CHARACTERISTICS OF MORTARS AND PLASTERS OF SOME BATH BUILDINGS FROM AYDINOĞULLARI PRINCIPALITY IN SELÇUK, İZMİR

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İZMİR

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ABSTRACT

CHARACTERISTICS OF MORTARS AND PLASTERS OF SOME BATH BUILDINGS FROM AYDINOĞULLARI PRINCIPALITY IN SELÇUK, İZMİR

This study examines the properties of the lime mortar and plaster of Isa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath, the last bath buildings that preserved their authentic material structure from the Aydınoğulları Principality in Selçuk. The basic physical properties, raw material, chemical and mineralogical composition, pozzolanic and hydraulic properties of the mortars and plasters were investigated using RILEM standard test methods, SEM-EDS, XRD, and TGA.

The interior walls of the baths exhibit two distinct plaster layers in different colors at the lower and upper levels. Lower-level plasters consist of plaster with natural stone aggregate and Horasan plaster, while upper-level plasters consist of plaster with natural stone aggregate and/or lime plaster. The average density and porosity values of Horasan plasters and plaster with natural stone aggregate were 1.71 - 1.48 g/cm³ and 26.83% - 30.70%, respectively. Plasters with natural stone aggregate have higher lime content and lime/aggregate ratio. The natural aggregates used in the mortars and plasters were derived from a raw material source containing minerals of volcanic origin. Brick aggregates were manufactured using low calcium clay at temperatures below 850 ºC. Mortars and plasters have hydraulic characteristics due to the pozzolanic properties of the aggregates used.

Basic physical, chemical, and mineralogical compositions and hydraulic properties of mortars and plasters did not have significant differences according to the buildings. The use of raw materials and production techniques with similar properties to produce hydraulic mortars and plasters in historical bath buildings for many years indicates the continuity of local knowledge.

ÖZET

İZMİR SELÇUK'TAKİ AYDINOĞULLARI BEYLİĞİNDEN BAZI HAMAM YAPILARININ HARÇLARININ VE SIVALARININ ÖZELLİKLERİ

Bu çalışmada, İzmir'in Selçuk ilçesinde Aydınoğulları Beyliği Dönemi'nden özgün malzeme yapısını koruyarak günümüze ulaşan son hamam yapıları olan İsa Bey Hamamı, Kale Altı Hamamı ve Yahşi Bey Hamamı'nın kireç harcı ve sıvalarının özellikleri belirlenmiştir. Harç ve sıvaların temel fiziksel özellikleri, hammadde kompozisyonları, kimyasal ve mineralojik yapıları ile puzolanik ve hidrolik özellikler; RILEM standart test yöntemleri, SEM-EDS, XRD ve TGA analizleri kullanılarak tespit edilmiştir.

Hamamların iç duvarlarında alt ve üst seviyede farklı renklerde olmak üzere iki ayrı sıva tabakası görülmektedir. Alt seviyedeki sıvalar doğal taş agregalı sıva ve tuğla agregalı Horasan sıvadan; üst seviyedeki sıvalar ise doğal taş agregalı sıva ve/veya kireç sıvadan oluşmaktadır. Horasan sıvaların ve doğal taş agregalı sıvaların ortalama yoğunluk ve porozite değerleri sırasıyla 1,71 g/cm³ ila 1,48 g/cm³ ve %26,83 ila %30,70 olarak bulunmuştur. Doğal taş agregalı sıvalar ise daha yüksek kireç içeriğine ve kireç/agrega oranına sahiptir. Hamamların harç ve sıvalarında kullanılan doğal agregalar, volkanik kökenli mineraller içeren bir hammadde kaynağından elde edilmiştir. Tuğla agregalar ise 850 ºC'nin altındaki bir sıcaklıkta düşük kalsiyumlu kil kullanılarak üretilmiştir. Harç ve sıvalar, kullanılan agregaların puzolanik niteliklerinden dolayı hidrolik özellik göstermektedir.

Harç ve sıvaların temel fiziksel, kimyasal ve mineralojik bileşimleri ile hidrolik özellikleri, yapılara göre önemli bir farklılık göstermemiştir. Hidrolik harç ve sıva üretimi için benzer özelliklere sahip hammaddelerin ve üretim tekniklerinin tarihi hamam yapılarında uzun yıllar boyunca kullanılması, yerel bilgi birikiminin sürekliliğini göstermektedir.

To my dear family...

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CHAPTER 1

INTRODUCTION

Mortars and plasters are artificial materials that are well suited for obtaining archaeometric information about historical buildings. The manufacturing technologies of historical lime mortars and plasters should be investigated to preserve their historical, aesthetic, technical, and documentary values and to ensure that they are preserved for future generations (ICOMOS 1964). To preserve the historic integrity of a building, it is essential to respect the aesthetic values inherent in traditional craftsmanship and materials while also preserving the original material characteristics of the building (ICOMOS 1993; 1999).

The production of mortars depends on the technological knowledge of the manufacturer who produces them, while their composition largely depends on the raw materials available in the area where the mortars are produced (Barca et al. 2013). Depending on its characteristics, the mortar used can tolerate small deformations in the structure, thus ensuring a longer life of the structure (Budak 2005). The lime mortars and plasters have been used to hold the materials of structures built using brick or stone together, to fill joints, serve as an adhesive mortar, or to protect surfaces (Davey 1961). Such as, plasters protect building surfaces against water and moisture penetration, salt crystallization, wetting-drying and freeze-thaw cycles, and biological formations. They support fire resistance and additionally improve heat and sound insulation. They also offer high-impact resistance and can be easily repaired (Watts 2013). In addition to their functional requirements during the construction of the structures, they also have an aesthetic value depending on the construction techniques and applied locations (Arıoğlu and Acun 2006; Davey 1961). The use of plaster provides an aesthetic finish and an even, smooth surface suitable for painting or decoration (Watts 2013).

Throughout history, each generation has added its own practical experience to the production and use of mortars and plasters, and knowledge has been developed through trials and errors (Ward-Perkins 1970). The lime mortars and plasters are mixtures prepared by adding additives or fine aggregates compatible with various binders (Davey

1961). Materials that accelerate the setting time of lime mortars include ash, brick dust, and heated ceramic materials. These volcanic additives, called pozzolans were used by the Romans and were added to the mortar to enhance durability and ensure a positive setting time in lime mortars (Gibbons 1997; ASTMC618-03 2003; Ward-Perkins 1970). Roman lime mortar and plaster manufacturing techniques were used by various civilizations and extensive regions, until the widespread adoption of modern cement in the late 19th century (Arıoğlu and Acun 2006). Historic building materials should be preserved for future generations because lime mortars and plasters developed by civilizations over centuries are historical documents of technical knowledge, construction and material techniques, experience, and craftsmanship of the time and geography in which they were used (ICOMOS 1964).

Basic physical properties, raw material compositions, mineralogical and chemical components, microstructural properties, pozzolanic activity of aggregates and hydraulic properties, identities of mortars and plasters. For this reason, determining the characteristics of mortar and plasters will contribute to the decisions to be taken for the conservation of historical buildings, will enable the determination of the qualities that new materials to be used in restoration studies should have, and will facilitate the understanding of the manufacturing technologies of the period.

1.1. Problem Definition

Plaster and mortar are the building materials with the most rapid change and deterioration problems. These materials, which are historical documents, should be investigated and their characteristics should be determined. Research on plasters and mortars used in the 14th – 15th centuries is limited. The recent research was mostly focused on the characterization of mortar with natural or brick aggregates, horasan plasters, and lime plaster used in the Roman, Byzantine, or Ottoman Periods (Budak 2005; Çizer, Böke, and İpekoğlu 2004; Uğurlu 2005; Böke, Akkurt, and Ipekoğlu 2004; Solak 2016; Oğuz 2013; Oğuz, Türker, and Koçkal 2015; Gürhan 2018; Işık 2022).

The limited number of bath structures from the Principalities Period have survived, and some of them were restored which complicates the determination of the authentic characteristics of lime mortar and plaster. Research on the Aydınoğulları

Principality revealed that there were 10 baths in Selçuk dating from this period (Bellibaş, Ladstätter, and Kürüm 2013; Telci 2010; Ladstaetter et al. 2015). Only the Isa Bey, Kale Altı, Yahşi Bey and Saadet Hatun Baths have survived to the present day. The Saadet Hatun Bath has been restored, while the others have survived by preserving their authentic material properties.

In this study, the characteristics of the lime mortars and plasters used in the İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath were investigated. The increasing difficulty in accessing the authentic material of this period and the lack of information on the characteristics and manufacturing techniques of the lime mortars and plasters used by the Aydınoğulları Principality in Selçuk in the 14^{th}-15^{th} centuries emphasize the importance of this study.

1.2. Aim and Scope of the Study

This study aims to determine the characteristics of the lime mortars and plasters used on the interior walls of the 14^{th} -15^{th} century bath buildings in Selçuk, İzmir, and to understand the manufacturing technologies of mortars and plasters in Principalities Period according to levels, and layers. They will be examined to contribute to the studies to be carried out for the conservation of these structures.

In the scope of the study, three bath buildings, which are İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath were chosen. The proximity of these buildings, their construction and use during the same Principalities Period, and the fact that they have not been restored preserving their original material properties contributed to their selection for this study. In addition, those buildings were selected since they had similar architectural features, spatial organization, construction techniques, and material usage.

This study on the lime mortars and plasters used in the 14^{th} – 15^{th} century bath buildings will determine the characteristics of the materials to be used in future restoration studies.

1.3. Literature Review

The determination of the material properties of plasters and mortars and the effect of production techniques on these properties have been the subject of numerous articles and theses. These studies mainly aim to determine the physical, chemical, mineralogical, and microstructural properties of plaster and mortar samples from Roman (Kozlu and Ersen 2011; Taşcı and Böke 2018; Uğurlu Sağın, Duran, and Böke 2021; Miriello et al. 2011a; 2018), Byzantine (Işık 2022; Stefanidou et al. 2014; Oğuz 2013; Oğuz, Türker, and Koçkal 2015), and Ottoman (Çizer, Böke, and İpekoğlu 2004; Binici et al. 2010; Kozlu and Ersen 2011; İpekci, Uğurlu Sağın, and Böke 2019; Gürhan, Uğurlu Sağin, and Böke 2017) structures.

The study conducted by Miriello et al (2011) at the archaeological site of Kyme (Turkey) focused on the compositional characterization of Roman, Byzantine, and Medieval mortars. The mortars were analyzed by elemental (SEM-EDS, XRF), mineralogical (XRPD, De Astis calcimeter), and petrographic (polarized optical microscopy) analysis. The comparison between the samples revealed compositional differences within mortars from the same historical period. In particular, the widespread use of cocciopesto as a pozzolanic additive to create hydraulic mortars in the Roman and Byzantine periods was identified. Furthermore, compositional similarities allowed attributing a sample of unknown exact periods to the Roman period. Finally, the study points out that lower-quality mortars belong to the medieval period.

The characteristics of ancient lime mortar from Aigai and Nysa in Western Anatolia were examined to understand their technological practices (Uğurlu Sağın, Duran, and Böke 2021). Analysis through X-ray diffraction, X-ray fluorescence spectroscopy, scanning electron microscopy, and thermogravimetric analysis revealed hydraulic properties due to natural pozzolanic aggregates, predominantly dacite. While these aggregates shared mineralogical similarities, statistical analysis indicated distinct chemical compositions, suggesting varied local sources. This study underscores ancient Roman knowledge and the deliberate use of pozzolans for producing durable hydraulic mortars in the eastern provinces.

The properties of the mortars used in the ancient Roman structures of Xanthos (Antalya), Patara (Antalya), and Tlos (Muğla) in southern Turkey were investigated by

Taşcı (2018) to determine the properties of new mortars to be used in the conservation works of the buildings (Taşcı and Böke 2018). In this context, the basic physical properties, raw material compositions, chemical, mineralogical, and microstructural properties of the mortars were determined using XRD, FTIR, and SEM-EDS.

The properties of the lime mortars and plasters from Kadıkalesi (Anaia) and Ayasuluk Hill were evaluated by taking into consideration the sites, construction periods, and functions of the samples by Tuğçe Işık (Işık 2022). Within the scope of this study, the basic physical, geological, mineralogical, and chemical composition, hydraulic, and microstructural properties of the mortars and plasters were analyzed by SEM-EDS, XRD, and TGA methods. The results were compared with Byzantine lime mortar studies.

In the study, the physical, chemical, mechanical, and microstructural properties of mortars from the Roman, Byzantine, and Seljuk periods at Andriake Harbour, Antalya, were investigated (Oğuz 2013; Oğuz, Türker, and Koçkal 2015). The samples were analyzed with petrographic evaluation, X-ray diffraction (XRD), scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDX), thermogravimetric analysis (TG/DTA), X-ray fluorescence (XRF), and various physical tests including unit weight, water absorption, porosity, and acid loss. Despite variations in construction materials, the study reveals consistent properties across historical periods. These findings underscore the enduring quality and construction techniques employed over centuries in the structures of Andriake Harbour.

The study conducted by Hale Kozlu (Kozlu and Ersen 2011) aims to determine the characteristics of the plaster and masonry mortars used in Kayseri, which has a rich potential for volcanic materials, and to produce new mortar suggestions that can be used in the restoration of these buildings. Within the scope of the study, samples were taken from the Roman, Byzantine, Seljuk, and Ottoman periods structures and analyzed physically, chemically, mechanically, and petrographically. As a result of these analyses, original compositions were determined and samples with similar properties were grouped. Existing raw material sources for repair mortars that can be used in the restoration of the buildings were investigated, samples taken from reactive aggregate, lime, and stone quarries were analyzed and the materials closest to the original materials were determined. The physical and mechanical properties of mortars and plasters produced in the laboratory environment were measured within 6 months and compared with the original samples.

