No. 5 (2019), which aligns with ASTM C204 for fineness determination and other applicable ASTM standards. These tests are selected as they are commonly used to assess the durability and workability of cementitious materials in construction and aging studies.

D. Chemical Tests

The chemical composition of the unmixed cement is analyzed using standard gravimetric and titration methods, including Ethylene Diamine Tetra acetic Acid (EDTA) titration following the Iraqi Standard Specification No. 5 (2019). The analysis aims to determine the concentrations of major oxides such as SiO_2, SO_3, Fe_2O_3, CaO, Al_2O_3, MgO, and Loss on Ignition (L.O.I), as well as IR (Insoluble residue) as presented in Table (2). In addition, chemical analyses of both kaolin and the thermally activated metakaolin are conducted

to assess compositional changes resulting from calcination, with results summarized in Table (3)

Table 1: Ratios of mixing metakaolin with Portland cement.

Material	0% MK	10% MK	15% MK	20% MK	25% MK
Cement	1300 gm	1170 gm	1105 gm	1040 gm	**975 gm**
Metakaolin	0 gm	130 gm	195 gm	260 gm	**325 gm**

Table 2: Chemical and physical tests of the Cement Sample of Rafidain Cement Plant grade (32.5).

Chemical analyses			Compressive strength (MPa)		
CaO %	63.32			I.Q.S:5/2019 Limits	**Tested**
SiO_2 %	20.30		Days	2 Days	**18.55**
Al_2O_3 %	5.87			28 Days	**34.35**
Fe_2O_3 %	3.34			60 Days	**37.55**
SO_3 %	1.90	Max 2.8%			
MgO %	2.89	Max 5%	**Physical tests**		
Insoluble residue %	0.31	Max 1.5%	Fineness, Blaine method (g/cm^2) Standard		**2846**
*L.O.I%	1.09	Max 4%	Setting time	Initial (min) > 45	**105**
Total	98.71			Final (hrs.) < 10	**3:05**
Free CaO %	1.55		Soundness	Autoclave 0.8% Max	**0.17**
			Specific gravity		**3.13**
Abbreviation	Determined		Water for standard consistency (g) per 400 (g) of cement		**110**
C3S %	47.38				
C2S %	22.72				
C3A %	9.91				
C4AF %	10.15				

*L.O.I loss of ignition **L.S.F lime saturation factor

Table 3: Comparison between Raw Kaolin and Metakaolin (Based on Chemical Composition).

Oxide Component	Kaolin (%)	Metakaolin (%)	Observation
SiO_2	49.10	54.31	Increased due to the removal of volatile components
Al_2O_3	31.88	37.69	Increased due to dehydroxylation
Fe_2O_3	1.91	1.96	Slight increase
CaO	1.34	1.65	Slight increase
MgO	0.72	0.80	Slight increase
SO_3	0.60	0.72	Slight increase
L.O.I	13.14	1.92	Significant decrease due to loss of structural water
Total	98.69	99.05	Approaching full mass balance after calcination

4. Results and Discussion

A. Chemical analysis of Kaolin and Metakaolin

The rise in SiO_2 and Al_2O_3 content in metakaolin is due to the removal of physically and chemically bound water from kaolin during calcination, making it more reactive and amorphous. A sudden decrease in L.O.I confirms dehydroxylation of kaolinite for the formation of a pozzolanic active phase (metakaolin).

