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Unveiling the craftsmanship and knowledge behind Iranian stuccoes (11th–14th centuries): New insights from an archaeometric perspective

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ABSTRACT

Gypsum-based stucco decorations of 47 monuments in Iran, from the Seljuq to the Ilkhanid period (11th-14th centuries), were studied by multimodal analytical methods, including X-ray diffraction, X-ray fluorescence, scanning electron microscopy and image analysis to evaluate their composition properties. The assessment of results shows that stucco masters in those periods exerted control over the setting process of the gypsum-paste and its microstructure by adjusting water-to-plaster ratio, fine-clay addition, and by means of mechanical processing. Furthermore, the presence of anhydrite in the composition of stucco decorations located in the hot-desert climate of Iran provides evidence for the probability of gypsum-anhydrite transition, which has technical and preservation consequences for this less-investigated type of cultural materials.

1. Introduction

Stucco is a broad term that encompasses a wide range of mixed materials, including lime-based, and gypsum-based compositions, used for architectural decorations. This type of ornament is geographically and chronologically widespread in different cultures, though we focus on the Islamic period stuccoes in Iran. The Persian term *gach-buri* more directly denotes the application of gypsum (*gach* in Persian) as gypsum-based stucco decorations in architectural structures (Mishmastnehi & Stawski, 2025). This craft has been practiced for over two millennia on the Iranian plateau (Kröger, 1982; Mishmastnehi et al., 2023; Wulff, 1966:133–135), where gypsum deposits are abundant and accessible (Mineral commodity summaries, 2024).

It is generally assumed that gypsum-based stucco decorations are associated with various preparation techniques (Aslani, 2006:123–130; Salehi Kakhki and Aslani, 2011:89–106; Wilber, 1949:79–84) and complex craftsmanship (Mishmastnehi & Stawski, 2025). However, there is still a pending need for research to demonstrate such complex interactions between the resulting microstructure of stuccoes and their preparation methods. In this context, extracting information from stuccoes through archaeometric analyses, to shed light on their production technologies, social aspects or their preservation strategies, has been challenging (Mishmastnehi, 2016:1–14; Mishmastnehi & Stawski, 2025; Cotrim et al., 2008:41–49; Hamzavi, 2017:103–107). This challenge is related to the chemistry of gypsum plaster itself, which unlike other crafted materials such as ceramics, metal, or glass (Artioli, 2010; Roberts and Thornton, 2015; Tite, 2008:216–231), reveals limited information about its production process. This limitation arises from the fact that the raw material (gypsum rock) and the final product (gypsum plaster or stucco decoration) are very similar to each other from chemical, crystallographic and mineralogical standpoints. Consequently, clear traceable changes among various stages of the production process cannot be easily observed and analyzed (Mishmastnehi et al., 2023; Mishmastnehi & Stawski, 2025). However, the remarkable existence of numerous stucco masterpieces and the long-standing tradition of craftsmanship for gypsum-based stucco productions in Iran, as well as in other cultures and regions of the ancient world (Domene, 2014; Sapin, 2006; Goodall et al., 2007:666–673; Neumeyer, 1948:104–121;

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Debevoise, 1941), assure that professional techniques and knowledge existed over the ages to engineer this material (gypsum) and to produce desired artistic forms and properties with this medium.

Gypsum-based stucco decorations from the Seljuq and Ilkhanid periods (11th to 14th centuries A.D.) of Iran are considered among the greatest achievements of Persian arts in the Islamic period (Pope, 1967:1258–1364). The number of studies aiming to unfold the technical details of these stuccoes with modern analytical techniques is very limited (Hamzavi, 2021:37–54; Khakbaz Alvandian, 2012:159–175; Mishmastnehi, 2016:1–14). One of the astonishing findings in this context is a type of gypsum paste, the so-called *gach-e koshteh*, which was produced in Iran by controling the crystal-orientation of gypsum by mechanical treatment during the setting process. This method was probably invented around the 14th century in Iran (Mishmastnehi et al., 2023). How many more of such astonishing ancient engineering techniques existed in the past remains unknown. This article addresses this problem with a more systematic approach, applying modern analytical techniques to understand the composition and microstructure of the collected stucco samples. The elemental composition of the samples is studied by X-ray fluorescence (XRF), their mineralogical content by X-ray diffraction (XRD), and their microstructure by scanning electron microscopy (SEM).

1.1. Problem setting and research questions

Here, we aim to address more holistically the core challenge of extracting information from a collected set of samples of gypsum-based stucco decorations from Iran, produced during the Seljuq and Ilkhanid periods (11th to 14th century A.D.). Cultural materials from the past, partly carry their production processes as well as their life-cycle, or even fragments of their historical or archaeological context (Caple, 2006:13–16; Hodder, 2012:2–5). Archaeological science might help reveal some of this information and shed light on past craftsmanship and societies. The preliminary step for such an investigation is a careful *in situ* observation of the macrostructure, followed by composition and microstructural studies.

Key issues examined in the literature so far have focused on whether archaeometric analyses can recognize the similarity between products created by the same master/craftsperson or a workshop under his supervision, particularly from a small sample pool. In addition, they have tested whether it is possible to identify the technological similarities/differences between two stuccoes. Another important question has been the age and chronology of a stucco, but also the source of the raw materials.

Regarding the outlined issues, this study focuses on the following research questions.

1) Is it possible to extract technical information or to find meaningful materialistic similarities (or differences) among various stuccoes from different buildings, locations and periods (Seljuq or Ilkhanid)?
2) Considering the extensive distribution of the samples in various climate zones of Iran, is there any observable deterioration or changes caused by the ageing, with regards to the different local climate zones, which would in turn influence our technical understanding of historical stuccoes?

For this purpose, two monuments (the Great Mosque of Saveh/Loc.13, and Pa-Minar mosque of Zavareh/Loc.27) were crucial and studied in more details (Supplementary Materials_1). This was mainly due to the fact that those monuments contained several different types of stucco decorations, probably from a range of different periods or created by various masters, all within a single monument.

2. Materials and methods

We compiled a list of the most important monuments with preserved stucco decorations from the 11th to 14th centuries in Iran. This list was updated based on the accessibility for the sampling procedure, resulting in a final set of 215 samples from 47 locations, which were studied with a multimodal analytical approach in different laboratories (Supplementary Materials_1, and Supplementary Materials_2). Additionally, two samples of contemporary plaster of Paris, which were produced in traditional plaster kilns in the Isfahan region in central Iran, were also collected and analyzed (see the last two samples in Supplementary Materials_2).

The monuments and their stucco decorations are geographically distributed throughout various parts of Iran (Fig. 1). There are some important monuments such as the Great Mosque of Isfahan/Loc.34, or the Great Mosque of Saveh/Loc.13 that contain stucco decorations from both the Seljuq and Ilkhanid periods, whereas some others such as Gonbad Mosque in Sangan-e Payin/Loc.17 or the tomb of Khajeh Atabak/Loc.46 in Kerman only includes Seljuq stuccoes, and the mausoleum of Seyed Rukn al-Din/Loc.42 in Yazd retains only Ilkhanid stucco decorations. Some significant monuments from these periods, such as the Dome of Soltaniyeh from the Ilkhanid period, for example, were not available for sampling due to administrative restrictions.

The sampling strategy was designed to extract maximum technical information from the stucco while minimizing damage to their form or authenticity as cultural heritage items. Samples were taken from an area of approximately 4–9 mm^2 and had a thickness of 2–5 mm. They were mostly collected from the broken edges of stucco or the areas where there was minimal risk of damage to the aesthetic character of the decorations (i.e. from where it would not be seen). In cases where two distinct layers were identified in a stucco during *in situ* observations prior to sampling, such as the dado of Imamzahed Yahya in Varamin (location 11 in Fig. 1, Supplementary Materials_1), each layer was sampled and labeled individually (StB-125/Loc.11, StB-126/Loc.11). This approach aimed to detect any intentional changes in the composition or microstructure among the layers, which could indicate technical variations or the use of different materials in producing stuccoes. Sampling was conducted carefully using a sharp scalpel, with adjustments made based on the individual conditions and properties of each sample. For instance, if a fragment that met the criteria of our sampling strategy was already broken and posed no aesthetic issues for the stucco, that fragment (or a part of it) would be preferentially sampled.