Emre İpekçi examined the horasan and lime plasters of Zeyrek Çinili Bath (16th c.), an Ottoman period building, to determine the application techniques, basic physical properties, and raw material compositions of the plasters (İpekci, Uğurlu Sağın, and Böke 2019). The microstructural properties, hydraulicity, mineralogical properties, chemical composition of binders, and pozzolanic activity of crushed brick aggregates were analyzed using standard test methods, binocular microscope, XRD, SEM-EDS, and TGA.

In the study, the characteristics of horasan and lime plasters from Aydın Eski Hamam, a typical Ottoman bath building dating back to the 15th-16th centuries, were examined (Gürhan, Uğurlu Sağin, and Böke 2017). Samples of plasters, joint mortars, and bricks were analyzed for physical properties, raw material compositions, microstructural details, hydraulic properties, and mineralogical and chemical compositions. Results indicated consistent low density and high porosity across all samples. Horasan plasters were found to contain pure lime and brick aggregates, exhibiting hydraulic properties due to pozzolanic aggregates. The study underscores the significance of material selection and production techniques in the restoration of historical structures.

Özlem Çizer conducted a study investigating the properties of lime mortars used in the construction of Ottoman bath buildings (Çizer, Böke, and İpekoğlu 2004). The study aimed to determine the physical properties, microstructures, raw material compositions, mineralogical and chemical properties of the raw materials used in the mortars. The results of the study highlight the importance of correct material selection and production techniques in the restoration of historical buildings.

1.4. Limits of the Study

The characteristics of the plasters and mortars used in the Isa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath of the Principalities Period in Selçuk, Izmir were examined within the scope of the Master of Science thesis. Only a limited number of bath structures from the Principalities Period have survived in Selçuk, one of them has been restored, while the others examined in this study have preserved their authentic mortar and plaster properties. The accessibility to the baths is limited due to their location and physical condition. The mortar and plaster samples from the interior walls of the baths, which did not exhibit any deterioration problems, were investigated.

The basic physical, chemical, and mineralogical properties, raw material compositions, and hydraulic properties of mortars and plasters were analyzed and the pozzolan activities of natural and brick aggregates were evaluated by scanning electron microscopy coupled with energy-dispersive X-ray spectrometry (SEM-EDX), X-ray diffraction (XRD), and thermogravimetric analysis (TGA). The investigation of the microstructural and morphological properties of the mortars and plasters could not be carried out due to the breakdown of the scanning electron detector (SE), Philips XL 30S FEG Scanning Electron Microscope (SEM) coupled with Energy Dispersive X-ray analysis (EDX) device of the IZTECH Materials Research Center.

1.5. Methodology and Content of The Study

This case study examines the characteristics of the lime mortars and plasters of the 14^{th} – 15^{th} centuries, based on the samples taken from the İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath in Selçuk, Izmir. The data were collected through, field surveys, and experimental studies.

The field survey, including the documentation and sampling, was conducted in March 2022. Sampling locations were documented with photographs and sketches. The samples were collected from the interior walls of the baths.

In the experimental studies, the samples were analyzed with laboratory investigations between June 2022 and January 2024 to determine the basic physical, chemical, and mineralogical properties, raw material compositions of the mortars and plasters were analyzed and pozzolan activities of natural and brick aggregates were evaluated. The samples were analyzed at the Materials Conservation Laboratory of Architecture Faculty and Materials Research Center (scanning electron microscopy coupled with energy-dispersive X-ray spectrometry (SEM-EDX), X-ray diffraction (XRD), and thermogravimetric analysis (TGA)) of Izmir Institute of Technology (IZTECH). The results were discussed in consideration of the construction period which was built between the 13th and 16th centuries, proximity, use of mortar and plaster, and aggregate type (Budak 2005; Çizer, Böke, and İpekoğlu 2004; Uğurlu 2005; Böke, Akkurt, and Ipekoğlu 2004; Solak 2016; Oğuz 2013; Oğuz, Türker, and Koçkal 2015; Gürhan 2018; Işık 2022).

The thesis is structured into six chapters: Introduction, Historical Mortars and Plasters, The Geographical and Historical Features of Selçuk and the Architectural Features of Studied Bath Buildings, Experimental Methods, Results and Discussion, and Conclusion.

In the second chapter, the manufacturing history and techniques of lime mortars and plasters were explained, and recent studies on the characteristics and composition of historical lime mortars and plasters were given.

In the third chapter, the geographical and historical features of Selçuk and the general architectural features of studied bath buildings were explained.

The fourth chapter defined sampling procedures, sample definitions, and experimental methods used to characterize the mortars and plasters of studied bath buildings.

The fifth chapter contains the results of basic physical properties, raw material composition, and hydraulic characteristics of lime mortars and plasters. The mineralogical and chemical composition of aggregates, binders, and lime lumps, and the pozzolanic activity of aggregates were explained. The results were discussed with previous studies. In addition, tables and graphs of the results of the analysis were given under the relevant headings together with the results of previous studies.

The results were evaluated In the sixth section of the study.

CHAPTER 2

HISTORICAL MORTARS AND PLASTERS

This chapter examines the historical development and manufacturing techniques of mortars and plasters. In addition, the results of recent studies on the characterization of historical lime mortar and plasters were investigated in terms of their basic physical, raw material compositions, chemical and mineralogical compositions, hydraulic properties, and pozzolanic properties of aggregates.

2.1. Properties of Mortars and Plasters

Lime mortars and plasters are mixtures prepared by adding suitable additives or fine aggregates to various binders to connect building materials, make joints, or protect surfaces (Davey 1961; Borrelli 1999). One of the oldest uses of lime mortar is as a floor covering, and one of the earliest examples (6000 BC) of this use was found in Çayönü and Lepenski Vir (Oates 1998; Adam 2001; Öztan 2009; Özdoğan 2007). Throughout history, different civilizations have used lime mortars as a non-structural plastering material for wall paintings, stuccos, and coatings (Adam 2001). The Romans generally preferred lime plaster as a cladding material for modest buildings (Başgelen 1993; Adam 2001). In the 2nd century BC, the Romans improved the mechanical strength of lime mortar by adding volcanic materials with pozzolanic properties into traditional mortar mixtures (Ward-Perkins 1970; Adam 2001). The use of lime mortars became more prevalent, allowing builders to utilize them for structural purposes. This resulted in their application in bonding rubble masonry, stabilizing arches, and the construction of vaulted structures (Adam 2001). Roman lime mortar technology continued to be used by many civilizations. In the Byzantine period, the use of lime plaster and mortar was a common practice for the interior walls of buildings, also there were wall paintings on the plaster (Başgelen 1993). During the Principalities Period, lime mortar was used in the walls of buildings, often with the addition of building stone dust. For structures associated with water, horasan mortar, and plasters manufactured with broken tiles or bricks were preferred, while in a smaller number of buildings, mortar mixed with ash was used (Kuban 2002).

In the production of lime mortars and plasters, organic and inorganic materials, natural and/or brick aggregates were mixed with lime which is used as a binder to improve the properties of mortars and plasters (Böke, Akkurt, and Ipekoğlu 2004). Lime was produced by the calcination of limestone and the slaking of quicklime (Lynch 1998). Limestone, a naturally occurring mineral composed of calcium carbonate ($CaCO_3$), is the primary raw material used in the production of lime (Davey 1961; Boynton 1980).

Lime classification has been approached by Davey (1961) and Vicat (1837). Davey categorized limes based on the origin of the limestone, its mineralogical and chemical composition, and classified them as hydraulic and non-hydraulic (Davey 1961) (Table 2.1). According to Vicat, non-hydraulic limes are fat or rich limes and lean or poor limes; hydraulic limes are divided into feebly, moderate, and eminently (Vicat 1837) (Table 2.1).

Lime is a general term that includes various chemical and physical forms such as quicklime, slaked lime, and hydraulic lime; it is an inorganic binder obtained by firing limestone ($CaCO_3$) or dolomite ($CaMg(CO_3)_2$) at high temperatures (850-1400 ºC), when mixed with water, it hardens either in water or air, depending on the type (Ashurst and Ashurst 1990; Torraca 1996; Borrelli 1999)

Table 2.1. Classification of Lime (Vicat 1837; Davey 1961)

Classification	Non-Hydraulic Limes		Hydraulic Limes		
	Fat or Rich Limes	Lean or Poor Limes	Feebly 0.1<H.I<0.2	Moderately H.I>0.2	Eminently H.I>0.2
Raw Materials	Carboniferous and pure oolitic limestone ($CaCO_3$) including <5% MgO, or magnesian (dolomitic) limestone ($MgCO_3$) containing >5% MgO.		Siliceous, grey chalks, argillaceous limestones or magnesian limestones containing CaO+ MgO (78–92%) SiO_2 (4–16%), Al_2O_3 (1–8%), and Fe_2O_3 (0.3–6%).		
Composition	>94% CaO, MgO	>70% CaO, MgO Residue inert	< 12% active clay, etc.	12–18% active clay, etc.	18-25% active clay, etc.
Set in Water	–	–	> 20 days	15-20 days	2-4 days
Calcination Temperatures	550 – 900 °C		>1000 °C		

Lime production consists of several stages, including calcination, slaking, and carbonation (Figure 2.1). Limestone ($CaCO_3$) is heated to 900°C to form quicklime (CaO). During calcination, CO_2 and moisture are removed from limestone, leaving behind reactive quicklime (CaO) (2.1) and losing weight, which makes transportation to construction sites more convenient (Davey 1961; Boynton 1980; Borrelli 1999).

$$CaCO_3 \xrightarrow{\text{Heat} \sim 900\,^{\circ}C} CaO + CO_2 \qquad (2.1)$$

$CaCO_3$: Limestone

CaO : Quicklime

Historically, lime production involved a calcination process that was mostly conducted in lime kilns located near limestone quarries to minimize labor requirements

(Figure 2.2 – 2.3) (Oates 1998; Davey 1961). Traditional lime kilns, which utilized wood and coal as fuel, had lower calcination temperatures of approximately 900°C. Consequently, this may have been produced quicklime with a higher specific surface area and greater reactivity, leading to a higher quality of lime (Antonia Moropoulou, Bakolas, and Aggelakopoulou 2001).

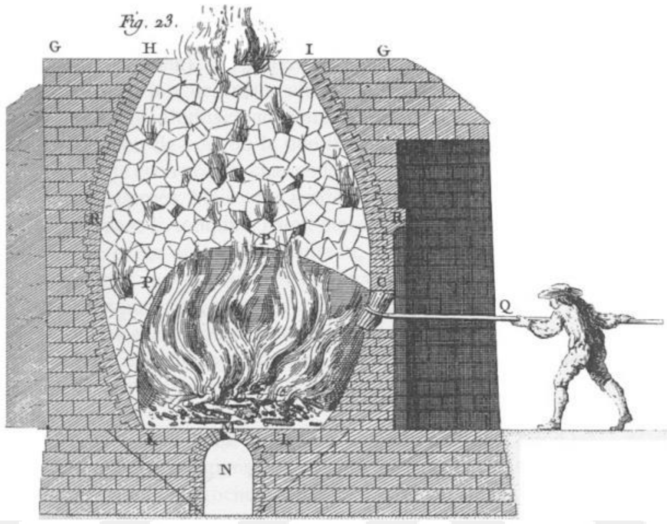


Figure 2.1. Traditional lime Kiln (Source: Vaschalde, Durand, and Figueiral 2008)

Following the calcination process, quicklime is converted to slaked lime (calcium hydroxide ($Ca(OH)_2$)) by the addition of water (2.2) (Davey 1961; Boynton 1980; Borrelli 1999). Lime slaking aims to produce calcium hydroxide, which can react with carbon dioxide to form calcium carbonate ($CaCO_3$) for use as a binding agent in mortars and plasters (Ashurst and Ashurst 1990; Borrelli 1999). To improve plasticity and carbonation by reducing the size of calcite crystals, it is recommended that slaked lime be aged for at least three years (Vitruvius 1914; Koenraad Van Balen 2003; Cowper 1998).

$$CaO + H_2O \longrightarrow Ca(OH)_2 \quad \text{(Heat)} \tag{2.2}$$

CaO: Quicklime Ca(OH)2: Slaked lime (Calcium Hydroxide)

Carbonation is the process by which slaked lime (calcium hydroxide) reacts with carbon dioxide (CO2) from the air (Koenraad Van Balen 2003). The carbonation depends on factors such as the amount of water, the concentration of CO2 in the air, and the permeability of the lime (Balen and Gemert 1994; Balen 2003).

$$Ca(OH)_2 + CO_2 \longrightarrow CaCO_3 + H_2O \qquad (2.3)$$

$Ca(OH)_2$: Slaked lime (Calcium Hydroxide) $CaCO_3$: Calcium Carbonate

Lime mortars/plasters are produced by mixing homogeneously aggregates with lime. The quality of historic lime mortars and plasters depends on the properties of the lime and aggregates, and the proportions of it used (Vitruvius 1914). Lime is divided into two categories:

- Hydraulic Lime
- Non-Hydraulic Lime

Aggregates are divided into two categories:

- Pozzolanic Aggregate
  - Natural Pozzolans: Natural stones of volcanic origin like volcanic dust and ash.
  - Artificial Pozzolans: Heated ceramic materials like bricks, tiles, ceramic dust, etc.
- Inert Aggregate: Sand, gravel etc.

Inert aggregates consist of inactive silicates and aluminates and do not react with lime, pozzolanic aggregates consist of active silicates and aluminates and react with lime (Cowper 1998; Boynton 1980). Lime mortars and plasters have hydraulic properties due to the pozzolanic properties of the aggregates used in their composition (A. Moropoulou, Bakolas, and Bisbikou 1995; A. Moropoulou et al. 2002). The production of non-hydraulic lime mortars/plasters occurs by using non-hydraulic lime (pure lime) with inert aggregates, whereas hydraulic lime mortars/plasters can be manufactured using hydraulic lime with inert aggregates or by mixing non-hydraulic lime with pozzolanic aggregates.