B. X-ray Diffraction Analysis

X-ray diffraction (XRD) analysis is conducted at Al-Khawrah Laboratory in Baghdad on raw kaolin and after calcination at 600 °C for three hours to confirm its transformation into metakaolin as illustrated in Figures (3 and 4). The XRD pattern of raw kaolinite shows well-defined crystalline peaks characteristic of the kaolinite phase. The most prominent peak appears at $2\theta \approx 12.4°$ corresponding to a d-spacing of 7.2 Å, which reflects the (001) plane. Another significant peak occurs at $2\theta \approx 24.9°$ with a d-spacing of 3.57 Å corresponding to the (002) plane. These peaks are commonly used as markers to identify kaolinite in clay samples. After calcination, these kaolinite peaks disappear, indicating the dehydroxylation and transformation into amorphous metakaolin. The resulting XRD pattern displays a broad diffuse hump between $2\theta \approx 20°$ and 30°, typical of an amorphous structure. This confirms that the

kaolinite structure has collapsed into a disordered and highly reactive pozzolanic phase.

Quartz is detected as a crystalline impurity in both raw kaolin and metakaolin samples, with its most prominent peak appearing at $2\theta \approx 26.6°$ corresponding to the (101) plane. Other minor quartz peaks may also appear at $2\theta \approx 20.8°$, 36.5° and 50.1°.

Upon calcination, the disappearance of these kaolinite peaks indicates the complete structural transformation into amorphous metakaolin. Since amorphous materials do not exhibit sharp diffraction peaks, the XRD pattern of metakaolin presents a broad diffuse hump in the $2\theta = 20°–30°$ range, which is a hallmark of its non-crystalline nature.

The structural transformation can be represented by the following dehydroxylation reaction:

$$Al_2Si_2O_5(OH)_4 \rightarrow Al_2Si_2O_7 + 2H_2O$$

This reaction describes the loss of hydroxyl groups from the kaolinite structure leading to the formation of an amorphous aluminosilicate phase with high pozzolanic activity. Minor residual crystalline peaks may still appear, particularly that of quartz at $2\theta \approx 26.6°$ as quartz remains stable and crystalline under the applied thermal conditions (600 °C).

The conclusion is that the sample consists of relatively pure metakaolin. Calcination at 600 °C for 3 hours is sufficient to transform kaolinite into an amorphous metakaolin phase. Quartz remains as a stable crystalline impurity. The absence of peaks corresponding to new crystalline phases confirms that the transformation was purely thermal and occurred without any recrystallization.

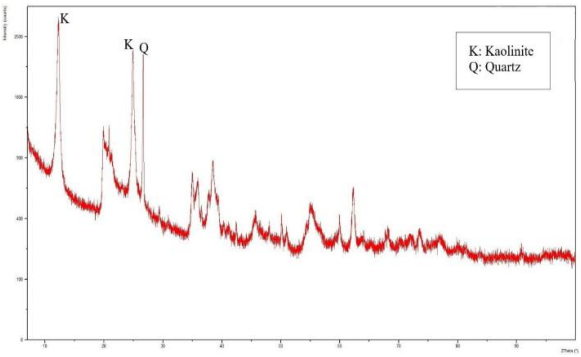

Fig. 3: X-ray diffractogram of the kaolin sample of the Dweikhla mine in western Iraq.

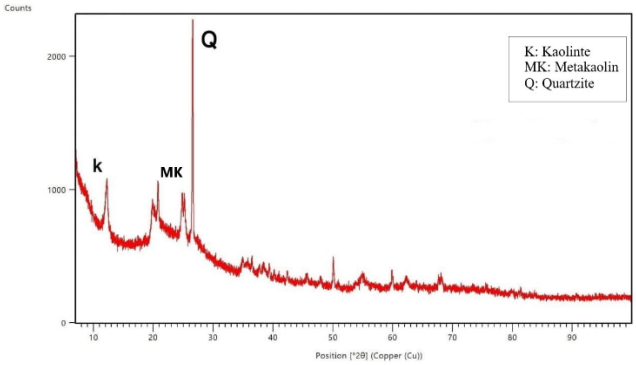

Fig. 4: X-ray diffractogram of thermally activated metakaolin.