A large number of samples, with different dimensions were analyzed in several laboratories. Some samples lack XRD or XRF analyses, and some others are missing SEM micrographs (Supplementary Materials_2). This is mainly caused by sample perperation procedures. For instance, coating a sample for SEM renders it, and makes it useless for XRD or XRF. Conversely, grinding a sample for XRD or XRF analyses, destroyes the microstructure that needed to be studied by SEM. Furthermore, some samples were analyzed with different SEM instruments, while others were too small to be powdered and analyzed by any of the aforementioned methods (Supplementary Materials_2); the later were accordingly taken out of the study, but still are included in the supplementary materials (because they were sampled and numbered during the field work). Despite the mixture of approaches in the analyses of the samples, this study represents the most extensive research on a large number of stuccoes from this time period in Iran and stands as a pioneering effort in the archaeometry of gypsum-based stucco decorations.

2.1. Compositional analysis (p-XRF)

The elemental composition of the 100 samples was identified using X-ray fluorescence (XRF) method with a portable spectrometer (TRACER 5g) by Bruker (Supplementary Materials_2). Analyses were conducted on a small amount of powdered sample, collected within a small polypropylene ring-shaped holder with 2 mm thickness and 5 mm in diameter to ensure reproducible sample preparation. Spectra of samples were collected at three different conditions, i.e., 15 kV without filter, 30 kV using a Ti:Al filter, and 50 kV using a Cu:Ti:Al for optimal

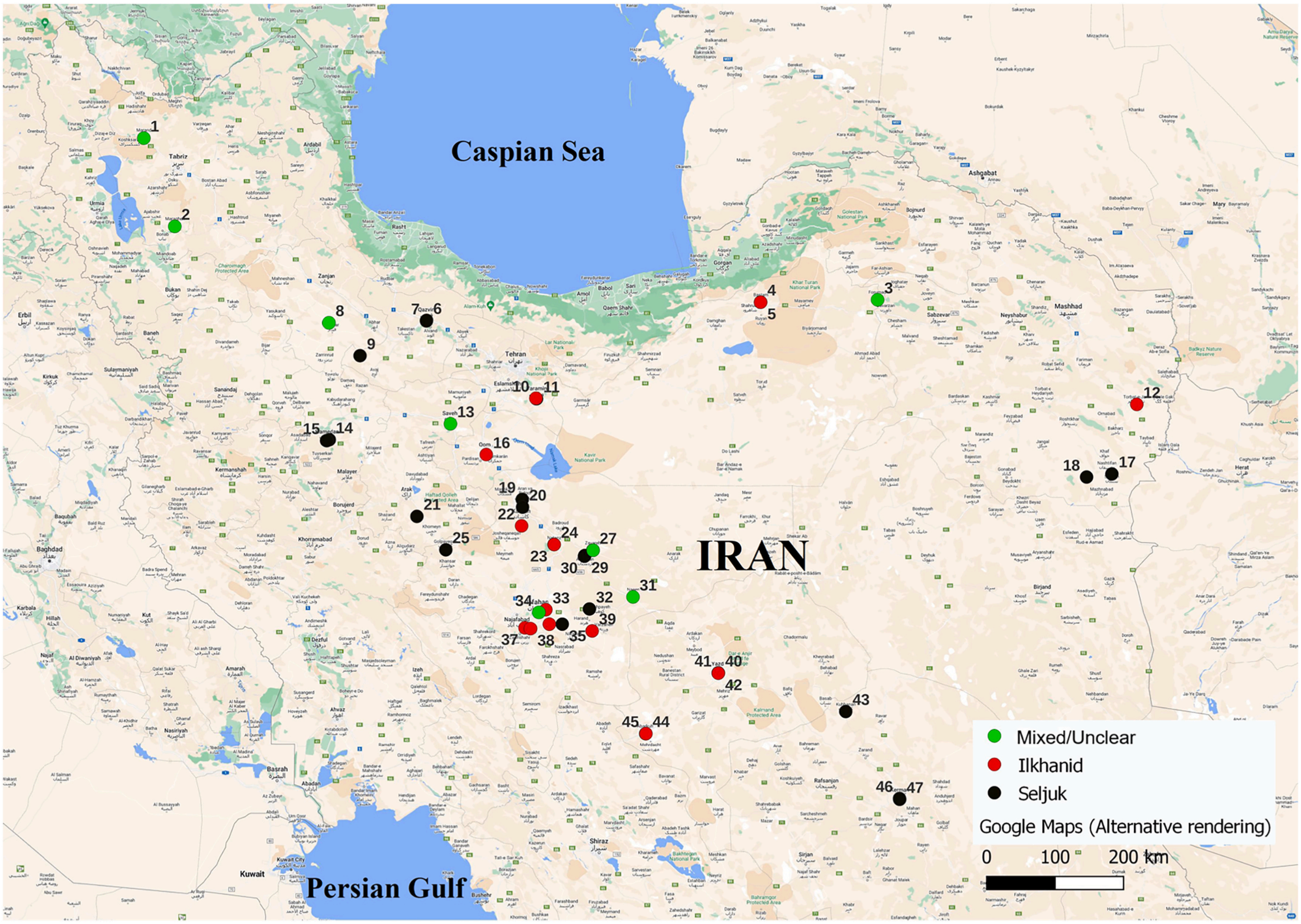

Fig. 1. The location of the sampling sites on the map of Iran. Black circles show monuments with only the Seljuq stuccoes, while red circles represent buildings with Ilkhanid stuccoes. Monuments with green circles either possess stuccoes from both periods or stuccoes of unclear date.

excitation of the low, medium, and high energy elements, respectively. The analytical results were assessed using Artax 7 software. In addition to the main objective of gaining information about the major components of the samples, traces of strontium were also investigated to determine if they could possibly provide insights into the resources or the type of gypsum paste used for different stuccoes (see below, 4.1), thus highlighting technological changes.

2.2. Phase analysis (XRD)

The crystallographic texture and mineralogical phases of 150 samples were characterized using Bragg-Brentano X-ray diffraction (XRD) on powdered samples employing a D8 Bruker diffractometer equipped with an LYNXEYE XE-T detector. Diffraction was measured with Cu Kα radiation (1.5406 Å, 40 kV, and 40 mA) from 5 to 100° using a step size of 0.01° (2θ) and a scanning time of 0.5 s per step. The phase composition was quantified using the Rietveld method with "X-pert HighScore Plus'' software, version 3.0. The main focus of this part of the study was to determine the content of major mineral phases, particularly the ratio between various calcium-sulfate phases (β-anhydrite, $CaSO_4$; bassanite, $CaSO_4 \bullet 0.5H_2O$; gypsum, $CaSO_4 \bullet 2H_2O$), and other mixed minerals such as silicate and/or aluminosilicates, and carbonate on the other hand.

2.3. Microstructural analysis (SEM)

The microstructural analysis encompasses two aspects: a) the form and the habit of gypsum crystals; and b) the microstructure of the matrix formed by the accumulation of newly grown gypsum crystals; or the presence of other minerals or materials. For this purpose, electron micrographs of 90 samples were acquired using several instruments: the Carl Zeiss SMT Ultra 55 Plus and Supra 40 field emission scanning electron microscopes, as well as a flexible variable pressure Carl Zeiss EVO MA 15 scanning electron microscope. The samples intended for higher resolution pictures (for detailed examination of their crystallographic and microstructure features using the Supra 40 machine) were sputtered with a 10 nm gold layer, while those collected for image analysis of their matrix and porosity were examined at lower resolution and left uncoated. This latter group was studied at 20 kV and an 8.5 mm working distance under variable pressure (VP) conditions using the Carl Zeiss EVO MA 15 device.

For the image analysis, three images were captured at lower magnification (250x) to identify the best location for studying the matrix of each sample. From these, a set of final six images at higher magnification (2000x) was acquired. These images were utilized for the analysis of the matrix using JMicroVision software, version 1–27, to estimate the void space among newly grown gypsum crystals. It is important to note that this approach, which involves analyzing freshly broken stucco fragments for their macropores, is a comparative technique and should be employed within the same conditions and device across the entire set of samples for this purpose, to ensure the reproducibility and reliability of the results (here just Carl Zeiss EVO MA 15 device). At the high magnification of 2000x, the issue of dimensionality is partially resolved for the question at hand, which pertains to the amount of voids among gypsum crystals during their growth. This space is correlated with the water-to-plaster ratio (W/P). When there is more water, the plaster particles are spread farther apart, resulting in a less dense and more open structure. Once the water evaporates, the remaining structure has larger gaps between the crystallized gypsum particles, contributing to the porosity. Furthermore, studying the microstructure, such as how crystals are oriented in relation to each other or their shapes and forms, provides insights into their growth conditions, which is associated with production composition and techniques (Mishmastnehi et al., 2023) or aging processes, which must be observed and interpreted with great care.