In historical buildings, especially in areas with high humidity, horasan plaster was produced with artificial pozzolans obtained by crushing and firing clay products such as bricks and tiles. The combination of lime and artificial pozzolans in mortars and plasters provides water setting and high mechanical strength (Böke, Akkurt, and Ipekoğlu 2004). When clay-rich raw materials are fired at 600°C to 900°C, the crystalline structures of the clay minerals break down, resulting in the formation of amorphous substances with pozzolanic properties (Davey 1961). At firing temperatures higher than 900°C, minerals and amorphous substances are degraded due to the high heat, leading to a decrease in pozzolanic activity (Cowper 1998).

2.2. Mortar and Plaster Used in Recent Studies

Studies on mortars and plasters focus on the characterization of the materials and the properties of their use from different archaeological sites and historical buildings. The material characterization of mortar and plasters from the 14th – 15th centuries were investigated in limited studies (Budak 2005; Esen et al. 2004; Çizer, Böke, and İpekoğlu 2004; Böke, Akkurt, and Ipekoğlu 2004; Uğurlu 2005; Oğuz, Türker, and Koçkal 2015; Solak 2016; Stefanidou et al. 2014; Gürhan 2018). The general aims of these studies were to determine the characteristics of mortars and plasters, to understand their manufacturing technologies, and to contribute to the conservation and restoration process of historic buildings.

2.2.1 Basic Physical Properties

The basic physical properties of lime mortars and plasters were generally defined by density (g/cm^3) and total porosity (%) the RILEM test (Budak 2005; Çizer, Böke, and İpekoğlu 2004; Böke, Akkurt, and Ipekoğlu 2004; Uğurlu 2005; Gürhan 2018; Stefanidou et al. 2014; Işık 2022; Solak 2016) and TS EN 1936 (Oğuz 2013; Oğuz, Türker, and Koçkal 2015) were used to determine the basic physical properties.

The density and porosity values of mortars with natural aggregates from 14th-century structures such as Çukur Bath, Hacet Mescidi (Budak 2005), Kızıl Han, Karapaşa Madrassah, and Yelli Mosque (Solak 2016), as well as Taşdibi Andriake Port (Oğuz 2013; Oğuz, Türker, and Koçkal 2015), and 15th-century baths like Düzce Bath, Herekzade

Bath, and Kamanlı Bath Bath (Çizer, Böke, and İpekoğlu 2004), along with Byzantine structures including Kadıkalesi Anaia (13-14th c.) and Ayasuluk Hill (6th c.) (Işık 2022), were less dense and more porous.

Plasters with brick aggregates, used in various structures, exhibit similar range density and total porosity values. The horasan plaster which is manufactured with lime and brick aggregate from Çukur Bath (14th c.) (Budak 2005), Ördekli Bath (14th c.), Beylerbeyi Bath (15th c.), Saray Bath (15th c.) (Böke, Akkurt, and Ipekoğlu 2004), Düzce Bath (15th c.), Herekzade Bath (15th c.), Kamanlı Bath (15th c.) (Uğurlu 2005), Eski Bath (15-16th c.) (Gürhan 2018), Kadıkalesi Anaia (13-14th c.) (Işık 2022) were less dense more porous materials. The multi-layer horasan plaster application creates a waterproof surface with a less porous finishing layer that prevents the structure from absorbing water at bath structures.

On the other hand, the lime plasters had density values ranging from 1.3 g/cm^3 to 1.8 g/cm^3 in baths examined (15th c.) by Uğurlu 2005; density values were found 1.3 – 1.7 g/cm^3 in Eski Bath by Gürhan (2018). The porosity values of the lime plasters ranged from 24% to 41% in Baths studied by Uğurlu (2005) and were found 29 – 52% in Eski Bath (Gürhan 2018).

2.2.2 Raw Material Compositions

The identification of raw material compositions was conducted through the analysis of lime/aggregate ratios and particle size distributions. In recent studies, lime/aggregate ratios were determined according to the weight ratios of lime, which is the acid-soluble part, and aggregate, the insoluble part by dissolving mortar and plaster using diluted hydrochloric acid. Also, in all studies, particle size distributions were determined by sieve analysis on the acid-insoluble (aggregate) components of the mortars and plasters after the dissolution (Budak 2005; Böke, Akkurt, and Ipekoğlu 2004; Çizer, Böke, and İpekoğlu 2004; Uğurlu 2005; Işık 2022; Oğuz 2013; Oğuz, Türker, and Koçkal 2015; Solak 2016; Gürhan 2018) (Table 2.2).

According to the research, the lime/aggregate ratios of the mortars with natural aggregate used in the bath structures of the 14-15th centuries (Çizer, Böke, and İpekoğlu

2004; Budak 2005; Böke, Akkurt, and Ipekoğlu 2004) are almost in the same range. In addition, the mortars of some historical buildings in different regions and periods such as the 13th-century Byzantine structure Kadıkalesi Anaia, the 6th-century Ayasuluk Hill (Işık 2022), the 13th-century buildings of the Principalities period in Muğla (Solak 2016) and Antalya (Oğuz 2013) also have similar lime/aggregate ratios.

The lime/aggregate ratio of plasters with brick aggregates from 14-15th century bath buildings was found 1.1, 0.5 – 1.25, 0.37 – 1.38, 0.6 – 0.7 by Budak (2005), Uğurlu (2005), Işık (2022), Gürhan (2018) respectively. According to the lime/aggregate ratios of the lime plasters, the proportion of pure lime used in lime plaster production is higher than the ratio of fine aggregate and sand in the Ottoman Period buildings such as Düzce, Herekzade, Kamanlı, and Eski Baths (Uğurlu 2005; Gürhan 2018).

The aggregate grain size distribution, as determined by sieving analysis, is another factor that determines the composition of raw materials used in the production of mortars and plasters. The particle size distribution of the mortars indicated that aggregates with particle sizes of 1180µm were a major fraction of the total aggregates (Budak 2005; Çizer, Böke, and İpekoğlu 2004; Işık 2022; Solak 2016; Stefanidou et al. 2014; Oğuz, Türker, and Koçkal 2015; Oğuz 2013). The size distribution of aggregate particles used in historical mortars produced in different periods is similar. The analysis of the lime plasters and horasan plasters revealed that aggregates less than 125 µm are the main fraction in recent studies (Uğurlu 2005; Gürhan 2018; Budak 2005).

Table 2.2. Methods from recent studies for determining the basic physical properties and raw material compositions of mortars and plasters in different structures

Location - Referances		Period of Samples	Sample Types	Aggregate Type	Basic Physical Properties	Raw Material Compositions	
						Lime/Aggregate Ratio	Particle Size Distributions
Çukur Hamam - Manisa	Budak 2005	14th century	Mortar	Natural	RILEM test methods	Acid loss analysis	Sieve analysis on acid insoluble parts
			Plaster (H)	Brick			
Hacet Mescidi - Manisa			Mortar	Natural			
Ördekli Bath - Bursa	Böke, Akkurt, and Ipekoğlu 2004	14th century	Plaster (H)	Brick	RILEM test methods	Acid loss analysis	Sieve analysis on acid insoluble parts
Beylerbeyi Bath - Edirne		15th century	Mortar	Brick			
			Plaster (H)	Brick			
Saray Bath - Edirne			Mortar	Brick			
			Plaster (H)	Brick			
Düzce Bath - Urla, İzmir	Çizer, Böke, and İpekoğlu 2004; Uğurlu 2005	15th century	Mortar (Çizer et al. 2004)	Natural	RILEM test methods	Acid loss analysis	Sieve analysis on acid insoluble parts
			Plaster (H_L) (Uğurlu 2005)	Brick_Natural			
Herekzade Bath - Urla, İzmir			Mortar (Çizer et al. 2004)	Natural			
			Plaster (H_L) (Uğurlu 2005)	Brick_Natural			
Kamanlı Bath - Urla, İzmir			Mortar (Çizer et al. 2004)	Natural			
			Plaster (H_L) (Uğurlu 2005)	Brick_Natural			
Kadıkalesi Anaia (K) - Kuşadası	Işık 2022	13th - 14th c.	Mortar	Natural	RILEM test methods	Acid loss analysis	Sieve analysis on acid insoluble parts
			Plaster	Brick			
Ayasuluk Hill (A) - Selçuk, Izmir		5th- 6th c.	Mortar	Natural			
Taşdibi Andriake Port- Antalya (Oğuz, Türker, and Koçkal 2015; Oğuz 2013)		13th century	Mortar	Natural	TS EN 1936 standard	Acid loss analysis	Sieve analysis on acid insoluble parts
Kızıl Han - Muğla	Solak 2016	14th century	Mortar	Natural	RILEM test methods	Acid loss analysis	Sieve analysis on acid insoluble parts
Karapaşa Madrasah - Muğla			Mortar	Natural			
Yelli Mosque - Muğla			Mortar	Natural			
Byzantine Bath - Greece	Stefanidou et al. 2014	13th - 14th c.	Mortar	Natural	RILEM test methods	X	X
Pazar Bath - Greece		16th c.	Mortar	Brick			
Eski Bath - Efeler, Aydın (Gürhan 2018)		15th - 16th c.	Plaster (H_L)	Brick_ Natural	RILEM test methods	Acid loss analysis	Sieve analysis on acid insoluble parts

2.2.3. Characteristics of Lime Lumps, Aggregates, and Binders

In recent studies, chemical and mineralogical composition analyses were focused on three specific aspects: aggregates, binders, and lime lumps.

A scanning electron microscope equipped with an X-ray energy dispersing system (SEM-EDS) was used to determine the chemical compositions, and data were averaged from three different regions of the pellet prepared using samples finer than 53 μm; also X-ray fluorescence spectroscopy (XRF) was used in studies to determine major, minor and trace elemental compositions (Budak 2005; Böke, Akkurt, and Ipekoğlu 2004; Çizer, Böke, and İpekoğlu 2004; Uğurlu 2005; Solak 2016; Stefanidou et al. 2014; Işık 2022; Gürhan 2018; Oğuz 2013; Oğuz, Türker, and Koçkal 2015) (Table 2.3).

X-ray diffraction (XRD) analysis was utilized in previous studies to investigate the mineralogical compositions (Budak 2005; Böke, Akkurt, and Ipekoğlu 2004; Çizer, Böke, and İpekoğlu 2004; Uğurlu 2005; Solak 2016; Stefanidou et al. 2014; Işık 2022; Gürhan 2018; Oğuz 2013; Oğuz, Türker, and Koçkal 2015) (Table 2.3).

The pozzolanic activity of aggregates in mortars and plasters was determined by the electrical conductivity method and/or the high amount of SiO_2 + Al_2O_3 + Fe_2O_3 content, in previous studies (Budak 2005; Çizer, Böke, and İpekoğlu 2004; Böke, Akkurt, and Ipekoğlu 2004; Uğurlu 2005; Solak 2016; Oğuz 2013; Oğuz, Türker, and Koçkal 2015; Stefanidou et al. 2014; Gürhan 2018; Işık 2022) (Table 2.4).

The previous studies investigating the characteristics of historical materials have examined the hydraulic properties of mortars and plasters using thermogravimetric analysis (TGA), the hydraulic (H.I) and cementation (C.I) index according to the Boynton formula (Budak 2005; Çizer, Böke, and İpekoğlu 2004; Böke, Akkurt, and Ipekoğlu 2004; Uğurlu 2005; Solak 2016; Oğuz 2013; Oğuz, Türker, and Koçkal 2015; Stefanidou et al. 2014; Gürhan 2018; Işık 2022) (Table 2.4).

2.2.3.1. Chemical and Mineralogical Composition of Lime Lumps

The chemical and mineralogical composition of lime lumps, which define the properties of lime used in mortar and plaster production, has been investigated in various studies.

The chemical compositions of the lime lumps from different periods and structures were composed of higher amounts of CaO, and lower amounts of SiO_2, Al_2O_3, Fe_2O_3, MgO, K_2O, and Na_2O (Budak 2005; Uğurlu 2005; Işık 2022; Çizer, Böke, and İpekoğlu 2004). The lime lumps from Ayasuluk Hill (6th c.), Kamanlı Bath (15th c.), Çukur Bath (14th c.), Hacet Mescid (14th c.), Düzce Bath (15th c.), and Herekzade Bath (15th c.), were composed of calcite (Budak 2005; Uğurlu 2005; Işık 2022; Çizer, Böke, and İpekoğlu 2004). The results of the analysis indicated that pure lime was used in the production of mortars and plasters. Additionally, the lime used in historical mortars and plasters did not possess hydraulic properties.

2.2.3.2. Chemical, Mineralogical Composition and Pozzolanic Properties of Aggregates

Natural aggregates in mortars and plasters:

The chemical compositions of the natural aggregates of mortars consisted of a higher amount of SiO_2, moderate amounts of Al_2O_3, and lower amounts of Na_2O, MgO, CaO, Fe_2O_3, K_2O, and TiO2 in from Çukur Bath (14th c.), Hacet Mescidi (14th c.) (Budak 2005), Ayasuluk Hill (6th c.), Kadıkalesi Anaia (13-14th c.) (Işık 2022). Düzce Bath (15th c.), Herekzade Bath (15th c.), and Kamanlı Bath (15th c.) consisted of a higher amount of SiO_2, moderate amounts of Fe_2O_3 and Al_2O_3, and lower amounts of K_2O, Na_2O, and MgO (Çizer, Böke, and İpekoğlu 2004). Stefanidou et al. (2014) calculated SiO_2+ Al_2O_3+ Fe_2O_3 (44.0 – 51.4%_22.15 – 33.2 %) and CaO values (20.7 – 28.7%_ 34.7 – 41.0 %) at Byzantine Bath (13-14th c.) and Pazar Bath (16th c.) respectively (Stefanidou et al. 2014).

