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RESEARCH ARTICLE

ANALYSIS OF MORTAR SAMPLES AND MINERAL COMPOSITION FROM DEIR EL-KAHF, JORDAN

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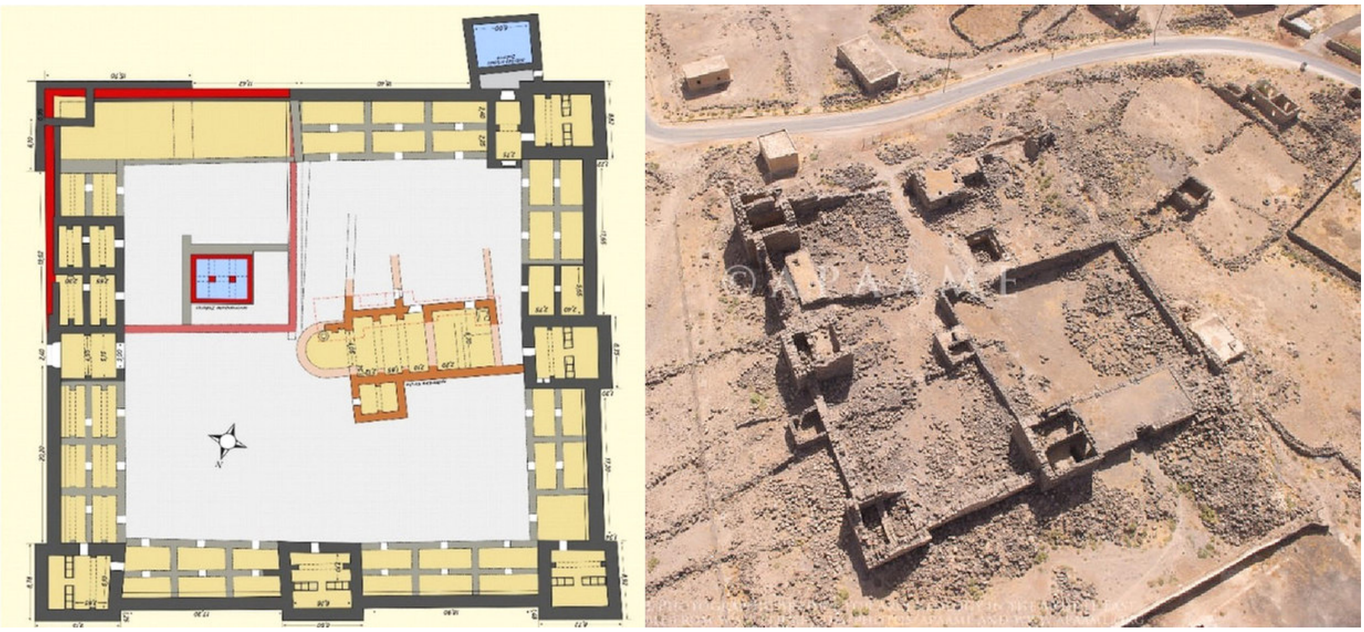

Figure 1. Site plan (Arce 2010) and aerial view of the site (APAAME).

ABSTRACT*. This study presents a detailed investigation of limestone mortar samples collected from three distinct zones within the Deir el-Kahf archaeological site in Jordan. The research objectives include the characterization of physical and chemical properties, evaluation of compositional homogeneity, and correlation of material properties with historical construction practices. Advanced analytical techniques of X-ray diffraction* (XRD) *and X-ray fluorescence* (XRF) *were employed to determine mineralogical phases and elemental composition. Results indicate a high degree of compositional consistency across samples, dominated by calcite* ($CaCO_3$) *derived from lime binders and quartz* (SiO_2) *aggregates. Minor variations in aggregate size and binder-to-aggregate ratios reflect localized adaptations in material sourcing or construction techniques. The integration of petrographic observations with chemical data underscores the effectiveness of combined analytical approaches in archaeological material studies. These findings provide critical insights for restoration strategies, emphasizing the need for compatible materials in conservation efforts. Future studies*

Received: December 20, 2024. Modified: January 16, 2025. Accepted: February 3, 2025. Published: February 11, 2025.