3. Results

3.1. p-XRF

According to the XRF results, there is a wide range of silicate and aluminosilicate materials observed in different samples from various locations (Supplementary Materials_2). This is clearly depicted on the biplot of Al_2O_3 versus SiO_2 graph (Fig. 2). However, it is important to note that the higher level of LOI and the inability of p-XRF to measure sodium present challenges here. Another important result is the strontium analysis, projected as a biplot of Sr versus Ca, which shows a large variety from 0 to 1.5% (Fig. 3). A more detailed discussion concerning these results and their interpretations is provided in the discussion section, where the composition of samples from different locations/monuments is compared.

3.2. XRD

The XRD analysis of the samples, with phase quantification using a Rietveld method, indicates that, considering the entire composition, most of the samples are dominated by calcium sulfate phases (Supplementary Materials_2, Fig. 4). However, samples with higher levels of silicate and aluminosilicate or calcium carbonate are also observable within the XRD results. Moreover, considering only the $CaSO_4.xH_2O$ phases in the composition, various amounts of gypsum and anhydrite were detected (Supplementary Materials_2, Fig. 5), the fact of which is further explored in the discussion section.

3.3. Microstructure

Gypsum is an anisotropic crystal, with different solubility of its various faces. It has been commonly asserted that despite the large variety of forms and patterns in the microstructure of gypsum-based plasters, mortars and stuccoes, acquiring useful information is not always feasible or straightforward (Gariani et al., 2018:23–32). The difference between the forms and habits of gypsum crystals in historical and recent plasters and mortars has been frequently reported (Kingery et al., 1988:219–243; Middendorf, 2002:165–176). The most important aspect of gypsum is, whether there are well-formed euhedral or acicular crystals of gypsum, which indicates a suitable environment for the growth of newly formed crystals. This is usually observed in most of the samples, but best in the samples with higher W/P ratios, where sufficient water supply promoted an extensive growth for most crystals (Table_1 first column, and Supplementary Materials_1). The presence of other materials such as charcoal or sand (unknown grains when not analyzed by EDX) is another phenomenon, which can be observed in SEM micrographs (Table_1 second and third columns, and Supplementary Materials_1). Finally, another important observation is the existence of pseudomorphic crystals, where the original monoclinic prismatic morphological forms of gypsum are preserved, but the actual crystal phases have converted into anhydrites, and internally consist of smaller crystals (Table_1 fourth column, and Supplementary Materials_1).

For studying the microstructure of the stuccoes as an accumulation of gypsum crystals, the observation includes assessing the amount of empty space among crystals (Image Analysis, see section 3.4), or observing the orientation of a group of crystals together creating a particular feature, such as the *koshteh* texture (Table_1 fifth column, and Supplementary Materials_1) (Mishmastnehi et al., 2023), or arranging and growing up around trapped air bubbles during the setting process (Table_1 sixth column and Supplementary Materials_1).

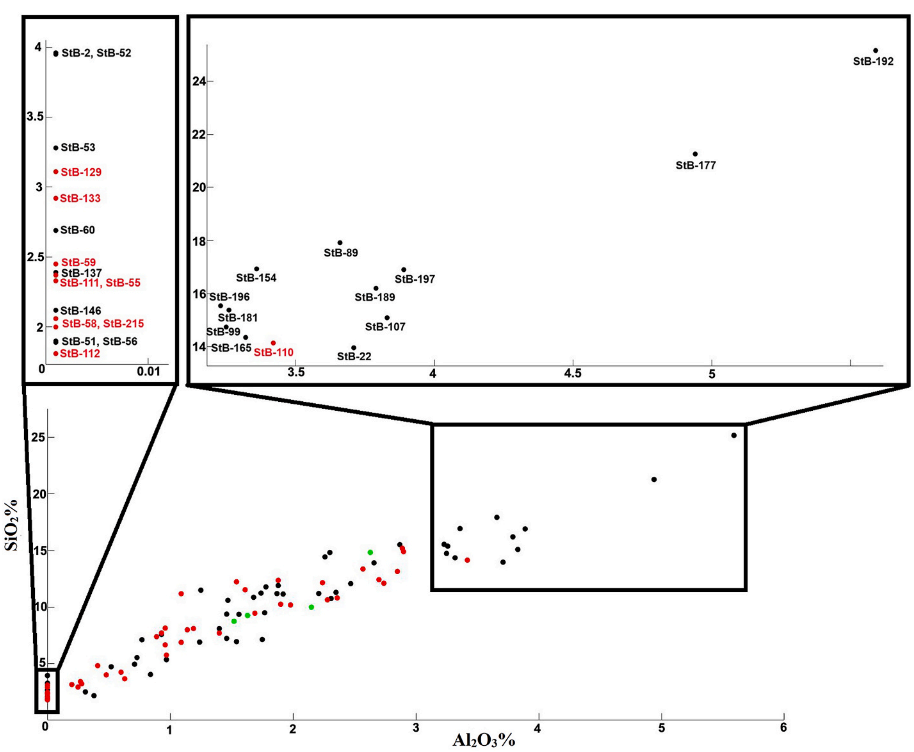

Fig. 2. Biplot of SiO_2% versus Al_2O_3% in the composition of stucco samples. The black dots represent samples from the Seljuq period, while the red dots show the Ilkhanid samples. The green dots indicate samples with unclear date.

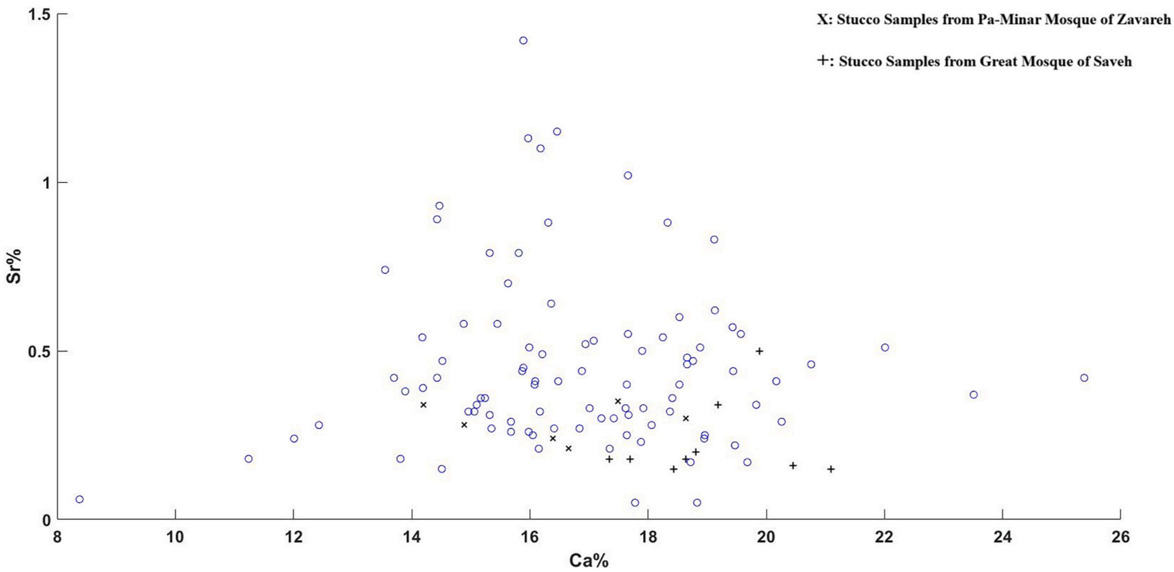

Fig. 3. Biplot of Sr% versus Ca% in the composition of stucco samples.