The natural aggregates In mortars from Çukur Bath and Hacet Mescidi were composed of quartz and albite, anorthite, or muscovite minerals (Budak 2005). In

Kadıkalesi Anaia, the natural aggregates in the mortar consisted of quartz, albite, muscovite, and clinochlore minerals; in Ayasuluk Hill the natural aggregates in the mortar were composed of albite, clinochlore, hornblende, muscovite, orthoclase phillipsite, and quartz minerals (Işık 2022). The hornblende and phillipsite minerals found in the natural aggregates may have originated from volcanic regions. The albite, muscovite, and quartz minerals were found in natural aggregates of mortars from Düzce Bath, Herekzade Bath, and Kamanlı Bath (Çizer, Böke, and İpekoğlu 2004).

Additionally, the natural aggregates in lime plaster from the Eski bath were mainly composed of SiO_2 and smaller amounts of Al_2O_3, Na_2O, MgO, CaO, Fe_2O_3, K_2O, and TiO_2 (Gürhan 2018).

Brick aggregates in plasters:

The brick aggregates of plaster from the Byzantine period Ayasuluk Hill (6^{th} c.) and the Ottoman period (15-16^{th} c.) Herekzade, Kamanlı, Ördekli Beylerbeyi, Saray, and Eski Baths consisted of a higher amount of SiO_2, a moderate amount of Al_2O_3, and lower amounts of Fe_2O_3, MgO, Na_2O, K_2O, CaO, and TiO_2 (Işık 2022; Uğurlu 2005; Böke, Akkurt, and Ipekoğlu 2004; Gürhan 2018). The aggregates of horasan plaster from the Ottoman period (15^{th} c.) in Düzce Bath consisted of a higher amount of Al_2O_3 moderate amounts of SiO_2, and lower amounts of Fe_2O_3, MgO, Na_2O, K_2O, and CaO; (Uğurlu 2005).

Quartz and albite minerals were found in the plaster with brick aggregates of the Byzantine period structure Kadıkalesi Anaia (Işık 2022) and the Ottoman buildings of Ördekli, Beylerbeyi, Saray, Düzce, Herekzade and Eski Baths (Böke, Akkurt, and Ipekoğlu 2004; Uğurlu 2005; Gürhan 2018). Hematite and muscovite minerals were only found in the plaster with brick aggregates of byzantine periods Kadıkalesi Anaia (13^{th} c.) and Eski Baths (15-16^{th} c.). In addition, the horasan plasters of the Ottoman period structure Düzce (15^{th} c.), Herekzade (15^{th} c.), and Eski Bath (15-16^{th} c.) contain potassium feldspar minerals. Calcite minerals were derived from carbonated lime, quartz, and other siliceous minerals from brick dust. The main XRD peaks of calcium silicate hydrate and calcium aluminate hydrate were not observed in the horasan plaster matrices and researchers explained this by the amorphous structure of these hydraulic products (Uğurlu 2005; Haga et al. 2002). On the other hand, quartz, albite, hematite, muscovite, potassium feldspar, and calcite minerals were found in aggregates of lime plasters from Eski Bath (Gürhan 2018).

<u>Pozzolanic properties of fine aggregates:</u>

Kadıkalesi Anai (13-14th c.) and Ayasuluk Hill (6th c.) of the Byzantine Period, Hacet Masjidi and Çukur Hamam of the Principalities period, and Düzce (15th c.), Herekzade (15th c.) and Kamanlı (15th c.) baths of the Ottoman period have pozzolanic properties according to the differences in the electrical conductivity of the natural aggregates of mortar. The electrical conductivity difference was found for natural aggregates of mortars under 1,2 mS/cm in Antalya Taşdibi Andriake Port (Oğuz 2013; Oğuz, Türker, and Koçkal 2015), and in Kızıl Han, Karapaşa Madrasah and Yelli Mosque (Solak 2016). Therefore, they do not possess pozzolanic properties.

The Çukur Bath from the Principalities Period (14th c) and the Düzce, Herekzade, Kamanlı, Ördekli, Saray, Beylerbeyi, and Eski Baths from the Ottoman Period (15-16th c.) exhibited pozzolanic characteristics in horasan plasters aggregates (Uğurlu 2005; Böke, Akkurt, and Ipekoğlu 2004; Gürhan 2018; Budak 2005). Additionally, lime plasters from Eski Bath have pozzolanic properties (Gürhan 2018).

Two methods were used to examine the pozzolanic properties at Kadıkalesi Anaia and Ayasuluk Hill and in both methods, the aggregates of mortars exhibited pozzolanic properties, likewise in plasters with brick aggregates from Kadıkalesi Anaia (Işık 2022). The pozzolanic activity of the Pazar Bath and Byzantine Bath in Greece was determined through the content of $SiO_2 + Al_2O_3 + Fe_2O_3$, which was found between 22.15% and 51.4% by Stefanidou et al (2014) and they don't possess pozzolanic properties.

Pozzolan aggregates react with lime to form amorphous silicates and aluminates that allow the mortar to harden underwater (Palomo et al. 2002). Using pozzolanic aggregates in mortars and plasters increases the hydraulic properties of mortars and plasters (Budak 2005; Uğurlu 2005).

Table 2.3. Methods for determining the chemical and mineralogical composition of mortars and plasters

Location	References	Period of Samples	Sample Types	Aggregate Type	Chemical Compositions	Mineralogical Compositions
Çukur Hamam - Manisa	Budak 2005	14^{th} century	Mortar	Natural	SEM-EDS	XRD
			Plaster (H)	Brick		
Hacet Mescidi - Manisa			Mortar	Natural		
Ördekli Bath - Bursa	Böke, Akkurt, and Ipekoğlu 2004	14^{th} century	Plaster (H)	Brick	SEM-EDS	XRD
Beylerbeyi Bath - Edirne		15^{th} century	Mortar	Brick		
			Plaster (H)	Brick		
Saray Bath - Edirne			Mortar	Brick		
			Plaster (H)	Brick		
Düzce Bath - Urla, İzmir	Çizer, Böke, and İpekoğlu 2004; Uğurlu 2005	15^{th} century	Mortar (Çizer et al. 2004)	Natural	SEM-EDS	XRD
			Plaster (H_L) (Uğurlu 2005)	Brick_Natural		
Herekzade Bath - Urla, İzmir			Mortar (Çizer et al. 2004)	Natural		
			Plaster (H_L) (Uğurlu 2005)	Brick_Natural		
Kamanlı Bath - Urla, İzmir			Mortar (Çizer et al. 2004)	Natural		
			Plaster (H_L) (Uğurlu 2005)	Brick_Natural		
Kadıkalesi Anaia (K) - Kuşadası	Işık 2022	13^{th} - 14^{th} c.	Mortar	Natural	SEM-EDS	XRD
			Plaster	Brick		
Ayasuluk Hill (A) - Selçuk, Izmir		5^{th}- 6^{th} c.	Mortar	Natural		
Taşdibi Andriake Port- Antalya (Oğuz, Türker, and Koçkal 2015; Oğuz 2013)		13^{th} century	Mortar	Natural	SEM-EDS & XRF	XRD
Kızıl Han - Muğla	Solak 2016	14^{th} century	Mortar	Natural	SEM-EDS & XRF	XRD
Karapaşa Madrasah - Muğla			Mortar	Natural		
Yelli Mosque - Muğla			Mortar	Natural		
Byzantine Bath - Greece	Stefanidou et al. 2014	13^{th} - 14^{th} c.	Mortar	Natural	SEM-EDS	X
Pazar Bath - Greece		16^{th} c.	Mortar	Brick		
Eski Bath - Efeler, Aydın (Gürhan 2018)		15^{th} - 16^{th} c.	Plaster (H_L)	Brick_ Natural	SEM-EDS	XRD

2.2.3.3. Chemical, Mineralogical Compositions and Hydraulic Properties of Binders

Binders with natural aggregates:

The chemical compositions of the binders in the mortars with natural aggregates of Byzantine period structures Ayasuluk Hill (6th c.), Kadıkalesi Anaia (13-14th c.), and Principalities Period structures Karapaşa Madrasah (14th c.) and Taşdibi Andriake Port (13th c.) consisted of larger amounts of CaO and SiO_2, moderate amounts of Al_2O_3, and smaller amounts of MgO, Fe_2O_3 K_2O, Na_2O, and TiO_2 (Işık 2022; Solak 2016; Oğuz 2013; Oğuz, Türker, and Koçkal 2015).

The binders of the natural aggregate lime mortars of the Byzantine Period structures on Ayasuluk Hill are composed of albite, calcite, and hornblende minerals, Kadıkalesi Anaia were composed of albite, calcite, and quartz minerals. (Işık 2022). (Oğuz 2013; Oğuz, Türker, and Koçkal 2015). The binders of the lime mortars used in the Principality Period were found to consist of calcite, quartz, and albite at Çukur Hamam and Hacet Mascid; quartz, calcite, dolomite and feldspar minerals at Kızıl Han, Karapaşa Madrasah and Yelli Mosque; and calcite, quartz and dolomite minerals at Taşdibi Andriake Port (Budak 2005; Solak 2016; Oğuz 2013; Oğuz, Türker, and Koçkal 2015). On the other hand, the albite, quartz, and calcite minerals were found in binders of lime plasters from the Ottoman Period, Eski Bath (Gürhan 2018). Calcite was derived from carbonated lime, whereas quartz, albite, muscovite, clinochore, dolomite, hornblende, and plagioclase feldspar were from aggregates.

Binders with brick aggregates:

The chemical compositions of the binders in the plasters with brick aggregates of Byzantine period structures Ayasuluk Hill and Kadıkalesi Anaia; Ottoman Period structures Düzce, Herekzade, Kamanlı, and Eski Baths were composed of larger amounts of CaO , and SiO_2 , moderate amounts of Al_2O_3, and smaller amounts of Fe_2O_3, MgO, K_2O, Na_2O, and TiO_2 (Işık 2022; Uğurlu 2005; Gürhan 2018). The calcium oxide (CaO) was obtained from carbonated lime, while the silicon oxide (SiO_2) and aluminum oxide (Al_2O_3) were obtained from brick dust. The major oxides of binders in lime plasters from Eski Bath were mainly composed of CaO, and partly MgO, SiO_2, FeO, K_2O, Na_2O, and Al_2O_3 (Gürhan 2018).

The binders of plaster with brick ”ggre’ates from Kadıkalesi Anaia were composed of calcite, quartz, albite, muscovite minerals (Işık 2022). In Düzce Bath, Herekzade Bath, and Kamanlı Bath calcite, quartz, and albite minerals were found in the horasan plasters (Uğurlu 2005). The binders in horasan plasters of Eski Bath were composed of quartz, albite, hematite, muscovite, potassium feldspar, vaterite, and calcite minerals.

Hydraulic Properties:

The hydraulic character of lime mortars or plasters is indicated by a CO_2/H_2O ratio of less than 10 also, the non-hydraulic character of the materials is indicated by hydraulic index (H.I) values less than 0.1 and cementation index (C.I) values less than 0.3 (Eckel 2005; Boynton 1980; Bakolas et al. 1998).

The mortars with the natural aggregate of Çukur Hamam, Hacet Mescidi, Kızıl Han, Karapaşa Madrasa and Yelli Mosque, which are the buildings of the Principalities period, are hydraulic according to the CO2/H2O ratio; however, Taşdibi Andriake Harbor was found to be non-hydraulic (Budak 2005; Solak 2016; Oğuz 2013). Mortars with natural aggregates obtained from Byzantine period structures such as Byzantine Baths, Kadıkalesi Anaia and Ayasuluk Hill were found to have hydraulic properties (Işık 2022; Stefanidou et al. 2014).

The horasan plaster with brick aggregate used in Düzce, Herekezade, Kamanlı, and Eski Baths, which were constructed in the Ottoman period, and Çukur Baths, which belong to the Principality period, had hydraulic properties (Uğurlu 2005; Gürhan 2018). The CO_2/H_2O ratio of lime plasters was found less than 10 in Düzce Bath, Herekzade Bath, and Kamanlı Bath, this indicated that they don’t have hydraulic properties (Uğurlu 2005). The hydraulic properties of the lime plasters of the Eski Bath may be due to the pozzolanic natural aggregates.(Gürhan 2018).

The hydraulic characteristics of mortars and plasters were also determined using the hydraulic index (H.I.) at Byzantine period structures Kadıkalesi Anaia, and Ayasuluk Hill were found hydraulic (Işık 2022). According to the cementation index (C.I.), the Byzantine Bath and Greece Bath were hydraulic characteristics (Stefanidou et al. 2014). The hydraulic properties are due to the pozzolanic properties of aggregates but the heterogeneous structure of mortars and plasters, and the lime/aggregate ratios can influence the hydraulic properties.