Edited & Published by Pascual Izquierdo-Egea [P. I. Egea]. Arqueol. Iberoam. Open Access Journal.

should incorporate advanced imaging techniques, such as scanning electron microscopy (SEM)*, and isotopic analysis to further elucidate material provenance and technological evolution.*

KEYWORDS. *Deir el-Kahf, lime mortars, chemical characterization, Roman archaeology, Jordan.*

INTRODUCTION: GEOGRAPHICAL AND HISTORICAL CONTEXT OF DEIR EL-KAHF

The study area is situated in the modern village of Deir el-Kahf, approximately 45 kilometers east of the city of Mafraq, Jordan. The village is located about 13 kilometers north of the main highway connecting Safawi and Mafraq. Within Deir el-Kahf lies an archaeological site of the same name, featuring a structure built from black basalt stone—a characteristic element of the region's ancient architectural heritage. To the north of the site, a cluster of caves dating back to the Nabataean period highlights the area's historical and cultural significance.

While the precise origin of the name *Deir el-Kahf* remains uncertain, another site in Jordan, *al-Raqim*, near Amman, shares the same name. This second site is traditionally associated with the Qur'anic narrative of the *Young Men of the Cave*, adding to the religious and historical importance of both locations (Tasrif 2024). The site of Deir el-Kahf is believed to have originally been referred to as *Speluncae* and was constructed during the second century AD (Brünnow 1907). Strategically positioned along the Roman road linking the Azraq castle to southern Syria, the settlement played a crucial role as a transit and military outpost. The structure, built from black basalt, stands on relatively flat terrain within a semi-arid environment. Despite these harsh climatic conditions, the local population developed an advanced water management system, demonstrating their ingenuity and adaptability in sustaining life. David L. Kennedy identified this Roman road as the *Via Severiana*, named after the Severan dynasty to distinguish it from other major roads in the Levant (Kennedy 2004).

Historical records suggest that this route was constructed between AD 208 and 210, during the reign of Emperor Septimius Severus (AD 193–211). To support Roman military logistics along this route, watchtowers and fortified structures, such as *Deir el-Qinn*, were strategically erected. One such tower, positioned approximately 11 kilometers northeast of Deir el-Kahf, further illustrates the region's military significance. The landscape surrounding Deir el-Kahf rises approximately 500 meters from the prominent fortress of *Qasr al-Azraq*, which lies about 55 kilometers to the south. To the northwest, approximately 45 kilometers away is the site of *Bostra*, which served as a major Roman legionary base, as well as the *Strata Diocletiana*, a critical defensive line of the Roman Empire. Additionally, an essential Roman border road extended west of Deir el-Kahf, providing a direct connection to the Al-Azraq oasis. This extensive network of roads and fortifications underscores the strategic role of Deir el-Kahf in facilitating communication, trade, and defense across the Roman frontier (Arce 2010).[1]

RESEARCH HISTORY OF DEIR EL-KAHF

The site of Deir el-Kahf has been a focal point for Western travelers and researchers for over a century, contributing significantly to the documentation and understanding of its historical and archaeological significance. Below is a chronological summary of key visits and studies:

— 1898. German scholars Rudolf E. Brünnow and Alfred v. Domaszewski conducted research trips to Deir el-Kahf in April, documenting the site in detail (Brünnow & Domaszewski 1909).

— 1899. René Dussaud and Frédéric Macler, French orientalists, explored the area, contributing to the knowledge of its archaeological features and inscriptions (Seyrig 1959).

— 1904. Princeton University's Second Syria Expedition led by Howard Crosby Butler. This expedition included topographer Enno Littmann, who translated inscriptions from the Deir el-Kahf fort. The team noted significant damage caused by local inhabitants repurposing stone for construction (Butler & Littmann 1905).

— 1909. Howard Crosby Butler produced the first comprehensive architectural plan of the fort, which remained the authoritative reference for nearly a century.

[1] Ignacio Arce's research is regarded as one of the most significant contributions to the study of the site, providing valuable insights from both engineering and historical perspectives.