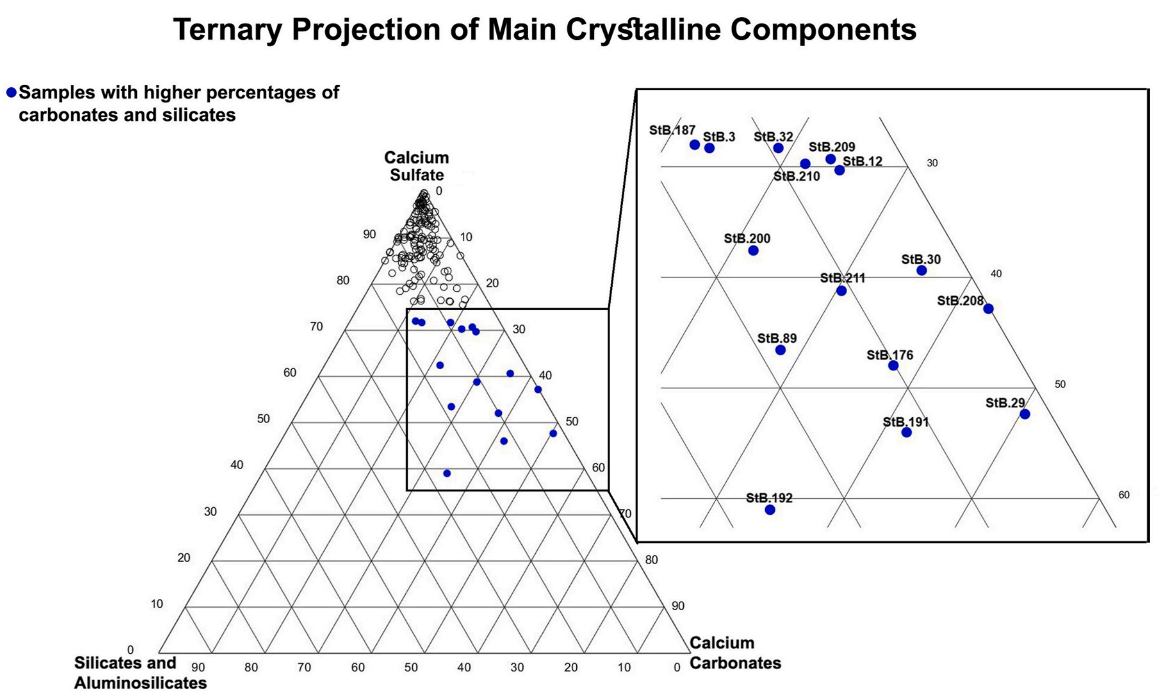

Fig. 4. Ternary projection of the main crystalline phases in the compositions of stucco samples, projected from the XRD results.

3.4. Image analysis

By analysing electron micrographs at 2000x magnification and focusing on regions that mostly consist of newly grown gypsum crystals, we can estimate the amount of voids in the matrix, which in turn is correlated with the original W/P ratio (more empty space implies higher porosity and a larger W/P).

Due to the large sample size and the aforementioned challenges of this method, we restricted this aspect of our study to comparing the results of image analysis for different layers of multi-layer stuccoes, rather than comparing all the samples together. Through the sampling process and *in situ* observation, we obtained 18 stuccoes from both periods with 1–2 mm thick finishing layers, and their under-layer. The results of image analysis are useless if all the inner layers (or outer layers) of different samples are compared with each other; but they become entirely meaningful when two layers of the same stucco sample are compared. The projection of results as a bar chart indicates that for most of the samples, the outer layer has a higher percentage of empty space than its inner layer (Fig. 6, and Supplementary Materials_1). This outer layer typically serves as a very fine final coat before pigmentation (Fig. 8d; if applicable, see Holakooei and Karimy, 2024:600–617). These results reveal technical insights into the Seljuq and Ilkhanid stucco masters and their technical decision-making in the plaster.

4. Discussion

The material body of a historical object can be analyzed using various analytical methods provided by material science, to extract raw

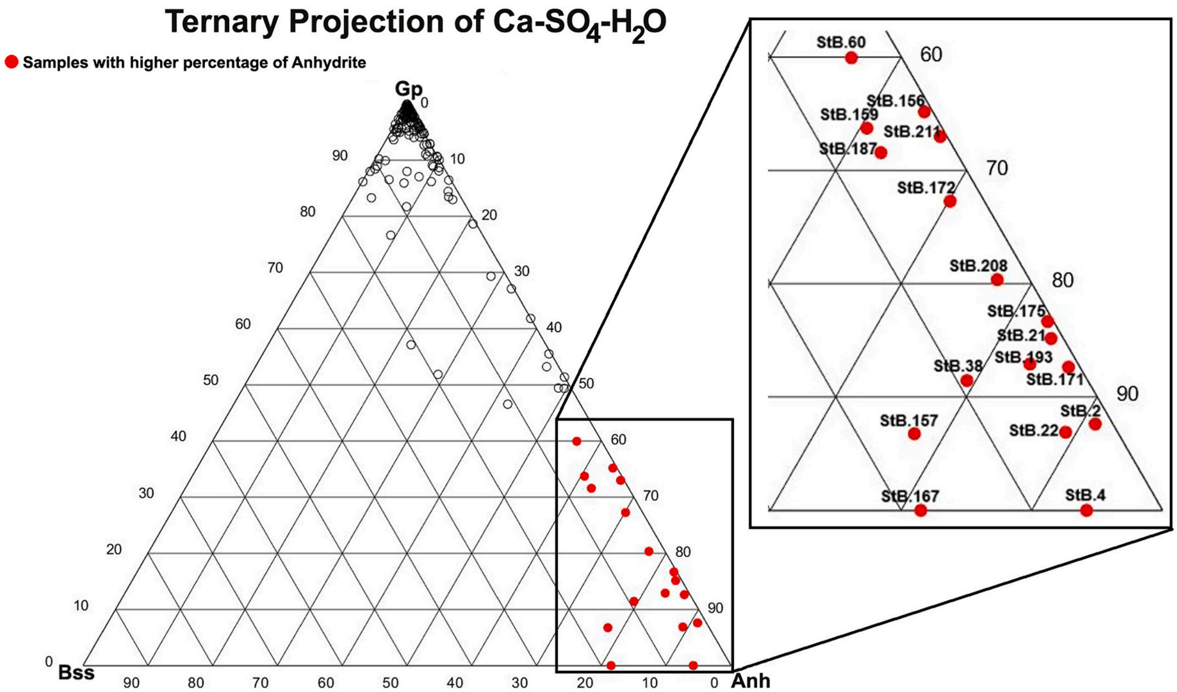

Fig. 5. Ternary projection of the main Ca-SO_4-H_2O phases in the compositions of stucco samples, projected from the XRD results.

Table 1
Microstructure of different samples.

Low density matrix with well-formed euhedral or acicular crystals of gypsum	Remains of Charcoal	Large grains of other minerals	Pseudo-morphic anhydrite in form of gypsum	Koshteh or partly Koshteh	Trapped bubbles
StB_104; StB_125; StB_143; StB_147; StB_207; StB_215	StB_36; StB_111; StB_145	StB_35; StB_36; StB_95; StB_109; StB_110; StB_160; StB_186; StB_192; StB_196; StB_197; StB_202; StB_204	StB_12; StB_39; StB_157	StB_27; StB_87; StB_102	StB_19; StB_54; StB_55; StB_57, StB_77; StB_110; StB_187

parameter values and numbers. However, drawing a network of relationships among existing materials and historical events in the past (from *chaîne opératoire* to social context or historical event, etc.) requires careful observations, sampling, analyses, and interpretation.

The analytical part of this study aimed to shed light on three aspects of stucco decorations from the Seljuq and Ilkhanid periods of Iran.

A) Resources of raw materials
B) Technical characteristics
C) Deterioration of stuccoes in relation to their age and climate that will be discussed in the following parts.

4.1. Resource

It is suggested that the ratio between calcium and strontium (Ca/Sr) is a helpful parameter for determining the origin of gypsum-based cultural materials (Franceschi and Locardi, 2014:522–527; Gariani et al., 2018:23–32). The reason for this is that natural gypsum contains a significant amount of strontium in its crystal lattice as a substitution for calcium (Denison et al., 1998:1–18), therefore the Ca/Sr ratio hypothetically acts as a gypsum-fingerprint correlating a raw material with its original geological location. Two monuments (Great Mosque of Saveh, and Pa-Minar mosque of Zavareh, see Supplementary Materials_1 and Supplementary Materials_2) with several stuccoes, probably from different periods or makers, provided a useful set of samples within a larger group of specimens to evaluate the utility of this trace element analysis (Fig. 3).