C. Chemical Examination of Each Mixture Ratio

A comparative chemical analysis is performed to evaluate the effect of metakaolin (MK) on the oxide composition of cement mortar mixtures. Five mixes are examined: one reference mix made with ordinary Portland cement (OPC) from Rafidain Cement Factory, and four mixes in which OPC was partially replaced with metakaolin at varying ratios. The concentrations of major oxides such as SiO_2, Al_2O_3, Fe_2O_3, CaO, and others are determined for each mixture to assess the chemical changes resulting from MK incorporation. The results of the chemical analysis for all mixtures are presented in Table (4).

D. Chemical Composition of Cement Mortars Incorporating Thermally Activated Metakaolin

Table (4) illustrates the chemical composition of cement mortar mixtures incorporating various proportions of thermally activated metakaolin (MK) and compares it with a control mix composed solely of ordinary Portland cement (Rafidain cement). The results reveal several compositional trends linked to the progressive addition of metakaolin to the cement blends.

The results of the chemical tests reveal several trends associated with the progressive incorporation of metakaolin into the cement mixtures. Silica oxide (SiO_2) exhibited a noticeable increase with higher metakaolin replacement levels. This is attributed to the inherently high siliceous content of metakaolin, which supplements the silica already present in the reference cement. The increased silica content enhances the formation of secondary calcium silicate hydrate (C–S–H) through pozzolanic reactions, thereby improving the microstructure and overall durability of the hardened matrix.

Table 4: Chemical test results of reference cement and cement mixed with metakaolin.

Oxids	Cement Control	Cement + 10% MK	Cement + 15% MK	Cement + 20% MK	Cement + 25% MK
SiO_2	20.30	23.93	25.88	27.80	**28.50**
Al_2O_3	5.87	9.08	11.13	11.56	**13.83**
Fe2O3	3.34	3.45	3.27	3.39	**3.21**
CaO	63.32	56.26	52.75	50.58	**47.41**
MgO	2.89	2.64	2.52	2.52	**2.52**
SO_3	1.9	1.84	1.91	1.78	**1.66**
L.O.I	1.09	1.44	1.42	1.14	**2.32**
TOTAL	98.71	98.64	98.88	98.77	**99.45**
Fr.L	1.55	0.74	0.72	0.68	**0.63**
C3S	47.38				
C2S	22.72				
C3A	9.91				
C4AF	10.15				
In.R	0.31				
SiM	2.20				
HM	2.14				
AM	1.75				
L.S.F	93				

Fr. L: Free Lime, L.S.F: Lime Saturation Factor, AM: Alumina Modulus, HM: Hydraulic Modulus, SiM: Silica Modulus, In. R: Insoluble Residue

In contrast, aluminum oxide (Al_2O_3) shows a slight decrease as the metakaolin content increases. Although metakaolin itself contains aluminum, its reactivity with calcium hydroxide ($Ca(OH)_2$) and its role in forming calcium aluminosilicate hydrate (C–A–S–H) likely contribute to this reduction, as some aluminum becomes chemically bound within the hydration products.

Calcium oxide (CaO) shows a declining trend as metakaolin content increased. This is explained by both the lower CaO content in metakaolin and the pozzolanic reaction, which consumes $Ca(OH)_2$ during hydration. The reduction in CaO alters the hydration dynamics and lowers portlandite formation, thereby promoting pozzolanic activity.

Magnesium oxide (MgO) levels decline across the mixes as well. Since the MgO content in metakaolin is low, this decrease is expected and generally considered beneficial, given the potentially harmful effects of excess MgO on concrete performance. Similarly, iron oxide (Fe_2O_3) is also decreased with the increased metakaolin dosage, reflecting its relatively low presence in the metakaolin source used.

Loss on ignition (L.O.I) values slightly increase with metakaolin incorporation but remain within the acceptable specification limit of 4%. This increase may result from residual bound water and volatile components remaining in the thermally activated metakaolin.