— 1939. Aurel Stein and Royal Air Force Aerial Surveys: Austrian-British archaeologist Aurel Stein visited Deir el-Kahf while documenting the Roman *Limes* (frontier defenses) in Jordan and Iraq. Aerial photographs taken by the Royal Air Force that same year captured the site's condition before extensive modern modifications by Druze families.
— 1950. Frederick Victor Winnett. This Canadian archaeologist visited Deir el-Kahf as part of an epigraphic study but found that inscriptions documented in 1904 had since been removed (Winnett 1951).
— 1976. Survey of the *Limes Arabicus* (Samuel Thomas Parker): Parker led a survey funded by the American Schools of Oriental Research, focusing on Roman border fortifications. His team collected ceramic materials and analyzed the defensive architecture of Deir el-Kahf (Parker & McDermott 1978).
— 1978. David L. Kennedy conducted a brief site visit while studying Roman military installations in northeastern Jordan. He later established the Aerial Photographic Archive for Archaeology in the Middle East (APAAME), significantly advancing the understanding of regional archaeology.
— 1980. Geoffrey R.D. King, British art historian and archaeologist, visited Deir el-Kahf as part of his research on Byzantine and Islamic-era archaeological features in the Hauran (King 1982).
— 2010. Ignacio Arce and the German-Jordanian University: Under the leadership of Ignacio Arce, researchers examined Deir el-Kahf as part of a broader project investigating construction techniques from Late Antiquity to the Early Islamic period. Arce is credited with confirming the existence of a Severan period fort at the site, a long-suspected but previously undocumented feature (Arce 2010).

CONSTRUCTION HISTORY

During the reign of the *Severi*, the initial fort was constructed adjacent to the *Via Severiana,* which was built concurrently. This early fortification, a small, towerless square fort (Figure 1), covered approximately one-third of the area of the later *Quadriburgus*.

The surrounding wall of the fort, 0.90 meters thick, was constructed using meticulously placed basalt masonry with narrow joints, reflecting advanced engineering precision for the time. The fort's northeast-southwest orientation resulted in a square layout measuring approximately 28 × 28 meters (0.08 hectare) (Arce 2010). The single entrance was located on the northeast wall. Based on Ignacio Arce's research, this early fortification included a cistern placed in the central courtyard, a feature that remains visible today. The cistern roof was supported by two central square pillars and corbels.

Arce proposed that the basalt used for the fort's construction was partially sourced from the excavation of the water reservoir, which was positioned in the northwest corner of the fort. During the late Roman period, the southwest and northwest walls of the Severan fort were dismantled to make way for the construction of the larger *Quadriburgus*. However, the northeast and southeast walls were partially incorporated into the new fortification (Arce 2010).

ARCHAEOLOGICAL ASSESSMENT AND MATERIAL ANALYSIS OF THE SITE

Following a comprehensive cleaning operation and partial reconstruction of walls previously damaged by various initiatives undertaken by the Jordanian Department of Antiquities, several significant features of the site were revealed.

Notably, a pond or water tank situated near the current entrance was uncovered. The eastern section of the site, adjacent to a nearby side street, was designated as the visitor entrance, as illustrated in the accompanying site plan. However, large portions of the structure remain in a state of disrepair and require further cleaning and stabilization efforts. Historical accounts, obtained through interviews with local residents, suggest that the site has experienced multiple phases of habitation.

Oral histories indicate that after 1923, Druze settlers from Syria, particularly the *Deir el-Qinn* region, occupied the site. In subsequent years, the site was repurposed for agricultural activities, functioning as a sheep pen and a storage area for fodder and grains. Structural observations revealed that the majority of the internal walls were coated with a mixture of mud and straw, a common construction material in the region. However, cleaning efforts exposed significant portions of the walls covered with lime mortar, an important architectural feature warranting further investigation. To analyze the composition and historical context of the lime mortar, three distinct samples were collected from different rooms within the site:

Figure 2. Samples from site 1, S1a (left) and S1b (right), obtained from the room located at the southernmost corner of the site, characterized by two arches. The sample was taken from the highest point of the room to ensure a representative and uncontaminated sample.

Figure 3. Samples from site 2, S2a (left) and S2b (right), collected from an adjacent room, specifically at the same height level as Sample 1, to minimize the influence of external contaminants such as dust or mud that might have accumulated on the surface.