A relevant geological map of Iran is required to investigate the probable sources of raw materials in this study. The study on the hydrology of gypsum formation in Iran (Raeisi et al., 2013:68–80), provides such a primary distribution map of gypsum occurrence in Iran. The location of main gypsum formations in Iran and active/inactive gypsum mines in Iran, mentioned by Raeisi and his colleauges (Raeisi et al., 2013:68–80), are well-matched with the sedimentation layers of Jurassic, Tertiary, Neogene and Quaternary periods on the geological map of Iran (Pollasro et al., 1997). Projecting the collected historical samples on this map indicates that most of the stuccoes in this study are in proximity of 20–50 km distance to natural gypsum resources (ranging from small-scale to large-scale, Fig. 7, and Supplementary Materials_3). This positioning suggests that the Ca/Sr ratio for stuccoes within a single monument should be grouped together. However, highlighting the two aforementioned monuments in terms of Ca/Sr demonstrates a wide range of scattering of these samples ("+" and "x" symbols in Fig. 3). It is also important to compare the calcium and sulfate content of the same group (due to other calcium-containing minerals such as calcite in the soil, which can affect the analysis) and investigate the impact of impurities/additives in the analyzed samples, which can influence the overall analytical results and interpretation (Supplementary Materials_2, and Supplementary Materials_4). Additionally, as shown in Fig. 2, samples exhibit a varying soil content indicated by a positive correlation of SiO_2 vs. Al_2O_3. Soils also contains small amounts of Ca, which could alter or dilute the Ca/Sr ratio to a point where it cannot be used any longer for the determination of the gypsum source. However,

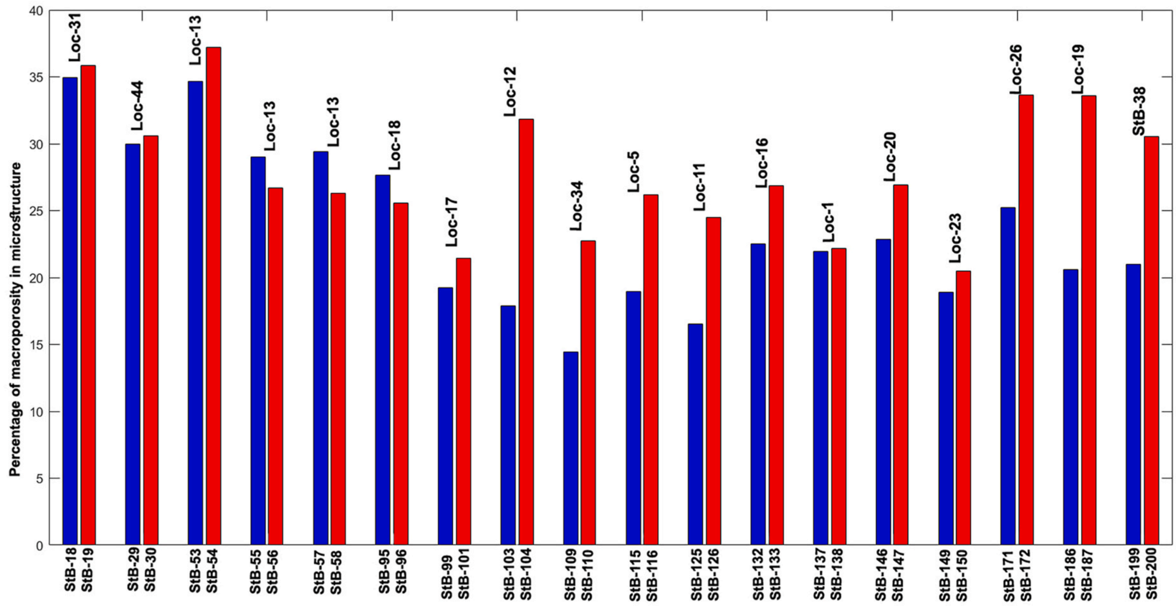

Fig. 6. Bar chart comparing the percentage of macropores between gypsum crystals in the microstructure of stucco, based on image analysis of two-layered stuccoes. The blue colour represents the inner layer, while the red color shows the outer layer of stucco.

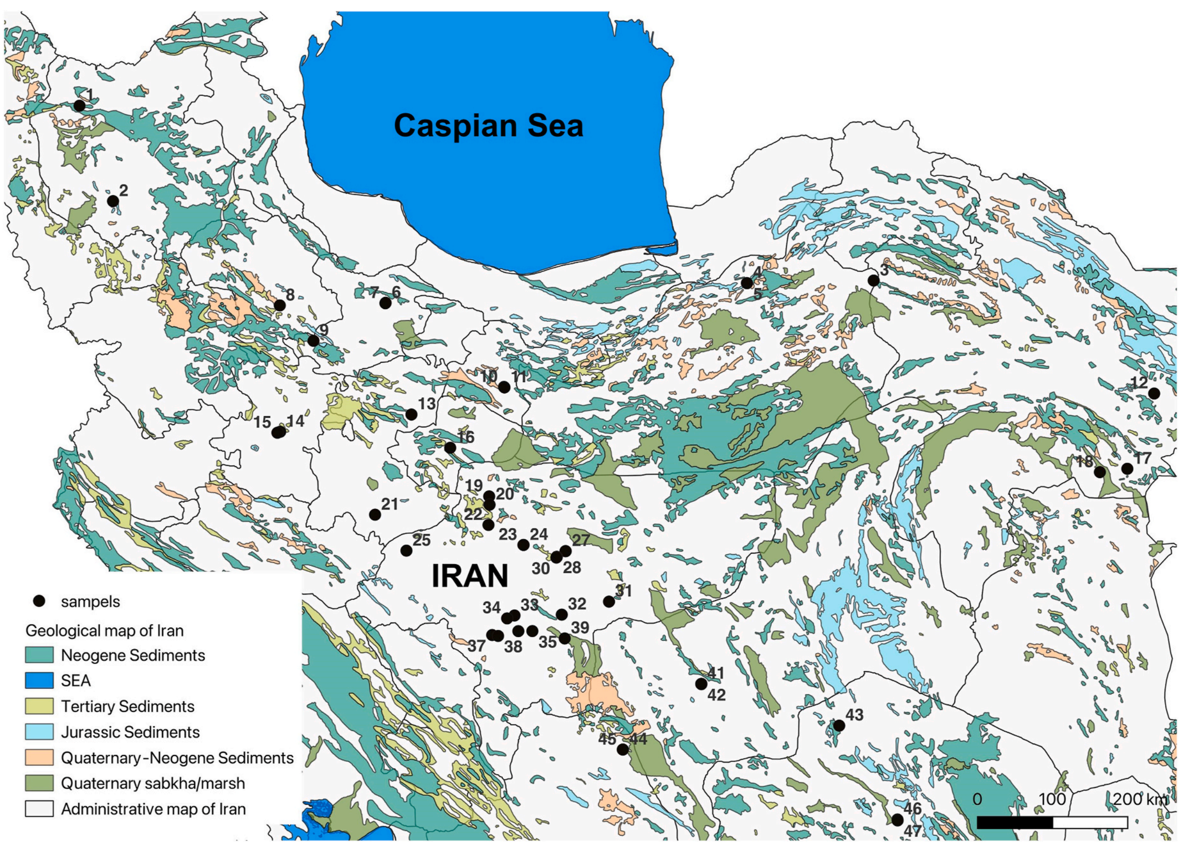

Fig. 7. The projection of collected samples on the relevant sedimentation layers of the geological map of Iran (© USGS), illustrating the relationship between locations of monuments with stuccoes and probable gypsum deposits.

the samples from the Great Mosque of Saveh display an exceptionally low content of detrital material indicated by their low concentrations of SiO_2 and Al_2O_3, representing a high gypsum-purity (see Supplementary Materials_2, and Supplementary Materials_4). Consequently, for very pure samples such as those from the Great Mosque of Saveh, the Ca/Sr ratio can be used to indicate the use of different gypsum raw materials from various neighboring locations. However, for more impure samples, i.e., samples with a higher silica and aluminum oxide content, such as those from the Pa-Minar mosque of Zavareh, the Ca/Sr ratio is not very helpful (Fig. 3). Although more sophisticated preparation and analytical

Fig. 8. Six samples of stucco decoration subjected to the multi-analytical study, a) the small mihrab in the Greate Mosque of Ardestan/Loc.28, b) the stucco inscription with applique plaster technique (*fetilehiy*) in the mihrab of Jame mosque of Oshtorjan/Loc.37, c) the narrow space between stucco-decorated graves in Baba Abdullah mosque in Nayin/Loc.31, d) two distinct layers of gypsum plaster on the inscription of miharb in Koochemir Mosque of Natanz/Loc.23, e) molded/cast stuccoes fragments found in the storage of Malek Mosque, Zuzan/Loc.18, f) deteriorated stucco decoration on the exterior wall of tomb of Khajeh Atabak in Kerman/Loc.46.

methods, such as first purifying gypsum from impurities before Ca/Sr analysis, and more accurate analytical techniques (e.g. inductively coupled plasma mass spectrometry) could theoretically produce more meaningful results, more sample material would be needed. The cultural value of historical materials and their sampling limitation should be taken into consideration.