Table 2. 4. Methods for determining the pozzolanic and hydraulic properties of mortars and plasters in previous studies

Location - Referances		Period of Samples	Sample Types	Aggregate Type	Methods for Pozzolanic Activity	Hydraulic Properties
Çukur Hamam - Manisa	Budak 2005	14^{th} century	Mortar	Natural	Electrical Conductivity Method	Thermogravimetric analysis (200-600 ^{o}C /600-900 ^{o}C)
			Plaster (H)	Brick		
Hacet Mescidi - Manisa			Mortar	Natural		
Ördekli Bath - Bursa	Böke, Akkurt, and Ipekoğlu 2004	14^{th} century	Plaster (H)	Brick	Electrical Conductivity Method	Thermogravimetric analysis (200-600 ^{o}C /600-900 ^{o}C)
Beylerbeyi Bath - Edirne		15^{th} century	Mortar	Brick		
			Plaster (H)	Brick		
Saray Bath - Edirne			Mortar	Brick		
			Plaster (H)	Brick		
Düzce Bath - Urla, İzmir	Çizer, Böke, and İpekoğlu 2004; Uğurlu 2005	15^{th} century	Mortar (Çizer et al. 2004)	Natural	Electrical Conductivity Method	Thermogravimetric analysis (200-600 ^{o}C /600-900 ^{o}C)
			Plaster (H_L) (Uğurlu 2005)	Brick_Natural		
Herekzade Bath - Urla, İzmir			Mortar (Çizer et al. 2004)	Natural		
			Plaster (H_L) (Uğurlu 2005)	Brick_Natural		
Kamanlı Bath - Urla, İzmir			Mortar (Çizer et al. 2004)	Natural		
			Plaster (H_L) (Uğurlu 2005)	Brick_Natural		
Kadıkalesi Anaia (K) - Kuşadası	Işık 2022	13^{th} - 14^{th} c.	Mortar	Natural	Electrical Conductivity Method & High amount of $SiO_2 + Al_2O_3 + Fe_2O_3$	Thermogravimetric analysis (200-600 ^{o}C /600-900 ^{o}C) & Hydraulic Index(H.I)
			Plaster	Brick		
Ayasuluk Hill (A) - Selçuk, Izmir		5^{th}- 6^{th} c.	Mortar	Natural		
Taşdibi Andriake Port- Antalya (Oğuz, Türker, and Koçkal 2015; Oğuz 2013)		13^{th} century	Mortar	Natural	Electrical Conductivity Method	Thermogravimetric analysis (200-600 ^{o}C /600-900 ^{o}C)
Kızıl Han - Muğla	Solak 2016	14^{th} century	Mortar	Natural	Electrical Conductivity Method	Thermogravimetric analysis (200-600 ^{o}C /600-900 ^{o}C)
Karapaşa Madrasah - Muğla			Mortar	Natural		
Yelli Mosque - Muğla			Mortar	Natural		
Byzantine Bath - Greece	Stefanidou et al. 2014	13^{th} - 14^{th} c.	Mortar	Natural	High amount of $SiO_2 + Al_2O_3 + Fe_2O_3$	Thermogravimetric analysis (200 - 600^{o}C / 600-800^{o}C) & Cementation Index (CI)
Pazar Bath - Greece		16^{th} c.	Mortar	Brick		
Eski Bath - Efeler, Aydın (Gürhan 2018)		15^{th} - 16^{th} c.	Plaster (H_L)	Brick_ Natural	Electrical Conductivity Method	Thermogravimetric nalysis (200-600 ^{o}C /600-900 ^{o}C)

CHAPTER 3

THE GEOGRAPHICAL AND HISTORICAL FEATURES OF SELÇUK AND THE ARCHITECTURAL FEATURES OF STUDIED BATH BUILDINGS

This chapter examines Selçuk’s location, geographical features, and brief history in general, with a particular focus on the architectural features of the İsa Bey, Kale Altı, and Yahşi Bey baths.

3.1. Geographical Features

Selçuk is a district of Izmir Province in western Turkey. It is surrounded by Menderes and Torbalı districts in the north, Tire and Germencik in the east, and Söke and Kuşadası in the south (Figure 3.1). It is in the Küçük Menderes River Basin, surrounded by the Panayır, Bülbül Mountains, and Ayasuluk Hill (Figure 3.2). To the west, it has a coast on the Aegean Sea with Pamucak Beach.

Figure 3.1. Locations of studied bath buildings with neighboring provinces

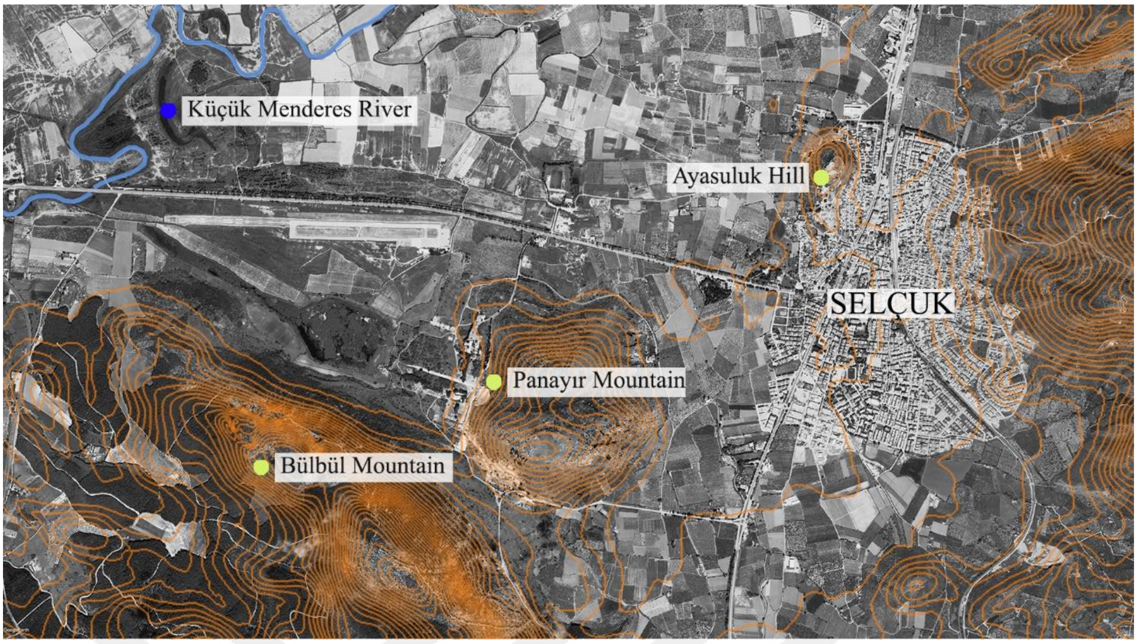


Figure 3.2. Map of Selçuk, mountains, and river
(Revised from Google Earth: 10.11.2023)

3.2. A Brief History of Selçuk

Selçuk is a town with a complex historical background, with evidence of human habitation dating back to 10.000 – 8.000 BC. Rescue excavations conducted in 1995 at the Çukuriçi (6.000 – 2.500 BC) and Arvalya (10.000 – 5.500 BC) mounds by the Ephesus Museum exposed that the earliest settlements in the region date back to the Neolithic period (Evren and İçten 1998). The Çukuriçi mound is located east of the Magnesia Gate of Ephesus, near the Küçük Menderes River (formerly the Kaysyros River). The Arvalya mound is located west of the city, near Pamucak Beach.

Excavations conducted between 1990 and 1996 exposed a part of the fortification wall and ceramic tiles thought to belong to the Hittite's Arzawa Kingdom (Erdemgil and Büyükkolancı 1991). Selçuk is accepted to have been the capital of the Arzawa Kingdom called Apasa (Büyükkolancı 1998). The name Apasas was eventually changed to Ephesos (Büyükkolancı 1998). The findings proved that the first settlement of Ephesus was located on Ayasuluk Hill. According to ancient writings, some civilizations, such as the Akhas, Lelegs, and Carians migrated to Anatolia to escape from the Dorian occupation, and it is possible that some of the immigrants settled in the primary settlement of the Apasas civilization (Strabon 1993).

Ephesus was conquered in the 11th century BC by Androklos, the leader of the Ionian colonization (Strabon 1993; Arıkan 1990). The societies that lived in the region settled around the Temple of Artemis and Ayasuluk Hill (Büyükkolancı 1998). In the 6th century BC, the Lydian king Croesus conquered Ephesus. After Lydian rule, Ephesus remained under the control of Persian king Cyrus (546 BC) , Alexander the Great (334 BC) and General Diadokhos Lysimakhos respectively (323 BC) (Scherrer 2000; Külzer 2011).

During the Hellenistic era, Ephesus functioned as a significant commercial hub but since many Roman buildings were added to the Hellenistic urban fabric, there are not many architectural remains that can be dated to this period (Arıkan 1990; Zimmermann and Ladstatter 2011). In 133 BC, Ephesus came under Roman control (Arıkan 1990; Scherrer 2000; Zimmermann and Ladstatter 2011). Ephesus retained its significance during the Roman era. Augustus declared Ephesus the capital city of Asia, and during this period construction activities increased in the city (Scherrer 1995; Zimmermann and Ladstatter 2011). The monumental structures that can be seen today reflect the wealth and prosperity of the period (Zimmermann and Ladstatter 2011).

Ephesus retained its significance and splendor until the 2nd century AD but suffered a severe blow with the Gothic invasion in 262 AD, which led to a significant slowdown in the development of the city (Foss 1979). A series of earthquakes in the 3rd century AD resulted in significant alterations to the overall appearance of the city (Ladstätter 2011). Research and excavations focusing on the Byzantine heritage indicate that the city retained its importance in the following centuries (Foss 1979; Ladstätter 2011; Pülz 2011).

Political, economic, and social activities influenced the spread of Christianity in the 3rd and 4th centuries AD, and it led to changes in the city during the Late Roman and Byzantine periods (Pülz 2011). In the 4th and 5th centuries, the city became an important Christian center (Külzer 2011). Construction activities increased, and religious buildings such as the Church of the Virgin Mary, the Basilica of St. John, and the Cemetery of the Seven Sleepers were built (Foss 1979; Külzer 2011). However, in the 7th century, the city lost its significance due to the invasions of the Persians and Arabs (Foss 1979; Ladstätter

2011). After the Battle of Malazgirt in 1071, the Turks captured Ephesus, but the city soon returned to Byzantine rule (Foss 1979; Külzer 2011). After this period, the settlement concentrated on Ayasuluk Hill, and construction activities decreased (Külzer 2011) The city was rebuilt and developed again in the 14th century, during the period of the Turkish Principalities (Beyliks) (Ladstaetter et al. 2015).

According to the Düsturnâme-I Enveri Ayasuluk was conquered by Aydınoğlu Mehmed Bey in 1304 (Uzunçarşılı 1969; Arıkan 1990; Baykara 1990). Aydınoğlu Mehmed Bey chose Birgi as the capital and divided the administration of cities among his sons, leaving Ayasuluk under the control of Hızır Bey (Telci 2010; Uzunçarşılı 1969). When Hızır Bey became the head of the Principalities after the administrations of his father and brother Umur Bey, he made Ayasluk the capital (Arıkan 1990; Telci 2010). During the administration of Hızır Bey, travelers who visited the city described Ayasuluk as an ancient and magnificent city (Foss 1979; Battuta 2004).

After Hızır Bey, the administration of the Principality passed to his brother, İsa Bey (Akın 1968). Under the rule of İsa Bey, the Principalities and Ayasuluk experienced heydays in the 14th century (Arıkan 1990). İsa Bey's emphasis on science, culture, and the arts increased construction activities in Ayasuluk (Arıkan 1990; Şeker 1998; Telci 2010). Archaeological findings on Ayasuluk Hill and its western and southern slopes indicated that this area, located between the Basilica of St. John and the Temple of Artemis, served as the city center (Arıkan 1990). Although written sources about the built environment of the 14th century fr Ayasuluk are limited, travelers (Ibn Battuta, Wilhelm von Boldensele, and Ludolf von Suchem) noted the wealth and well-maintained condition of the city (Foss 1979). Ephesus/Ayasuluk continued to exist as an important trade center and port city (Ladstaetter et al. 2015). The city experienced its last period of prosperity during the Aydınoğulları Principality until the early 15th century. It was attacked by the Mongols under the command of Timur in 1402. (Telci 2010; Arıkan 1990).

In 1424, the Ottoman Empire conquered Ayasuluk, and it was included in the Aydın region. With the use of the port of Kuşadası by the Aydın region, the importance of the port of Ayasuluk began to decrease. This development negatively affected the economic prosperity of Ayasuluk. Also, the long wars between the Ottomans and Aydınoğlu Principality caused damage to the city (Battuta 2004).

While construction activities continued in the early Ottoman period, the city lost its significance after the 15th century. Therefore, most of the structures seen in the city today belong to the Aydınoğulları Principality and the early Ottoman period (Figure 3.4.) (Arıkan 1990). In the 17th century, Evliya Çelebi and Cornelis de Bruyn, who visited the city, described the inhabitants of Ayasuluk as impoverished and the settlement was depicted as a ruined city (Evliya Çelebi 2011; Foss 1979). In the 19th century, travelers (Charles Texier, John Turtle Wood, James Dallaway, Joseph Pitton de Tourneford) who visited Ayasuluk stated that the city was a village consisting of a ruined city on the south slope of Ayasuluk Hill and inside the castle (Buch 1982; Texier 2002; Tournefort 2008; Wood 2014).

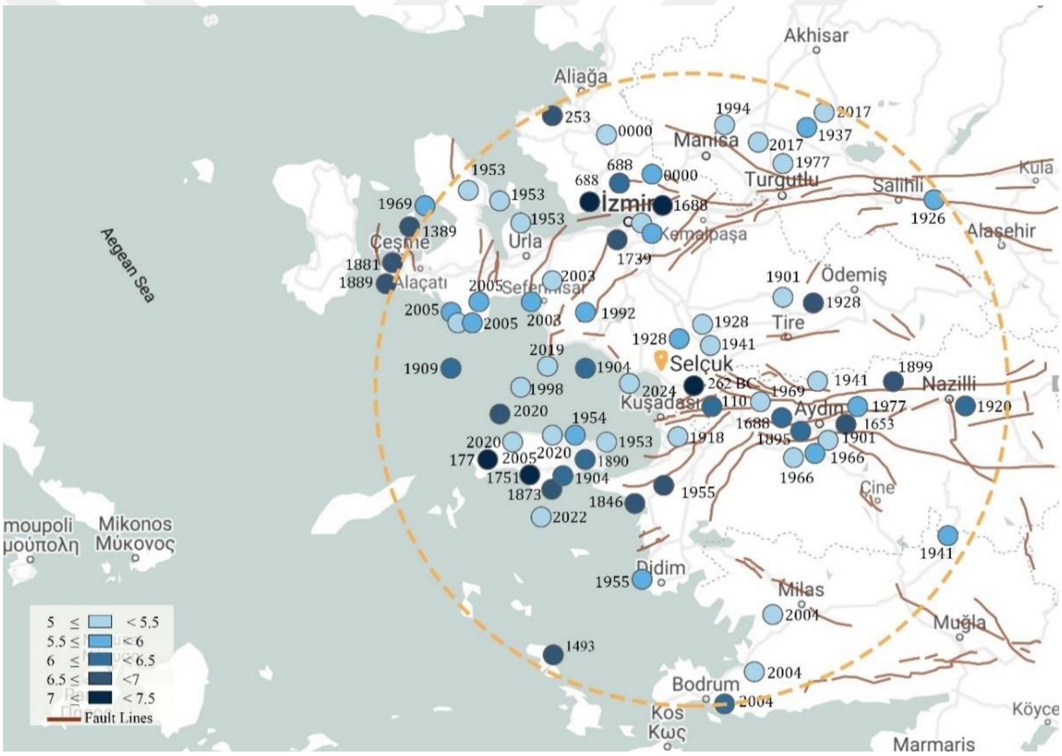


Figure 3.3. Earthquakes in Selçuk and its surroundings (Revised from (KOERI 2017) and (KOERI 2024) – 01.04.2024) (Circular scanning feature of KOERI 2024 with Selçuk centered 100 km Radius)

In 1862, with the construction of the Aydın-İzmir Railway in Ayasuluk, agriculture, and trade facilities developed rapidly due to the accessibility of transportation, so the population of the city increased. Following the construction of the railway, the settlement area shifted towards the west part of the railway station (Pekak and Aydın 1998).