Lastly, sulfur trioxide (SO_3) concentrations fluctuate slightly among the mixtures, but all values stay within permissible limits. This indicates that the addition of metakaolin does not significantly affect sulfate levels and is unlikely to introduce any sulfate-related durability concerns.

E. Physical Properties of Cement Mortars Containing Thermally Activated Metakaolin

Table (5) presents the physical properties of the cement mortars, including Blaine fineness, standard water consistency, and both initial and final setting times. The results reveal distinct trends associated with the incorporation of thermally activated metakaolin (MK) into the binder system.

Table 5: Fineness values, water ratio, initial and final setting times for reference cement and cement mixes with metakaolin.

Samples	Cement + 0% MK	Cement + 10% MK	Cement + 15% MK	Cement + 20% MK	Cement + 25% MK
Fineness	2846	4633	5158	5692	**7800**
W/C Ratio	110 ML	112 ML	114 ML	117 ML	**118 ML**

Initial Setting	105 MIN	75 MIN	60 MIN	45 MIN	**30 MIN**
Final Setting	3:05 H	2:40 H	2:55 H	3:00 H	**2:00 H**
W/C Ratio	225 ML	225 ML	225 ML	225 ML	**235 ML**

A significant increase in fineness is observed as MK content increased, reaching up to 7800 g/cm² at 25% MK replacement when compared to 2846 g/cm² in the control mix. This enhancement is attributed to the extremely fine particle size of metakaolin, which acts as a micro-filler, accelerating the hydration process and contributing to the formation of a denser microstructure with reduced porosity. As supported by Shetty (2009), increased fineness leads to a larger surface area, promoting rapid water interaction and early strength development.

The setting times of cement mortars are also notably affected by MK incorporation. The initial setting time is decreased progressively from 105 minutes in the control mix to 45 minutes at 25% MK content. This reduction is due to the high surface area and fine particle size of MK, which enhances water absorption and accelerates the hydration reaction. According to Shetty (2009), hydration begins on the surface of cement particles and proceeds inward, and smaller particles reduce the diffusion path of water, thereby shortening the setting time.

Furthermore, the pozzolanic activity of metakaolin through its consumption of calcium hydroxide {$Ca(OH)_2$} and formation of additional calcium silicate hydrate (C–S–H) improves the microstructure and reduces setting time. El-Diadamony et al. (2018) also reported that thermally activated kaolinite contributes to improve early-age compressive strength and enhance hydration kinetics. All measured values are within the acceptable limits of the Iraqi Standard Specification No. 5 (2019).

(Table 6) displays the compressive strength results at 2, 28, and 60 days of curing, along with results of stability and soundness testing using the autoclave method.

For each substitution level (0%, 10%, 15%, 20%, and 25% metakaolin), three replicates were tested to ensure statistical reliability, and the average values are presented in Table (6). The results demonstrate a general trend of increased compressive strength with the addition of metakaolin, particularly at the 10% replacement level, which yielded the highest compressive strength across all curing ages (2, 28, and 60 days).

This observation aligns with the findings of Gold and Shirvill (1992), who reported that the inclusion of metakaolin led to significant improvements in compressive strength, with an optimal substitution range between 5% and 10%. They also noted that increasing the MK content beyond this range did not result in further strength gains. The enhancement in strength is primarily attributed to the pozzolanic reaction between metakaolin and calcium hydroxide {$Ca(OH)_2$} forming additional calcium silicate hydrate (C–S–H) gels. These gels contribute to a denser microstructure, reduced pore connectivity, and improved cohesion within the cement matrix.

The increased fineness of metakaolin further amplifies this effect by reducing porosity and providing nucleation sites for hydration products. However, at 25% MK replacement, a slight decrease in compressive strength is observed. This decline is linked to the elevated water demand associated with the very fine particle size of MK, which increases the water-to-cement ratio, raises porosity due to water evaporation and introduces microstructural defects in the hardened mortar.