4.2. Technology

This is the most challenging aspect of studying stucco decorations from Iran and beyond using analytical methods (see also Bel-Anzué and Elert, 2021; Domene, 2014; Sapin, 2006). The topic remains underexplored, with very limited contextual literature and research background available. While various archaeologists (Debevoise, 1941; Kröger, 1982) and art historians and conservation scientists (Aslani, 2014; Pope, 1967:1258–1364; Salehi Kakhki and Aslani, 2011:89–106; Wilber, 1949:79–84; Grbanovic, 2023b) have examined this subject, speculating on possible techniques for design execution, layering order, or the use of molds in stucco formation, the research remains scarce, particularly from an archaeometric perspective. Another group of researchers, based on similarities in patterns or signature markings, has suggested the existence of specialized craftsmanship or workshops under the supervision of a master craftsman ("the grand-master"), with these workshops potentially moving across different regions of Iran (or even among different countries, see for example Bakhoum, 2016:17–32), over various periods. These researchers proposed that such movement of craftspeople could be linked to specific carving techniques, observable as a technical signature of the master and their workshop (Blair, 2003:39–47; Grbanovic, 2023a,b; Salehi Kakhki et al., 2016:19–31; Salehi Kakhki and Taghavinejad, 2017:55–74). However, the most significant challenge for this hypothesis is the absence of traceable techniques, or systematic studies on such a topic, that could be recognized beyond the inscription signature in the material body of stuccoes. A few studies, primarily by conservation scientists and conservators, have categorized stuccoes based on more general techniques, such as layering thicknesses and combinations with mirrors, colored glasses, or mud plaster (Aslani, 2014; Salehi Kakhki and Aslani, 2011:89–106; Grbanovic, 2023b). While this last group of researchers represents a progress in the research field, they are still far from discussing production techniques of specific stuccoes or distinguishing meaningfully among various stuccoes, particularly when they fall into the same category as defined by Aslani, Kröger, or Wilber (Aslani, 2014; Kröger, 1982; Salehi Kakhki and Aslani, 2011:89–106; Wilber, 1949:79–84).

There are material characteristics that can preserve aspects of past production technologies, one of which is the setting conditions for gypsum plaster. Quick setting gypsum plaster has been a challenge for stucco masters. Various techniques and innovations have been developed across different cultures and regions to control the setting process of gypsum plaster and its associated mechanical behavior. One of the most important factors, as noted by stucco masters in Iran (and other regions of the world), is the water-to-plaster ratio, which is also well-known and utilized within the mortar research community. The astonishing technique of creating *koshteh* paste is another approach to addressing the quick setting problem of gypsum for specific applications, where its crystallographic features were discovered with the aid of modern analytical instruments (Mishmastnehi et al., 2023). Another technique mentioned by traditional craftspeople (in Spain), who operated gypsum calcination kilns, is the control over the slow cooling process of the gypsum kiln (Bel-Anzué and Elert, 2021), which requires a proper investigation. On one hand, the application of various organic or inorganic additives to the plaster (Domene, 2014; Weijnen et al., 1987a, b), and on the other hand, the control over the water-to-plaster ratio (W/P), are the most common approaches for this purpose, which can also be identified analytically from the composition.

The composition analyses of the samples reveal two primary groups: the first one with a high percentage of calcium sulfate-based materials, and the other containing impurities such as silicates, aluminosilicates, and calcium carbonates. The ternary projection of the major composition indicates two main clusters of analyzed samples: empty circles, consisting primarily of calcium sulfate-based materials and low levels of silicate and calcium carbonate; and blue dots, with a high percentage of silicates or aluminosilicates, and calcium carbonate, highlighting the intentional addition of these materials into the gypsum composition (see Fig. 4). Two samples with a higher percentage of calcium carbonate and a lower level of silicate (StB-208/Loc.45 from Pir-e Hamzeh Sabzpush and StB-29/Loc.44 from the Great Mosque of Abarkuh, see Fig. 4, Supplementary Materials_1 and Supplementary Materials_2) suggest the intentional mixture of gypsum and lime to create these stucco decorations. Notably, both samples were collected from the same city, Abarkuh (locations 44 and 45), which might indicate a local tradition in this region. It is worth highlighting that these samples belong to two different periods, Pir-e Hamzeh Sabzpush from the Seljuq period and the Great Mosque of Abarkuh from the Ilkhanid period, suggesting that this local tradition (mixing gypsum with lime for stucco) has survived the Mongol invasion of Iran (1219–1258 A.D.). The samples represented by blue dots in the ternary projection (Fig. 4) show an average level of both major inorganic impurities in plaster—silicates or aluminosilicates and calcium carbonates—where the source for both impurities could be some fine clay or soil (sand and clay mixture, see Supplementary Materials_2) present in the sample.

Distinctive features can also be observed when considering the aluminosilicate content of the samples. Some stuccoes with a higher percentage of aluminosilicates, such as StB-107/Loc.21, and StB-197/Loc.30, have already appeared puzzling from a visual or technical standpoint and could be mistaken for clay-carved decorations, as the first authors initially presumed for the stuccoes in Imamzadeh Abdullah in Kudzar (Aghajani et al., 2023:819–836; Mishmastnehi & Stawski, 2025). In contrast, the present study shows that many samples have higher amounts of alumina relative to sulfur, where their end members (12 from 13 samples, such as the Great Mosque of Ardestan, see Fig. 8a) belong to stuccoes either dated to the Seljuq period or presumed to have been created before the Ilkhanid period (Fig. 2 and Supplementary Materials_1 and Supplementary Materials_2). The opposite scenario is less clear; not all stuccoes with pure gypsum compositions belong to the Ilkhanid period, such as stucco mihrabs in the Great Mosque of Saveh (Fig. 2, Supplementary Materials_2). These results highlight the possibility of the intentional addition of clay-based material to the stucco composition during the Seljuq period to retard its setting process while providing a fine texture for carving. However, it should be mentioned that this statement is based on the current state of the art and the samples analyzed here.

An important aspect of production technology for stucco decoration involves the transfer and execution of designs onto the plaster surface (Aslani, 2014). Recognizing this process is not directly achievable through compositional or microstructural analysis. It requires careful *in situ* observation, and to some extent luck in uncovering outer layer damage that reveals design lines or transferred patterns on the inner layer, or designs made through pigmentation (mostly red, but also black and blue, see (Holakooei and Karimy, 2024:600–617). These design lines, particularly evident in more complex and three-dimensional stuccoes with high relief, have been the primary basis for scholars to categorize stuccoes into high-relief, which are also multi-layer, and low-relief or single-layer groups (Aslani, 2014; Kröger, 1982; Pope, 1967:1258–1364; Salehi Kakhki and Aslani, 2011:89–106; Wilber, 1949:79–84). A recent study provides compelling evidence that intentional technical modifications may have been applied to the finishing layer in the past, particularly emphasizing the impact of troweling on the microstructure of gypsum crystals (Hamzavi et al., 2023: 87–113). However, while many stuccoes in this study could be classified as low-relief (single layer according to previous definitions), our careful observations revealed at least two layers in most of the low-relief samples (Fig. 8d). Signs of shallow carved patterns in the inner layer indicate that a stucco master applied the design on the inner layer and then covered it with a very thin layer (1–2 mm) of plaster as the final coat. This final layer was adjusted to the inner carved layer and, in some cases, showed no sign of pattern transfer; in others, the pattern was applied on the outermost layer and then carved. This raises the question: is this final layer different from the inner layer in a manner discernible by analytical methods, implying intentional modification and technical decision-making? Image analysis results indicate that in most samples, the outer layer exhibits a higher percentage of empty space (black space in SEM images), indicative of a higher water-to-plaster (W/P) ratio (Fig. 6). This suggests that some masters intentionally applied a higher W/P ratio to this outermost layer to retard quick setting of the plaster, while increasing workability, and achieving a perfect finishing layer appearance.