After the proclamation of the Republic, Selçuk became a district of Izmir. The municipality, administrative, and educational buildings were built close to the station. As the population increased, the housing stock developed towards the east and south of the city (Büyükkolancı, Erdemgil, and Heuber 1997). Thus, the modern urban texture started to form.

In addition, the active fault lines of the region have caused many destructive earthquakes in the city and these significant seismic activities have been effective in changing the urban fabric throughout history (Figure 3.3) (KOERI 2024; 2017). Probably due to a significant amount of destructive seismic activity, there is considerable structural damage to buildings. Especially, major earthquakes in 262 BC, during the 6th century, and 1360 caused significant damage and collapse of buildings in Selçuk (Wood 2014; Ladstätter 2011).

3.3. Architectural Features of Studied Bath Buildings

The Principalities period represents the transition between the Seljuk and Ottoman Empires (Kılıç 1998). The architecture of this period in Western Anatolia was influenced by the architectural traditions of the Archaic, Hellenistic, Roman, and Byzantine periods (Bellibaş 2015).

During the Aydınoğulları Principality, significant civil and religious buildings were constructed. Many buildings such as mosques (İsa Bey Mosque, Karakol Yanı Mosque, İshakbey Mosque, Kılıçarslan Mosque, Akıncılar Mosque, Alparslan Mosque, Kale Mosque), masjids, zaviye, madrasahs, tombs (Şahabettin Dede Tomb, Yahşi Bey Tomb, Tomb at Hospital), and baths (Saadet Hatun Bath, İsa Bey Bath, Kale Altı Bath, Yahşi Bey Bath) were built in Selçuk (Figure 3.4.) by architects and craftsmen using local materials and labor (Öktem 2002; Kalfazade 2013; Büyükkolancı 2023; Kuban 2009; Telci 2010; Ladstaetter et al. 2015).

However, some of these structures have not survived, which may be due to the earthquakes that have occurred throughout history in Selçuk (Figure 3.3.). Most of the structures are located within agricultural lands in poor condition. It is conceivable that the region which is located between Ayasuluk Hill and the Temple of Artemis was not prioritized during the development of the modern city, likely due to its marshy land or agricultural use; for this reason, these assets may have been partially conserved (Wood 2014).

During Aydınoğulları Principality, small-scale structures such as baths, tombs, and mosques were typically built using rubble stone, pitch-faced stone, and alternate brick/stone masonry methods in Selçuk (Kolay 1999). The alternate brick/stone masonry technique originated in Roman architecture and was also used during the Byzantine and Principalities periods in western Anatolia (Ahunbay 2006; Kolay 1999; Kuban 1982). The repetition of bricks in horizontal rows between the stones increases the strength of the wall, giving it a tie beam characteristic (Ahunbay 2006). In Selçuk, another technique used during this period was known as *"kasetleme,"* which involved framing the stones with horizontal and vertical bricks within the masonry walls (Kolay 1999).

Domes were the most common type of roof used in the architecture of the Aydınoğulları Principality in Selçuk (Kolay 1999). However, some buildings also were used vaults and wooden gable roofs (Isa Bey Mosque). In Selçuk, excavations and investigations have shown that the upper covers of buildings from the Aydınoğulları Principality period were not typically covered with tiles; instead, they were likely covered with plaster or mortar (Kolay 1999). Bath domes were covered with Horasan plaster, and they often featured openings for lighting *(Fil Gözü)* (Yavuz 2006).

As a result of the excavations and investigations related to the buildings belonging to the Aydınoğulları Principality period in Selçuk, the remains of 10 baths dating back to this period were found (Figure 3.4) (Bellibaş, Ladstätter, and Kürüm 2013; Telci 2010; Ladstaetter et al. 2015). However, some of these bath structures have not survived to the present day. Only the Saadet Hatun Bath has been restored and is currently used as a museum.

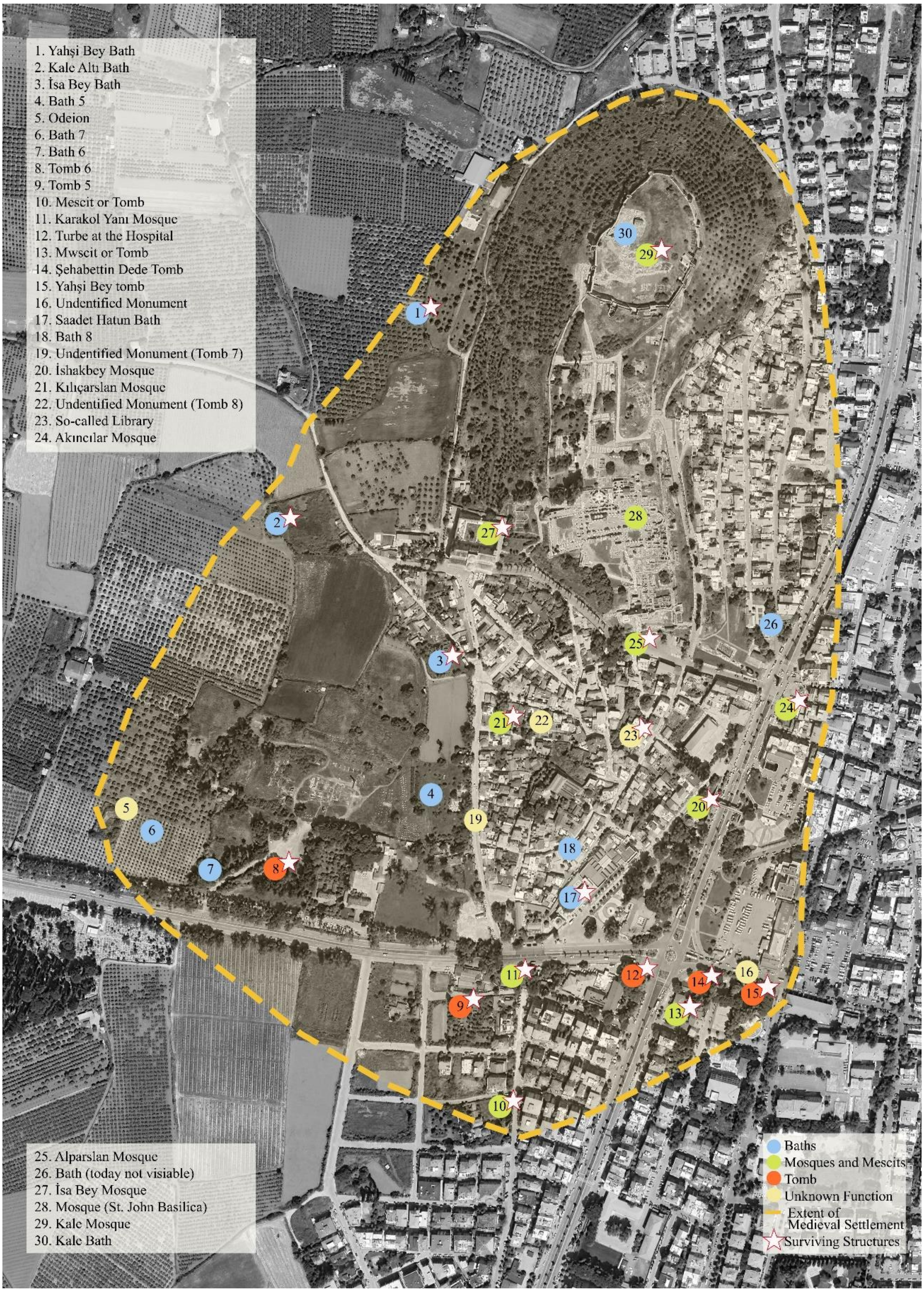


Figure 3.4. Remains of the Principalities Period (Ladstaetter et al. 2015)
(Revised from Google Earth (10.11.2023))

Publications and official inventories that examine the principality period used different definitions for the names of the structures ((S. K. Ertuğrul 1995; Daş 1997; Telci 2010; Özeren and Büyükkolancı 1977a; 1977b; 1977c; Ladstaetter et al. 2015). İsa Bey Bath was named Bath C and Bath II. Kale Dibi Bath. Kale Altı Bath was named Bath B, Bath III, Anonim Bath, and Yahşi Bey Bath was named Bath A, Bath IV, and Garden-Camping Bath.

Isa Bey and Kale Altı baths are in the Atatürk neighborhood, and Yahşi Bey Bath is in the Isa Bey neighborhood. Isa Bey Bath is located at 1055 Street, 343 Block, 1 Lot; Kale Altı Bath is located at 2045 Street, 322 Block, 3 Lot; and Yahşi Bey Bath is located at 2040 Street, 246 Block, 10 Lot. Baths are located west of the Isa Bey Mosque, Ayasuluk Hill, and St. Jean Basilica, and northeast of the Artemis Temple (Figure 3.3). The baths within the boundaries of the 1st Degree Archaeological Site were first registered on 14.07.1980 with the decision number A-1704 by GEEAYK (Cultural and Natural Heritage Conservation Board).

The Isa Bey Bath, Yahşi Bey Bath, and Kale Altı Bath are owned by the Vakıflar Genel Müdürlügü (General Directorate of Foundations). The General Directorate of Foundations and the Austrian Archaeological Institute carried out the conservation and documentation studies of the Isa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath (Ladstaetter et al. 2015; Öktem 2002). No extensive restoration and conservation work was carried out on the three bath buildings; however, they have survived to the present day with their authentic material characteristics preserved and are currently closed to visitors.

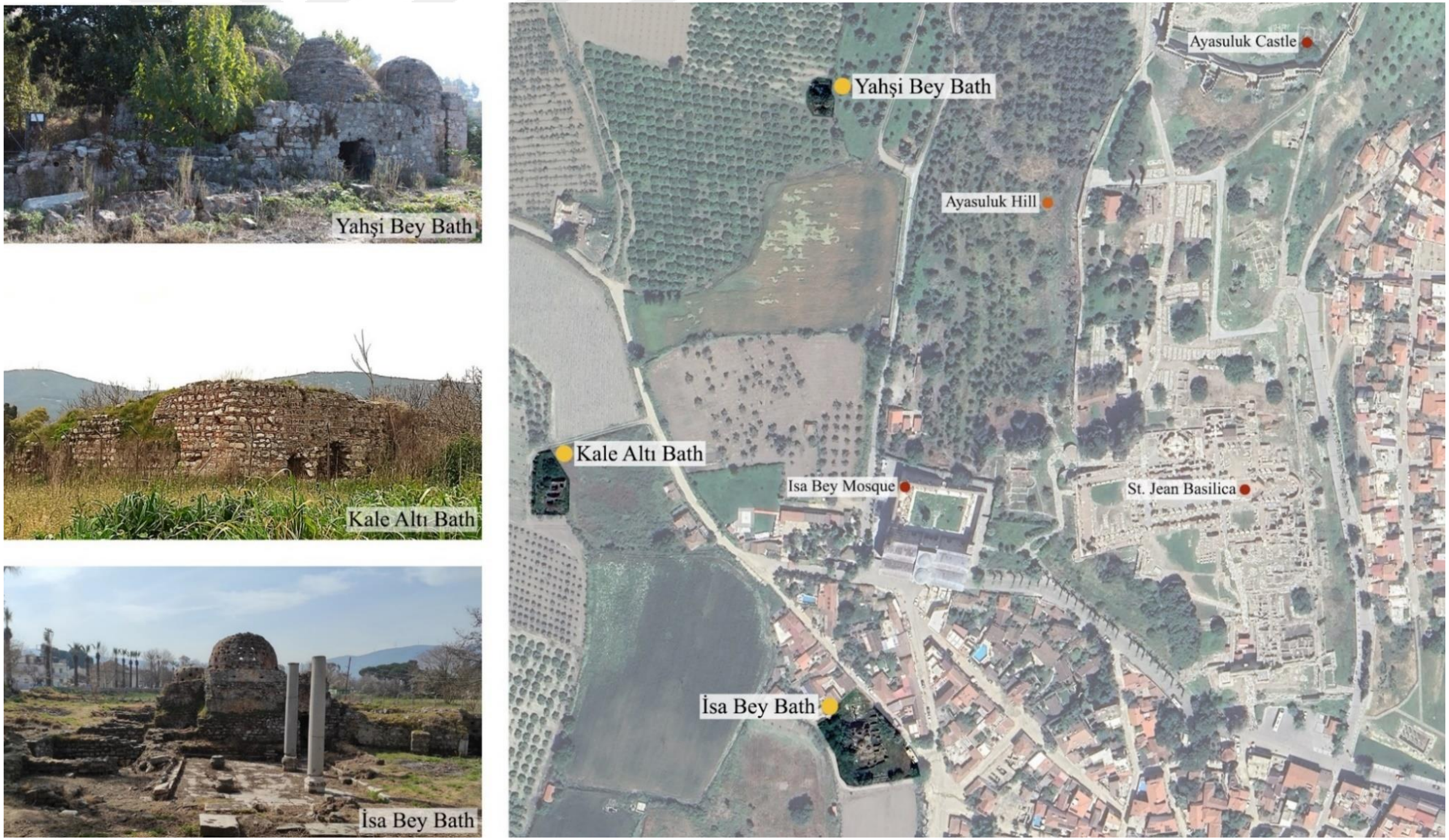


Figure 3.5. Location of Case Study Areas (Revised from Google Earth:10.11.2023)

3.3.1. İsa Bey Bath

Isa Bey Bath was built by Isa Bey in 1364 during the Aydınoğulları Principality period, according to the information obtained from the epigraph (Arıkan 1990; A. Ertuğrul 2009; Daş 1997). It was used for about 60-80 years and served as a cemetery until the mid-15th century (Ladstaetter et al. 2015). The plan layout of İsa bey Bath was more developed than the other principalities period baths in Ayasuluk, such that it is similar to Ottoman bath buildings with its symmetrical plan layout and spaces (Daş 1997).