Figure 4. Sample from site 3 (S3), extracted from a semi-demolished room near the water tank at the entrance. This area has not undergone comprehensive excavation, and the sample was collected with caution to preserve its integrity, despite the room's unstable condition.

The samples (Figures 2–4) will undergo laboratory analysis to determine their mineralogical and chemical compositions, shedding light on the construction techniques and materials employed at the site. Preliminary observations suggest that the lime mortar may contain regional aggregates and additives, reflecting local construction practices. These findings contribute to a deeper understanding of the site's historical usage, architectural evolution, and its role within the broader sociocultural and economic landscape of the region. Future work should focus on further excavation, detailed architectural documentation, and comparative studies of material compositions to contextualize the site's development across different historical periods.

RESULTS OF CHEMICAL ANALYSIS USING XRD AND XRF METHODS

This study presents the results of chemical analyses conducted on mortar samples using X-ray diffraction (XRD) and X-ray fluorescence (XRF) techniques. The analysis provides valuable insights into the composition and structural properties of the mortar used in the building and highlights areas requiring restoration.

1. Dominant Mineral Composition

The primary mineral in the analyzed samples is *Calcite* ($CaCO_3$). The highest concentration was observed in sample S3 (95.8%), while the lowest was 53.4% (S2a).

This variability in calcite content reflects differences in material composition and possible degradation processes across different areas of the site.

2. Quartz Silica

Quartz Silica (SiO_2) were present in all samples as a significant component of the clay used in the mortar. Sample 1C showed the highest silica content at 11.8%, whereas other samples exhibited lower values ranging from 1.7% to 2%.

3. Key Oxides and Their Reactions

The main chemical oxides found in the mortar include *Calcium Oxide* (CaO), *Aluminium Oxide* (Al_2O_3), *Iron Oxide* (Fe_2O_3), and *Silicon Oxide* (SiO_2).

These oxides undergo reactions during the mortar's melting stage, forming compounds critical to its structural integrity:

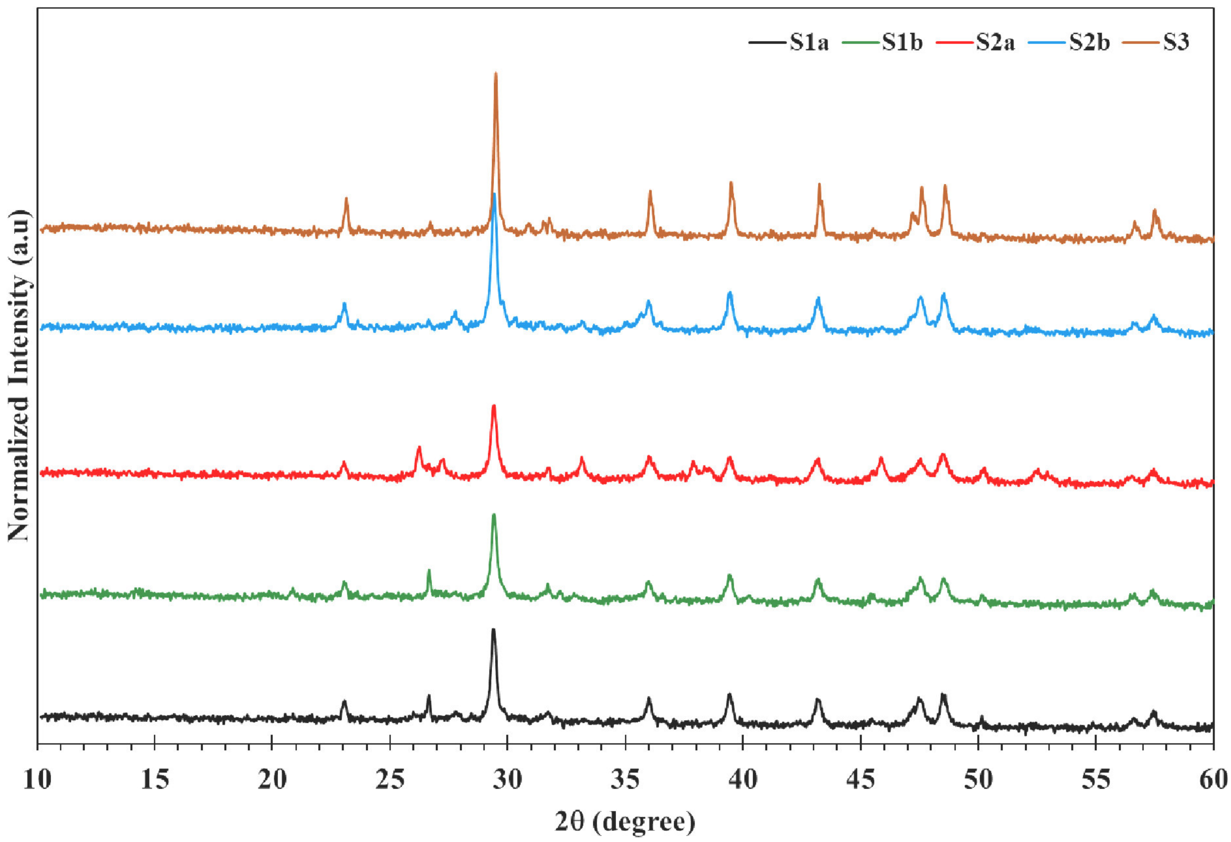

Figure 5. XRD diffractogram of mortar samples.

Table 1. Chemical composition of the mortar samples.

	Mineral	Chemical formula	Weight %				
			S1a	S1b	S2a	S2b	S3
	Aragonite	$CaCO_3$	3.4	1.4	38	1.3	0.0
	Calcite	$CaCO_3$	82	82	53	70	96
	Quartz	SiO_2	5.2	6.3	5.2	2.0	3.9
Plagioclase	Labradorite	$(Na,Ca)(Al,Si)_4\,O_8$	2.1	0.0	0.9	7.2	0.1
Plagioclase	Oligoclase	$(Na,Ca)(Al,Si)_4\,O_8$	2.0	1.7	0.3	0.0	0.0
	Dicalcium silicate	Ca_2SiO_4	2.6	6.1	2.1	13	0.1
Pyroxene	Augite	$(Ca,Mg,Fe)(Al,Si)_2\,O_6$	2.6	2.5	0.0	0.6	0.0

3.1 Tetra Calcium Alumina Ferrite (C_4AF)

Produced by reactions involving iron oxide, aluminium oxide and calcium oxide ($4CaO{\cdot}Al_2O_3{\cdot}Fe_2O_3$). This compound contributes to the mechanical stability and resistance of the mortar.

3.2 Dicalcium Silicate (C_2S)

Formed by reactions between calcium oxide and silica oxide ($2CaO{\cdot}SiO_2$). This compound is essential for high strength and durability, particularly against sulfate salts and weathering. Sample S2b contained the highest C_2S

Table 2. XRF for samples collected from different sites S1 (a and b), S2 (a and b), and S3, respectively.

	S1a			S1b		S2a		S2b		S3
Element	%	(+/-)	%	(+/-)	%	(+/-)	%	(+/-)	%	(+/-)
Al	18.89	1.1	15.96	1.23	12.39	1.07	18.46	1.34	21.32	1.23
Si	39.12	1.01	47.58	1.24	36.71	1.06	33.18	1.15	44.17	1.13
S	6.92	0.20	10.31	0.27	7.58	0.24	6.89	0.25	7.99	0.22
Ti	1.49	0.49	1.50	0.63	0.83	0.49	1.57	0.54	1.28	0.46
Cr	0.32	0.11	0.20	0.11	0.29	0.12	0.32	0.15	0.20	0.08
Mn	0.20	0.09	0.12	0.1	0.25	0.11	0.14	0.11	0.12	0.06
Fe	13.65	0.58	7.38	0.57	13.53	0.68	18.98	0.81	10.51	0.52
Ni	0.40	0.10	0.32	0.10	0.54	0.12	0.63	0.13	0.31	0.08
Cu	0.33	0.07	0.01	0.10	0.34	0.08	0.25	0.08	0.26	0.07
As	1.32	0.48	1.54	0.63	1.54	0.61	0	0.55	1.22	0.45
Zr	0.92	0.14	0.33	0.15	1.01	0.17	0.71	0.15	0.39	0.11
In	3.90	1.05	6.21	1.28	5.72	1.29	5.45	1.24	4.04	0.90

content at 13%, followed by sample S1b (6.1%). These areas demonstrated exceptional resistance to natural conditions such as erosion and weathering, indicating a longer material lifespan. Conversely, samples S1a, S2a, and S3 exhibited weaker cohesion and lower C_2S content, making these areas more susceptible to collapse and environmental degradation.