The preparation of gypsum paste, achieved by mixing plaster of Paris with water, may seem like a simple process, yet it is crucial for stucco masters and traditional builders. Whether for basic flat plastering on a wall, or a complex task of preparing mortar for different vertical zones of bricklaying in the shell of a dome—where each elevation may require a different style of gypsum mortar according to traditional masters —or for various layers of a stucco masterpiece, this step is the key. Distinguishing between the various pastes used for different purposes with modern analytical instruments is challenging, due to the nature of gypsum paste on one hand and the lack of sufficient research on the topic on the other. It is, however, noteworthy to mention a method applied in a recent study for measuring the porosity in the microstructure of gypsum-based cultural materials by micro X-ray tomography (Beaugnon et al., 2019:17–24), which was not accessible during this study. One frequently mentioned factor by traditional stucco masters in Iran is avoiding agitation of the plaster before it reaches its proper viscous state, which microscopically equates to allowing the primary growth of gypsum crystal seeds beyond the critical point. This situation is associated with the presence of trapped air bubbles in the paste, which should be removed to achieve a very fine texture on the outer layers of stuccoes for carving purposes. The presence of these bubbles (Fig. 9e) or their deformed versions (Fig. 9f) in a paste is not a significant issue for the inner layers, as seen in StB-187/Loc.19 (Supplementary Materials_1), but shows a low level of troweling and pressure applied during paste

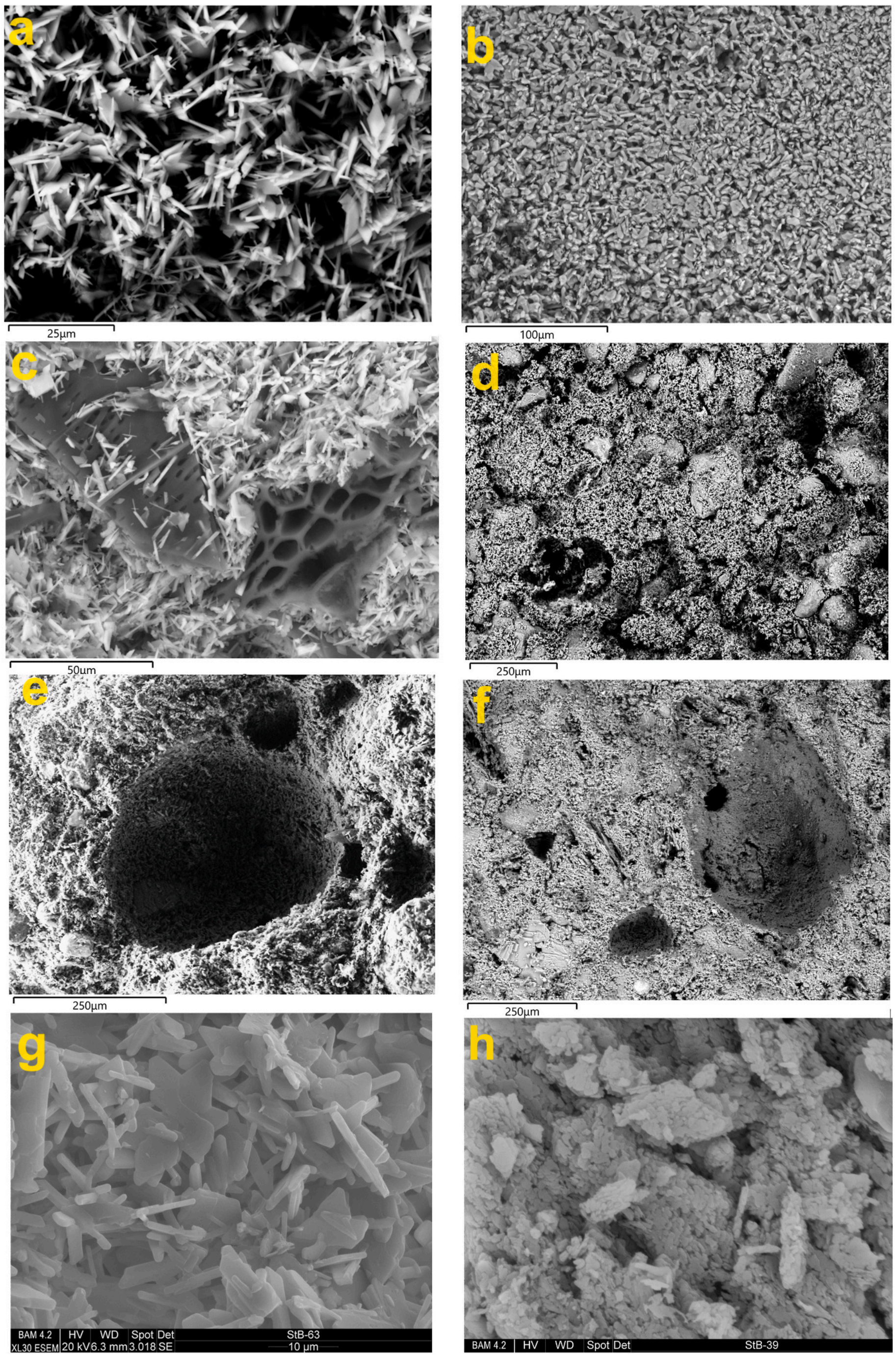

Fig. 9. Eight SEM images representing various technical conditions of analyzed samples, a) highly porous microstructure of sample StB-104/Loc.12, representing high W/P ratio, b) well-oriented microstructure of Koshteh paste from under-painting plaster in Mazar-e Jam complex StB-102/Loc.12, c) the presence of charcoal in the microstructure of StB-111/Loc.4, d) large grains of unknown minerals (likely sand) in the microstructure of StB-35/Loc.47, e) well-formed air-bubble in the microstructure of StB-98/Loc.18, f) deformed but preserved air bubbles in the microstructure of StB-183/Loc.22, representative of low-level mechanical processing on the plaster, g) well-formed euhedral gypsum crystals in the StB-63/Loc.15, f) deteriorated gypsum paste of sample StB-39/Loc.46, where anhydrite replaced the gypsum phase but preserved its crystalline form.

flattening (Mishmastnehi & Stawski, 2025). Removing these bubbles is impossible for molded or cast stuccoes such as StB-98/Loc.18 (Fig. 8e), found in the storage of Malik Mosque, Zuzan (Supplementary Materials_1). Their presence can sometimes resolve puzzling technical questions, such as the production techniques of stuccoes on the graves in Baba Abdullah Mosque, Nayin (Fig. 8c, Supplementary Materials_1), where it seems unlikely that the stucco master could have transferred and executed the patterns *in situ* and directly on the side of the grave, due to very narrow space between two graves. This suggests that the stucco was created on a panel or rectangular cast stucco on the ground and then transferred and installed on the grave after being carved (Mishmastnehi & Stawski, 2025). This technique might be the case for some of the aforementioned stuccoes on the graves in Baba Abdullah Mosque/Loc.31, but probably not for all of them. The presence or absence of air bubbles is valuable evidence for the level of mechanical work applied to the paste. This is especially true when the microstructure shows that newly grown gypsum crystals formed in a spherical orientation during the setting process around the trapped air bubbles, rather than as a result of aging damage. It should be noted that the presence of bubbles is not always associated with molding/casting techniques and can also be observed in other cases, where low level of flattening was applied.

4.3. Climate and preservation conditions

The presence of anhydrite and bassanite in gypsum-based cultural materials has long been, and continues to be, puzzling (Bel-Anzué and Elert, 2021; Charola et al., 2007:339–352; Kamel et al., 2014:313–327; Kamel, 2019). Typically, starting with a composition predominantly made of bassanite and given sufficient water for the reaction, the expected final product is gypsum, which should not readily revert to anhydrite or bassanite under the normal atmospheric conditions of historical buildings. A higher percentage of anhydrite in a composition weakens the plater/mortar, making it prone to damage, an issue frequently noted by conservators, conservation scientists, and archaeological scientists. These researchers often correlate anhydrite with past fire events or with impurities introduced during the calcination process (Ali et al., 2021:789–806; Kamel, 2019). Much of their work focuses on treating the damaging impact of anhydrite and attempting to convert it into gypsum (Kamel, 2019), among other approaches. Since anhydrite compromises the stucco mechanically (a fact we understand, though it is unclear if ancient masters were aware of this), its absence can be seen as indicative of the level of mastery in stucco production and the material choices made by past craftsmen. This underscores the importance of clearly understanding the presence of anhydrite and its sources (primary vs. secondary) in historical stuccoes.

Analysis of various products of traditional kilns (samples StB-216/ and StB-217, see Supplementary Materials_2) reveals a lack of homogeneous temperature and well-controlled environments, where a high percentage of anhydrite can already be present after firing. Accordingly, several scientists hypothesize that the majority of anhydrites in historical stuccoes are the result of impurities from the kiln and insufficient control over the firing process (Artioli, 2010:250–251; Bel-Anzué and Elert, 2021; Kamel, 2019; Pombo Cardoso and Pye, 2018:72–96). This heterogeneous calcination and firing atmosphere can produce anhydrite on the exterior surface of gypsum rocks, while also potentially leaving the center of the block as unreacted natural gypsum, failing to convert it fully into reactive bassanite. To address this, un-calcined gypsum or anhydrite grains may need to be removed through a sieving process or by decanting the slurry paste before the setting process finish, leaving the unreacted grains at the bottom of the container; a technique well-known among Iranian traditional masters to remove undesired grains from plaster (Mishmastnehi & Stawski, in Press). However, some of these grains also sometimes may be found in historical materials if plaster was not carefully prepared, or if a very fine paste was not intended to be applied.