Although the Isa Bey Bath is a structure of the Principalities period, it is a pioneer and one of the first examples of the classical Ottoman period baths in terms of its high dome and plan layout and the iwans placed around the central dome (Öktem 2002). The bath consists of *Soyunmalık, Aralık, Ilıklık, Sıcaklık*, and *Külhan* sections from north to south (Figure 3.6). The *Ilıklık* consists of shaving and a toilet; the *Sıcaklık* consists of four *halvet* and four iwans (Öktem 2002; Daş 1997; Erat 1997).

The *Soyunmalık* located in the north completely collapsed probably due to earthquakes. The square-plan *Soyunmalık* in the west for women and the pointed vault *Soyunmalık* space for men in the northwest are thought to have been constructed after the northern *Soyunmalık* was destroyed (Erat 1997; Öktem 2002). On the east, there are remains of buildings adjacent to the bath, which are thought to be shops (Öktem 2002; Daş 1997; Erat 1997).

The *Aralık* and *Ilıklık* are both square and covered with a dome. The domes of the *Aralık* and *Ilıklık* are half-collapsed. The domes of the bath have numerous *"fil gözü"* openings for lighting. On the eastern side of the *Ilıklık*, there is a square shaving room and toilet (Erat 1997). The entrance of the *Sıcaklık* is located on the southern side of *Ilıklık*.

The *Sıcaklık* has four iwans and four *halvets* and is laid out in a central plan schema (Figure 3.6.). The central space and the *halvets* are covered with a dome, while the iwans are covered with a barrel vault. Spolia marble floor tiles of *Sıcaklık* were removed during excavations and investigations (Erat 1997). The *Külhan*, located on the southern side of the bath, is covered with a barrel vault.

Figure 3.6. Plan of İsa Bey Bath (Erat 1997) North view (a), Southeast view (b), South view (c), Southwest view (d)

Figure 3. 7. Photos from İsa bey bath; General view from *Sıcaklık* (a), *Halvet*, (b), The dome of the *Ilıklık* (c), General view from *Ilıklık* (d)

The walls of the bath were constructed using rubble stone, pitch-faced stone, brick, and spolia materials in the masonry system. Bricks were used to surround the stones, and brick rows were used as beams in the masonry. The domes were built with bricks and covered with Horasan plaster on the exterior. Bricks were also used in some of the pillars of the hypocaust. Spolia was used in the floor coverings of all spaces, the columns of the *Soyunmalık*, the exterior walls, and even the pillars of the hypocaust (Erat 1997; Öktem 2002). As in other bath buildings, there was a clear boundary between the lower and upper levels of plaster in İsa Bey Bath. The lower-level plaster consisted of three layers; one Horasan plaster, and two layers of grayish plaster layers (plaster with natural stone aggregate). The upper-level plaster consisted of three layers of lime plaster (Table 3.1.). Previous research has revealed that more than one layer of plaster was applied in the construction of the baths on both the lower and upper levels (Table 3.1) (Gürhan 2018; Uğurlu 2005; Budak 2005; Böke, Akkurt, and Ipekoğlu 2004).

In the Isa Bey Bath, it was observed that the walls, domes, and vaults partially or completely collapsed probably due to a lack of maintenance, seismic forces, or vandalism. Structural cracks were identified at multiple points. The bath was surrounded by vegetation (Figure 3.8(a)). The discoloration of plasters, stones, and bricks and the growth of microorganisms (Figure 3.8 (c) – (d)) were caused probably due to weather conditions, rainwater penetration, and rising damp. The loss of integrity of plaster and mortars due to loss and abrasion caused joint discharge on some of the masonry walls (Figure 3.8(b)). The failure of the structure and material deterioration was probably caused by a lack of regular maintenance and repair, seismic forces, or vandalism (Figure 3.8.).

Figure 3. 8. Structure failures and material deterioration (probably due to weather conditions, rainwater penetration, rising damp, lack of maintenance, seismic forces, or vandalism): Plant growth (a), Joint discharge (b), Discoloration, (c), Microbiological colonization (d)

Table 3.1. Plaster levels and number of plaster layers in the studied bath buildings and previous studies

Location - Referances		Period of Samples	Sample Types	Level		Layer Number	
				Lower	Upper	Lower	Upper
İsa Bey Bath - Selçuk, İzmir		14^{th} century	Horasan Plaster	X	-	1	-
			Lime Plaster	-	X	-	3
			Nat. S. Agg. Plaster	X	-	2	-
Kale Altı Bath - Selçuk, İzmir		14^{th} - 15^{th} century	Horasan Plaster	X	-	1	-
			Lime Plaster	-	X	-	1
			Nat. S. Agg. Plaster	X	X	1	1
Yahşi Bey Bath - Selçuk, İzmir		15^{th} century	Horasan Plaster	X	-	1	-
			Lime Plaster	-	X	-	1
			Nat. S. Agg. Plaster	X	X	1	2
Çukur Bath - Manisa (Budak 2005)		14^{th} century	Horasan Plaster	-	X	-	2
			Lime Plaster		X		1
Böke, Akkurt, and Ipekoğlu 2004	Ördekli Bath - Bursa	14^{th} century	Horasan Plaster				
	Beylerbeyi Bath - Edirne	15^{th} century	Horasan Plaster				
	Saray Bath - Edirne		Horasan Plaster				
Uğurlu 2005	Düzce Bath - Urla, İzmir	15^{th} century	Horasan Plaster	X	X	3	2
			Lime Plaster	-	X	-	1
	Herekzade Bath - Urla, İzmir		Horasan Plaster	X	X	1	1
			Lime Plaster	-	-	-	-
	Kamanlı Bath - Urla, İzmir		Horasan Plaster	X	X	3	1
			Lime Plaster	-	X	-	1
Eski Bath - Efeler, Aydın (Gürhan 2018)		15^{th} - 16^{th} century	Horasan Plaster	X	-	2	-
			Lime Plaster	-	X	-	1

3.3.2. Kale Altı Bath

The construction date of the Kale Altı Bath is unknown due to the absence of an epigraph. However, historical research and architectural investigations suggest that the structure was built in the 15th century during the Aydinoğulları Principality (Özeren and Büyükkolancı 1977b; Öktem 2002; Daş 1997). The Kale Altı Bath has a rectangular, asymmetrical floor plan layout. It consists of *Aralık, Ilıklık, Sıcaklık* and *Külhan* sections from North to South. The *Aralık* is in the north of the eastern I of the bath. The square planed Aralık was covered by a dome, and the transition to the dome was provided by Turkish triangles. In the building, there was no trace of *Soyunmalık* space (Figure 3.9.) (Daş 1997; Öktem 2002).

The *Ilıklık* space is located west of the *Aralık.* It has a square plan and is covered with a dome. It consists of a shaving room and toilet. The shaving room and toilets are both square plan layouts and covered with a dome, and the transition to the dome was provided by muqarnas trompes (Öktem 2002).

The entrance of the *Sıcaklık* is located on *Ilıklık's* southern side. The *Sıcaklık* has two iwans and two *halvets* and is laid out in a transverse plan shema. The central square space and the *halvets* are covered with a dome, while the iwans are covered with a semi-dome. The transition to the dome was provided by Turkish triangles with muqarnas in the *Sıcaklık* and west *Halvet*. The dome transition of the east *halvet* was provided by pendentive (Öktem 2002; Daş 1997). To the south of the East *Halvet* room, there is a window opening to the cold-water reservoir. The cold-water reservoir, which was added later to the bath, is located to the west of the bath and it was supported by triangular buttresses (Özeren and Büyükkolancı 1977b; Daş 1997; Öktem 2002).

The *Külhan* located in the southern part of the building, has a rectangular plan, and is covered by a barrel vault. The south I of the *Külhan* was supported by triangular buttresses (Özeren and Büyükkolancı 1977b). The northwest of the bath contains the remains of a structure that was added later (Özeren and Büyükkolancı 1977b; Daş 1997).

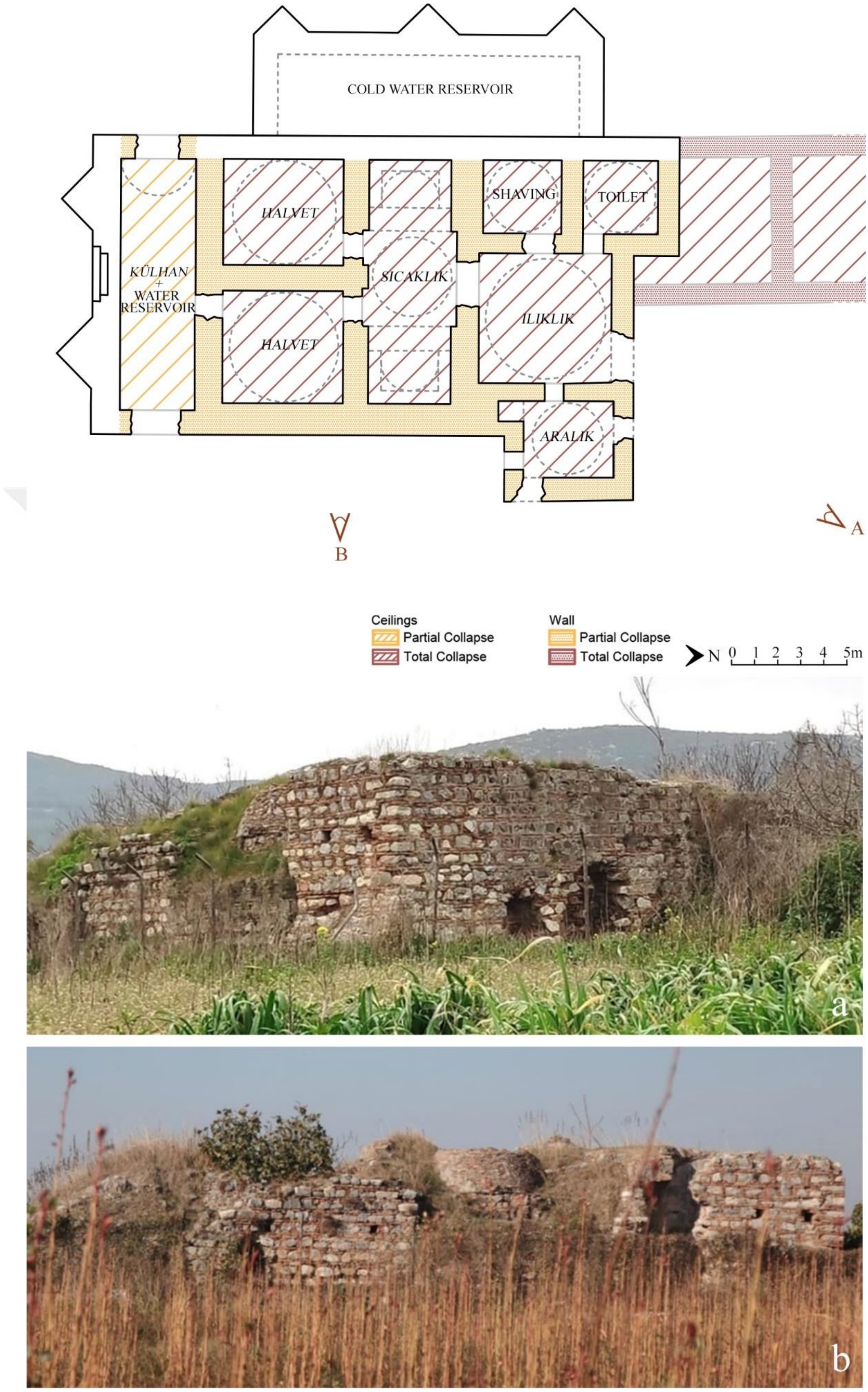


Figure 3. 9. Plan of Kale Altı Bath (Daş 1997), North-East view (a), East view (b)

Figure 3. 10. Kale Altı Bath interior photos; *Sıcaklık* (a), *Ilıklık* (b), *Külhan* (c), *Aralık* (d)

Figure 3. 11. Structure failures and material deterioration of Kale Altı Bath: Structure failures and material deterioration (probably due to weather conditions, rainwater penetration, rising damp, lack of maintenance, seismic forces, or vandalism): Joint discharge (a), Discoloration (b), Plant growth (c), Microbiological colonization (d)

The bath was constructed using rubble stone, pitch-faced stone, brick, and spolia materials in the masonry system like İsa Bey Bath and Saadet Hanım Bath. The exterior walls of the bath were built with stone-brick alternating masonry wall technique. Bricks were used to surround the stones, and brick rows were used as beams in the masonry. The domes were built with bricks (Daş 1997; Öktem 2002). In Kale Altı Bath there were two levels of plaster: lower, and upper levels of plaster similar to previous studies (Table 3.1). Only the *Külhan* had lower-level plaster and it consisted of two layers; one Horasan Plaster, and one grayish plaster layer (plaster with natural stone aggregate). The upper-level plaster consisted of two layers; one lime plaster, and one grayish plaster layer (Table 3.1.).