In terms of soundness, which reflects the volumetric stability of the hardened cement paste, the results show a noticeable improvement with metakaolin addition. The soundness value is decreased from 0.17% in the control mix to 0.10% at 10% MK replacement. This indicates a reduction in expansive compounds such as free lime and MgO, which are commonly responsible for delayed expansion and cracking in concrete. All values recorded are well below the permissible limit of 0.8%, as specified by relevant standards, confirming the compatibility of MK with OPC in terms of dimensional stability.

Table 6: Compressive strength and Soundness values of reference cements with cement mixes mixed with metakaolin.

Samples	**Cement + 0% MK**	**Cement + 10% MK**	**Cement + 15% MK**	**Cement + 20% MK**	**Cement + 25% MK**

Compressive strength 2 Days	18.55 MPA	23.7 MPA	21.3 MPA	17.9 MPA	**15.25MPA**
Compressive strength 28 Days	34.35 MPA	39.4 MPA	37.85 MPA	35.2 MPA	**29.3 MPA**
Compressive strength 60 Days	37.55MPA	42.14 MPA	38.58 MPA	37.7 MPA	**34.9 MPA**
Soundness	0.17	0.1	0.14	0.14	**0.14**

Finally, water demand (W/C ratio) increases progressively with MK content from 110 mL in the control mix to 118 mL at 25% MK. This increase is attributed to the higher surface area of MK particles, which absorb more water during mixing. While elevated water demand is typical for highly reactive pozzolanic materials, it must be carefully managed during mix design to avoid negative impacts on workability and strength development.

F. X-Ray Diffraction (XRD) Analysis of Cement Mortar Sample at 28 Days

X-ray diffraction (XRD) analysis is conducted for a cement mortar sample prepared using 32.5 normal Portland cement (Rafidain Cement Factory) and 10% thermally activated metakaolin, as shown in Figure (5).

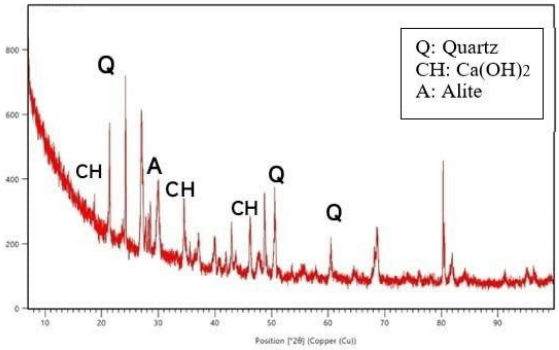

Fig. 5 : XRD diffractogram of cement mortar sample at 28 days.

G. Diffraction Interpretation and Evaluation of Mixing Ratios

The 10% metakaolin replacement level is selected based on previous chemical and physical results, which have demonstrated a superior performance in compressive strength, setting time, and pozzolanic reactivity. The XRD pattern of the cement mortar containing 10% metakaolin reveals a broad amorphous hump in the range of $2\theta = 20°–35°$, indicating the formation of poorly crystalline hydration products. This amorphous phase is primarily attributed to the development of calcium silicate hydrate (C–S–H) and calcium alumino-silicate hydrate (C–A–S–H) gels resulting from the pozzolanic reaction between metakaolin and calcium hydroxide.

The presence of this diffuse hump suggests ongoing gel formation and matrix densification, which are characteristic features of enhanced durability and strength development (Shaikh and Supit, 2015; Kim and Lee, 2020). According to Hwalla et al. (2025), metakaolin-based binders exhibit superior gel formation and reduced permeability, even under aggressive conditions such as underwater exposure, confirming their structural integrity.

Sharp crystalline peaks are observed at $2\theta = 26.6°, 20.8°, 36.5°, 50°, 59°$ and $60°$ corresponding to quartz. These peaks arise from both the inert quartz in metakaolin and the natural sand used in the mortar mix. Although quartz does not participate chemically in hydration, it contributes mechanically to the overall dimensional stability and skeletal structure of the hardened paste (Vejmelková et al., 2010).