The transformation between various phases of Ca SO_4-H_2O has been the subject of long-term studies (Jacques et al., 2009:421–432; Krejsová et al., 2024; Ossorio et al., 2014:16–21; Tang et al., 2019:7636–7642; Wehmann et al., 2023), most of which focus on these transitions in a hydrous environment. However, more recent evidence of this transformation in natural environments and laboratory works has been published (Krejsová et al., 2024). A recent study by Hamzavi and colleagues examines gypsum mortar and plasters from five historical monuments dating from the Sassanian to the Ilkhanid periods (3rd–14th centuries A.D.), across four distinct climatic regions in Iran, using multi-analytical methods to highlight the relationship between production technology, plaster/mortar preservation, and the climate (Hamzavi et al., 2023: 87–113). In contrast, the present study analyzes a significantly larger dataset, comprising samples collected from various climatic zones across Iran (Beck et al., 2018) and dating between the 11th and 14th centuries. This expanded dataset offers a more robust opportunity to test the hypothesis of a positive correlation between anhydrite content in the samples and hot-desert climates.

This analysis reveals that samples with a high percentage of anhydrite are predominantly located in hot-desert climates, which might be associated with the simultaneous high levels of UV and IR radiation from the sunlight, as well as the low levels of humidity (Fig. 5 and Fig. 10, Supplementary Materials_1 and Supplementary Materials_2). The macroscopic impact of this transition appears as deep cracks in the stucco decoration, which is uncommon for gypsum-based materials (Fig. 8f). Another macroscopic effect is the partial transition of gypsum plaster into a powdery matrix, with some parts still holding together. This contrasts with gypsum deteriorated by moisture, where the entire body typically becomes powdery. Microscopically, SEM micrographs show pseudomorphic gypsum crystals covered by smaller crystals (Fig. 9h), while XRD results of the same sample indicate the presence of anhydrite. However, not all samples from hot-desert climate zones exhibit high levels of anhydrite, which could be attributed to shadowing effects within interior spaces. Considering these results, projecting all the samples onto Iran's climatic map reinforces the hypothesis, showing that the majority of samples with higher anhydrite percentages are located in the country's hot-desert climatic zones.

5. Conclusion

The systematic approach in this study, combined with a large set of samples from 47 Seljuq and Ilkhanid stucco monuments located in diverse regions and climates of Iran, provides fresh insights into the underexplored field of archaeometry in stucco decorations. Our multimodal analytical approach revealed that the stucco samples contain varying amounts of calcium sulfate, calcium carbonate, and silicate/aluminosilicates. Some of these mixtures appear to have been intentionally crafted, such as the clay-gypsum mixture used during the Seljuq period across various locations and the gypsum-lime and calcite mixture identified in two monuments in Abarkuh. The microstructure analysis of the stucco, including the shape of newly grown gypsum crystals, their local orientation, and macroporosity (related to the water-to-plaster ratio), sheds light on production processes. For instance, the orientation of crystals around air bubbles point to the extent of mechanical work performed before the plaster setting. Spherical air bubbles suggest molded or cast gypsum, while deformed air bubbles in inner layers reflect less mechanical processing. The absence of air bubbles highlights effective troweling and flattening processes used for fine patterns on the uppermost layers. Our microstructural evidence also demonstrates that many stuccoes previously considered single-layer, low-relief decorations, were actually composed of at least two distinct layers.

The identification of various phases within the $CaSO_4 \bullet xH_2O$ mineral family in relation to Iran's diverse climates reveals the potential for solid-state transformation from gypsum to anhydrite, probably due to exposure to UV and IR in the absence of humidity. This transformation carries significant implications for understanding historical

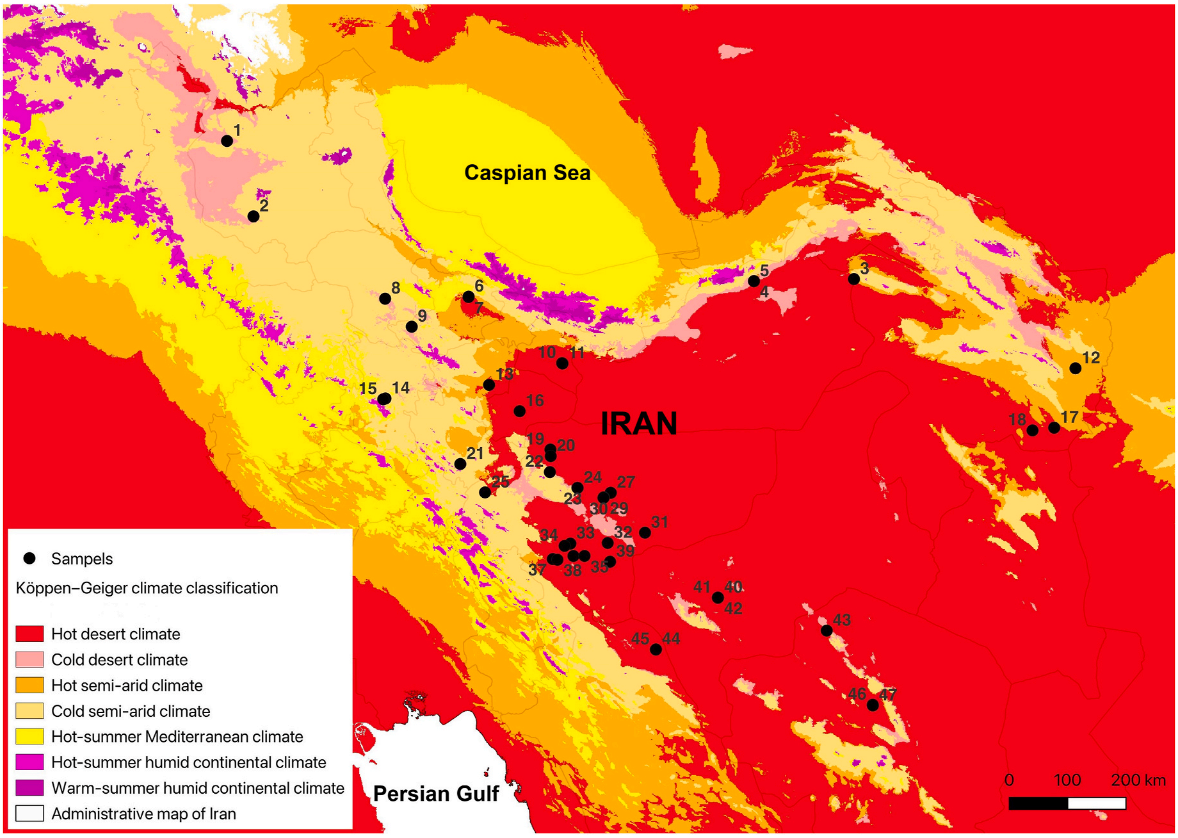

Fig. 10. The projection of collected samples on the relevant layers of the Köppen-Geiger climate classification map.

craftsmanship and poses challenges for preserving gypsum-based cultural materials. Considering the source of the raw materials, these findings challenge the reliability of using simple compositional analysis of the Ca/Sr ratio to determine raw material sources.

This research demonstrates that crucial insights into the production technology of historical stucco decorations can be effectively observed and analyzed using modern techniques. However, a comprehensive investigation at the current state of the art requires a large and diverse collection of samples. While the transformation of gypsum to anhydrite in hot-desert climates appears plausible, further experimental and theoretical research is necessary to definitively confirm this phenomenon.

CRediT authorship contribution statement

Moslem Mishmastnehi: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Data curation, Conceptualization. **Tomasz M. Stawski:** Writing – review & editing, Validation, Investigation, Formal analysis. **Negar Eftekhari:** Writing – review & editing, Software, Investigation, Formal analysis. **Kathrin P. Schneider:** Writing – review & editing, Software, Formal analysis, Data curation. **Carmela Vaccaro:** Writing – review & editing, Validation, Resources. **Iman Aghajani:** Writing – review & editing, Visualization, Data curation. **Ana Marija Grbanovic:** Writing – review & editing, Data curation, Conceptualization. **Lorenz Korn:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition, Conceptualization.

Data availability

All raw data is available as Supplementary Material.

Declaration of competing interest

The authors declare that there is no conflict of interest in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jas.2025.106199.

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