4. Magnesium Oxide (MgO)

Magnesium Oxide (MgO) content is a critical factor influencing mortar durability. Excess MgO can disrupt the internal structure of mortar, leading to reduced strength and efficiency. The internationally accepted MgO limit is 2%. Samples S1a and S1b exceeded this threshold, indicating potential risks to mortar stability in these areas. In contrast, samples A3 and A5 contained no detectable MgO, ensuring the structural integrity of mortar in their respective locations.

5. Resistance to Chemical Erosion

Samples S1a, S1b, S2a, S2b, and S3 exhibited strong resistance to chemical erosion, particularly against acidic rain, due to their favorable composition and low porosity.

DISCUSSION AND INTERPRETATION

The results of the XRD and XRF analyses provide a detailed understanding of the mortar's chemical composition and its implications for structural stability:

1. Structural Stability and Weaknesses

Mortar in areas corresponding to samples S2a and S3 demonstrated high resilience due to optimal calcite content and the absence of MgO. In contrast, areas corresponding to S1a, S1b, and S2b require intervention due to weaker cohesion, higher MgO levels, and susceptibility to environmental stressors.

2. Weathering Resistance

Samples with higher C_2S content, such as A4 and 1C, performed exceptionally well under natural weathering conditions. Mortar in these areas benefits from a prolonged lifespan and reduced vulnerability to erosion and sulfate attack.

3. Impact of Magnesium Oxide

Excess MgO poses a critical challenge, as it can destabilize the mortar's internal structure. The observed MgO content in certain samples highlights areas where restoration is urgently needed to prevent further degradation.

CONCLUSION

The chemical analysis indicates that while the building remains in generally good condition, specific areas exhibit vulnerabilities that necessitate restoration:

1. Stable Areas

Mortar in areas corresponding to samples A3, A5, and C1 demonstrates high stability and resistance to weathering.

2. Vulnerable Areas

Samples A1, A4, and B1 indicate structural weaknesses due to elevated MgO levels and weaker material cohesion.

Recommendations

1. Restoration Priorities

Focus restoration efforts on areas with higher MgO content and weaker mortar cohesion to reinforce structural stability.

2. Preventive Conservation

Implement protective measures in stable areas to preserve the existing mortar and prevent future degradation.

3. Long-Term Monitoring

Establish a periodic monitoring program to assess changes in mortar composition and structural integrity over time.

These results emphasize the importance of targeted restoration and proactive conservation strategies to ensure the longevity of the building for future generations.

Results

The chemical and mineralogical analyses of mortar samples from Deir el-Kahf, using X-ray diffraction (XRD) and X-ray fluorescence (XRF) methods, revealed the following key findings:

1. Dominant Mineral Composition

Calcite ($CaCO_3$) was the primary component in all samples, with concentrations ranging from 53.4% to 95.8%. Variability in calcite content indicated differences in material composition and possible degradation.

2. Silicate Oxides

Quartz (SiO_2) was identified in all samples, with concentrations between 2% and 6.3%. Silica-rich samples demonstrated higher resistance to erosion.

3. Key Chemical Oxides

Calcium Oxide (CaO), Aluminium Oxide (Al_2O_3), Iron Oxide (Fe_2O_3), and Silicon Oxide (SiO_2) were the primary oxides forming critical structural compounds: Tetra Calcium Alumina Ferrite (C_4AF) contributes to mechanical stability; Dicalcium Silicate (C_2S) enhances strength and durability, with higher content observed in areas resistant to weathering.

4. Magnesium Oxide (MgO)

Samples with MgO content exceeding 2% exhibited structural vulnerabilities, requiring restoration.

5. Weathering Resistance

Samples with optimal calcite and C_2S content showed superior resilience to environmental stressors, such as acidic rain and erosion.

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