In addition, residual peaks of calcium hydroxide (portlandite) are noted at $2\theta = 18.1°$, $34.1°$ and $47°$, although with relatively low intensity, indicating that the majority of $Ca(OH)_2$ had reacted with metakaolin—a confirmation of the high pozzolanic reactivity (Sabir et al., 2001).

No identifiable peaks of ettringite are detected in the region of $2\theta = 9°–15°$, likely due to the low gypsum content in the cement or the overlapping of their peaks with the broader amorphous hump typical of hydrated gel phases (Fernandez et al., 2011). Additionally, minor peaks in the $2\theta = 29°–32°$ range indicate the presence of unreacted cement phases, particularly alite (C_3S) and belite (C_2S). These signals suggest imperfect hydration, possibly due to mixing inefficiencies or water retention within fine particles of metakaolin and sand (Abulnour and Elgamouz, 2018).

Based on this XRD analysis, the 10% MK replacement is confirmed to enhance amorphous gel formation, reduce unreacted $Ca(OH)_2$, and promote a denser and stronger microstructure. This finding supports the conclusions of several contemporary studies (Hwalla et al., 2025).

H. Evaluation of Mix Performance and Metakaolin Role

The experimental results affirm that a 10% replacement of OPC with metakaolin yields optimal performance. This ratio provided the highest compressive strength values across all curing periods. Although a 15% replacement also resulted in strength improvement, it is slightly less effective than the 10% mix.

Metakaolin plays a dual role in cement matrices: as a pozzolanic material and as a micro-filler. Due to its ultra-fine particle size, approximately ten times finer than that of cement, it fills voids between larger particles, increasing packing density and reducing porosity (Morsy et al., 1997). Its reactivity with $Ca(OH)_2$ leads to the generation of additional C–S–H, which contributes to improved durability, reduced permeability, and long-term strength enhancement (Wang, 2004).

However, higher replacement levels (20% and 25%) show diminishing returns. This reduction in performance is attributed to insufficient $Ca(OH)_2$ availability causing metakaolin to behave more like an inert filler than an active pozzolan. Moreover, higher MK content necessitates an increased water-to-cement ratio, which raises porosity and hinders strength development (Morsy et al., 1997; Wang, 2004).

Finally, the interplay between chemical modifications and physical performance becomes apparent when the dosage of MK is finely tuned. The enhancement in SiO_2 and Al_2O_3 content is only effective when sufficient $Ca(OH)_2$ is available to generate reaction products that improve compressive strength. Similarly, chemical stability improvements through reduced CaO and MgO content contribute to long-term durability, but only if they do not lead to excessive porosity due to higher water demand. Thus, the optimal performance of cement paste modified with metakaolin is only achieved through a precise balance between its chemical composition and physical behavior as confirmed by the optimal 10% MK replacement range.

5. Conclusion

1. Essential Thermal activation: The raw Iraqi kaolin clay obtained from the Dweikhla quarry in the Al-Rutba region is not directly usable as a supplementary cementitious material. However, after calcination at 600 °C for 3 hours, the kaolinite is fully transformed into highly reactive metakaolin, rendering it suitable for cement substitution due to its significant pozzolanic activity.
2. Pozzolanic reactivity and strength development: The metakaolin synthesized under the optimum thermal conditions exhibited enhanced compressive strength in cement mortars. This improvement is attributed to the high content of reactive silica and alumina, which react with calcium hydroxide $Ca(OH)_2$ to form additional C–S–H and C–A–S–H gels, thereby densifying the microstructure.
3. Optimum replacement level of 10%: Among all tested mix ratios (10%, 15%, 20%, and 25%), the 10% metakaolin replacement achieved the highest compressive strength across all curing ages, with an efficient balance between water demand, workability, and mechanical performance.
4. Water demand considerations: Due to its high fineness and surface area, metakaolin increases the water demand of the cement mix. However, the 10% MK replacement level required the least additional water, maintaining a favorable water-to-cement ratio and avoiding excessive porosity.
5. Early strength and fast setting: The incorporation of metakaolin contributes to early-age strength development and accelerates setting time, making it suitable for applications requiring rapid demolding and early loading.
6. Dimensional and volumetric stability: The addition of metakaolin improved the soundness of the mix, reducing the risk of expansion from free lime and MgO and remaining within the acceptable limits per Iraqi and ASTM standards.
7. Environmental and technical benefits: The use of thermally activated metakaolin offers an eco-friendly solution to reduce cement clinker content and CO_2 emissions, while simultaneously improving long-term durability and sustainability of cement-based systems.
8. Recommendation for practical use: These findings support and encourage the partial replacement of ordinary Portland cement with locally sourced metakaolin, particularly at 10% dosage for the production of high-

performance and durable concrete in Iraqi construction practices.

9. The XRD pattern exhibited a broad hump at 2θ = 20°–35°, confirming the formation of amorphous phases such as calcium silicate hydrate (C–S–H) and calcium alumino-silicate hydrate (C–A–S–H) due to pozzolanic reaction. The relatively low intensity of portlandite $Ca(OH)_2$ peaks further indicates a high pozzolanic activity of metakaolin enhancing the matrix densification. These results confirm that the 10% metakaolin replacement provides optimum performance, balancing strength, durability, and water.

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تأثير استخدام الميتاكاؤولين المنشَّط حرارياً كمادة مضافة على السمنت البورتلاندي الاعتيادي

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الملخص

أصبحت المواد الأسمنتية الإضافية (SCMs) عنصرًا أساسيًا في تطوير الخرسانة عالية المقاومة والأداء العالي. وتشمل هذه المواد البوزولانات الطبيعية، والمخلفات الصناعية، والمواد ذات الطاقة التصنيعية المنخفضة، مما يساهم في ممارسات البناء المستدام. تتناول هذه الدراسة تأثير استخدام الميتاكاولين العراقي المنشَّط حراريًا، والذي يتم الحصول عليه من خلال معالجة طين الكاؤولين حرارياً عند درجة حرارة 600°C لمدة ثلاث ساعات، ثم تبريده سريعًا للحفاظ على حالته غير المتبلورة التفاعلية، كمادة بديلة جزئية عن السمنت البورتلاندي الاعتيادي (OPC) في الخلطات الخرسانية والمونة. تم اختبار نسب استبدال مختلفة بواقع 10%، 15%، 20%، و25% من وزن السمنت، وتمت مقارنتها مع خلطة مرجعية لا تحتوي على أي إضافة. أُجريت مجموعة من الاختبارات الفيزيائية والكيميائية، شملت نعومة Blaine، زمنَي التصلب الابتدائي والنهائي، مقاومة الانضغاط، والتحليل الكيميائي للاكاسيد الرئيسية. أظهرت النتائج تحسنًا ملحوظًا في الخصائص الميكانيكية والمتانة، خاصةً عند نسبة استبدال 10%، والتي سجلت أفضل أداء. بالإضافة إلى التحسينات الفنية، يقدم استخدام الميتاكاؤولين فوائد بيئية مهمة، مثل تقليل نسبة الكلنكر، وخفض انبعاثات ثاني أكسيد الكربون، والاستفادة من الموارد المحلية الوفيرة. وبذلك، يُعد الميتاكاؤولين العراقي مادة بوزولانية واعدة تتماشى مع الاتجاهات العالمية نحو البناء الأخضر والمستدام، مما يجعله حلاً عمليًا وصديقًا للبيئة في أنظمة السمنت الحديثة.

الكلمات المفتاحية:

كاولين، ميتاكاولين، سمنت، تكوين الكعره، قوة الانضغاط.

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