In the Kale Altı Bath, the domes of all spaces were completely collapsed. Only the vault of *Külhan* was partially existed. It was observed that some of the walls had structural cracks and some of them were out of plumb (Figure 3.11. (a)). There was plant growth inside and outside the bath (Figure 3.11. (c)). The ground level was raised due to vegetation and building remains and the original ground level could not be determined (Figure 3.10. (a)). The discoloration of plasters, stones, and bricks and the growth of microorganisms were caused probably due to water penetration, weather conditions, or rising damp (Figure 3.11. (b)). The absence of a roof was caused abrasion and the loss of materials. The loss of integrity of the plasters and mortars has caused joint discharge in some of the masonry walls (Figure 3.11. (a)). The deterioration of the structure and material was probably due to a consequence of a lack of regular maintenance and repair, seismic forces, or vandalism (Figure 3.11.).

3.3.3 Yahşi Bey Bath

Yahşi Bey Bath was built in the second half of the 15th century during the Aydınoğulları Principality, according to historical research and architectural investigation (Özeren and Büyükkolancı 1977c). Due to its asymmetrical plan scheme and irregular masonry technique, it is thought to have been built during the period when the Aydınoğulları Principality was losing economic power (Öktem 2002).

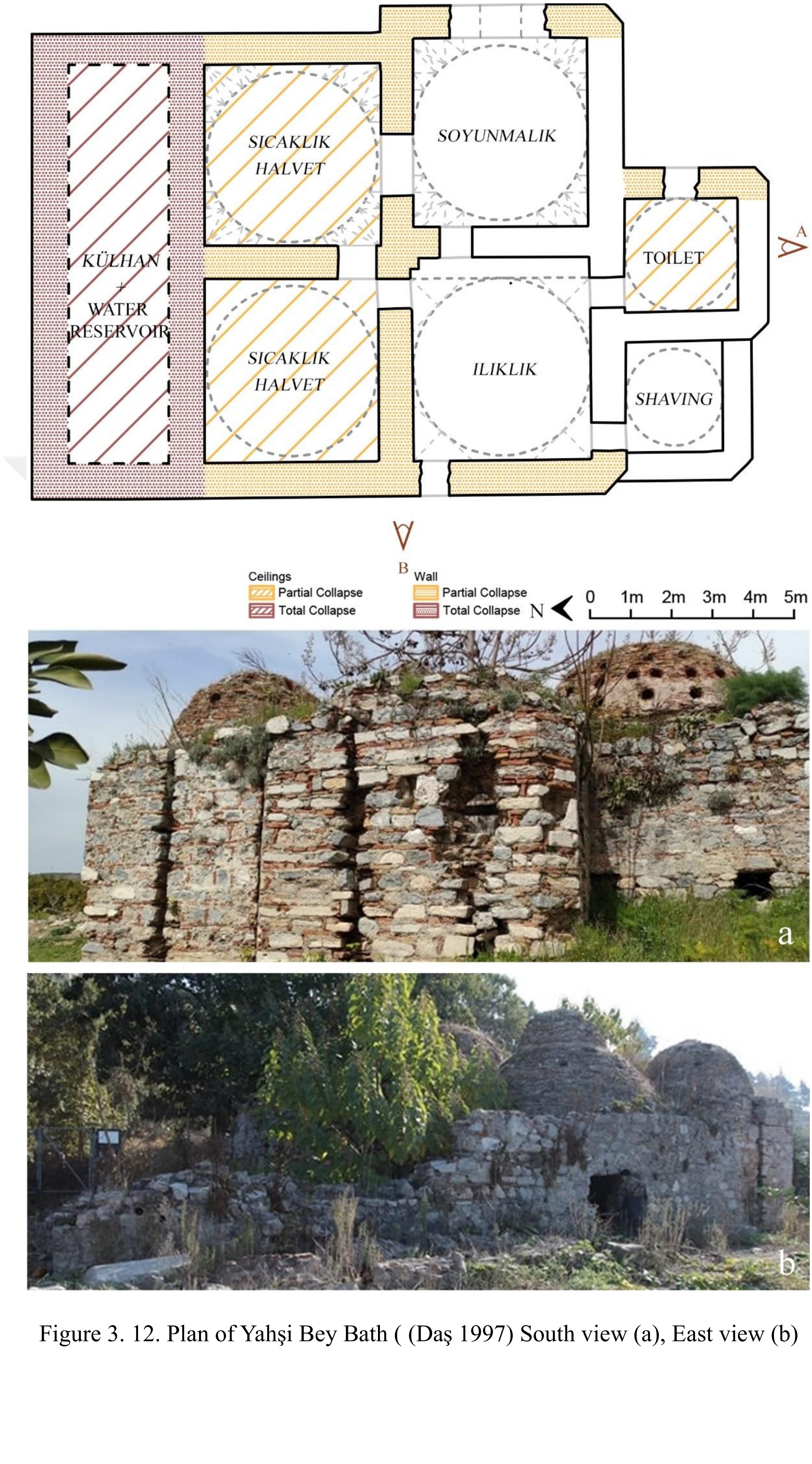


Figure 3. 12. Plan of Yahşi Bey Bath ((Daş 1997) South view (a), East view (b)

The Bath is located on the north-south axis, has an asymmetrical plan, and consists of *Soyunmalık, Ilıklık, Sıcakık* and *Külhan* (Öktem 2002; Daş 1997) (Figure 3.12). The *Soyunmalık* is in the southeast of the building, has a square plan, and is covered with a dome supported by Turkish triangles (Figure 3.13.). The north wall of the *Soyunmalık* was partly collapsed.

The *Ilıklık* is located eastern side of the *Soyunmalık.* It has a square plan with a dome and dome supported by trumpes. To the south of the *Ilıklık,* there is a square planned and domed shaving room and toilet (Figure 3.12.). According to Ertan Taş and Gül Öktem, the shaving and toilet rooms were added to the building as an early addition to the information from the building traces (Daş 1997; Öktem 2002). The *Sıcaklık* consists of square-plan Halvets covered with domes, and the dome transitions are constructed using Turkish triangles (Daş 1997) (Figure 3.14.). The dome of the western *Halvet* has completely collapsed. The *Külhan* is located on the north I of the bath. Its upper cover and walls have collapsed.

The bath was constructed using rubble stone, pitch-faced stone, brick, and spolia materials in the masonry system İsa Bey Bath, Saadet Hatun Bath, and Kale Altı Bath. The exterior walls of the bath were built with stone-brick alternating masonry wall technique (Öktem 2002; Özeren and Büyükkolancı 1977c; Daş 1997). The domes were built with bricks. In Yahşi Bey Bath there was a clear boundary between the lower and upper levels of plaster similar to previous studies (Böke, Akkurt, and Ipekoğlu 2004; Uğurlu 2005; Gürhan 2018). The lower-level plaster consisted of two layers; one Horasan plaster, and one grayish plaster layer (plaster with natural stone aggregate). The upper-level plaster consisted of three layers, one lime plaster, and two grayish plaster layers (Table 3.1.). In the Yahşi Bey Bath, it was observed that the walls, domes, and vaults partially or completely collapsed probably due to a lack of maintenance, seismic forces, or vandalism. Structural cracks were identified at multiple points. There was plant growth inside and outside the bath (Figure 3.14. (a)). It was observed that in some spaces of the bath building, the floor covering has been entirely collapsed. The discoloration of plasters, stones, and bricks and the growth of microorganisms were probably caused by rising damp (Figure 3.14. (b)). The loss of integrity of plaster and mortars due to loss and abrasion caused joint discharge on some of the masonry walls. The failure of the structure and material deterioration was probably caused by a lack of regular maintenance and repair of seismic forces or vandalism.

Figure 3. 13. Photos from Yahşi Bey Bath: *Soyunmalık* – Discoloration (a), *Ilıklık* – Vandalism (b)

Figure 3. 14. Photos from Yahşi Bey Bath: East *Halvet* – Plant growth (a), Weast *Halvet* – Microbiological colonization (b)

CHAPTER 4

EXPERIMENTAL STUDIES

In this chapter, sampling procedures, sample definitions, and experimental methods used in the characterization of the mortars and plasters were taken from the İsa Bey, Kale Altı, and Yahşi Bey baths were described. Experimental methods used for the determination of the basic physical properties, raw material compositions, hydraulic properties, mineralogical and chemical compositions, microstructural properties, and pozzolanic activities were explained. The stated properties of the samples were investigated by standard test methods, X-ray diffraction (XRD), thermogravimetric analysis (TGA), and scanning electron microscopy coupled with energy-dispersive X-ray spectrometry. (SEM).

4.1. Sampling

Samples were collected in March 2022, from three baths dated to the 14th – 15th century, the İsa Bey Bath, the Kale Altı Bath, and the Yahşi Bey Bath in Selçuk. Sampling locations were documented by photographs and sketches. Each sample was labeled and stored in polythene bags.

Samples from the interior walls of the baths were taken from relatively sound areas without damaging the structure. Samples were collected from *soyunmalık* (Disrobing room), *ılıklık* (Warm area), *sıcaklık* (Hot area), *halvet*, and *külhan* (Furnace area) spaces of the baths. The interiors of the baths had several levels of plaster, distinguishable by their different colors on the upper and lower wall surfaces, they were clearly separated from each other. The plaster on the lower level of the wall surfaces was applied up to one meter higher than the floor surfaces. Plaster samples were taken from both the lower and upper levels with all layers which is plaster with natural stone aggregate, horasan plaster and lime plaster.

In total three mortar 15 plaster samples were collected from all baths. The samples were labeled with the names of the baths and spaces from which they were collected. Also, the plaster samples were labeled with their levels and layers (Figure 4.1.). The first letter indicates the bath's name (I: *İsa Bey* Bath, K: *Kale Altı* Bath, Y: *Yahşi Bey* Bath). The second letter represents the space where samples were collected (*Soyunmalık*: D, *Sıcaklık*: T, *Halvet*: H, *Külhan*: F). The third letter indicates the sample types (Mortar: M, Lower-Level Plaster: LP, Upper-Level Plaster: UP). Plaster layers were identified by numbers, and the layers were numbered from the outside to the inside.

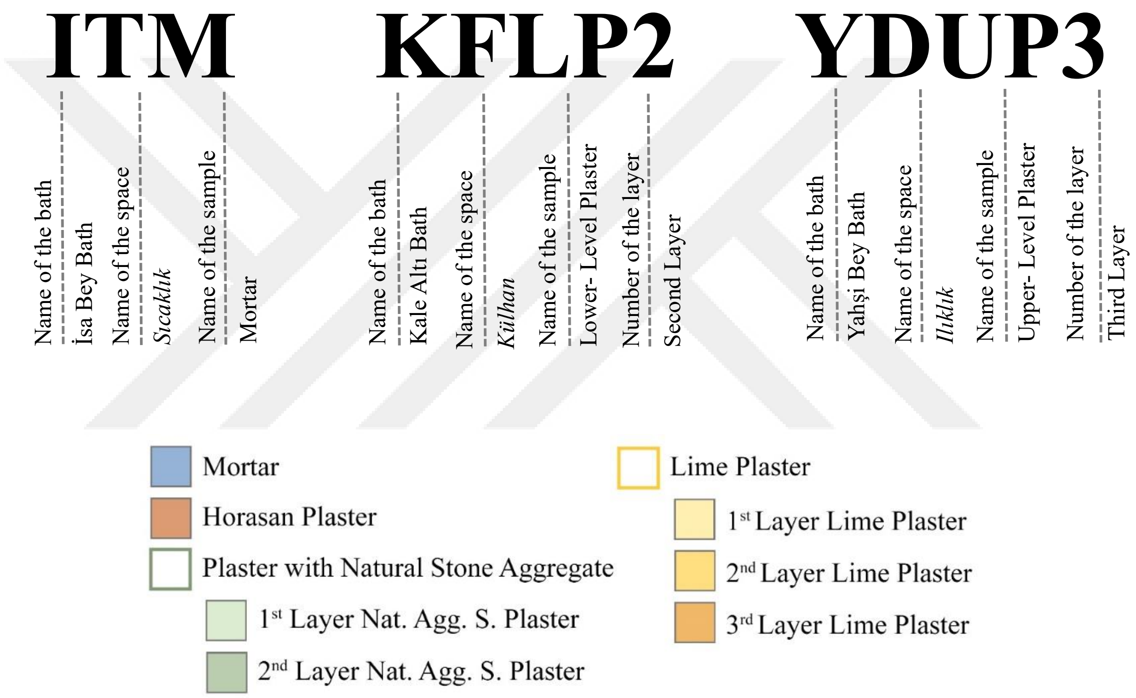
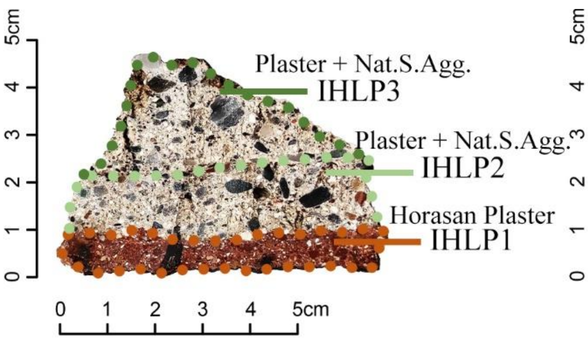
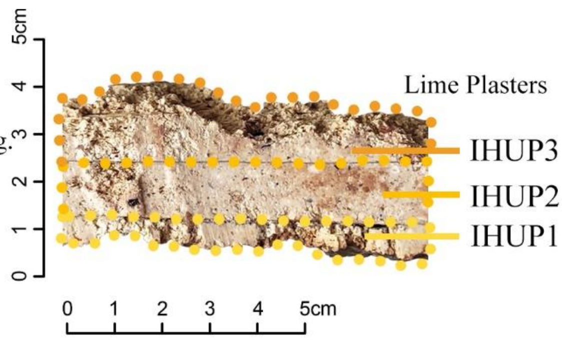


Figure 4.1. Abbreviations used for sample names and color codes of sample types

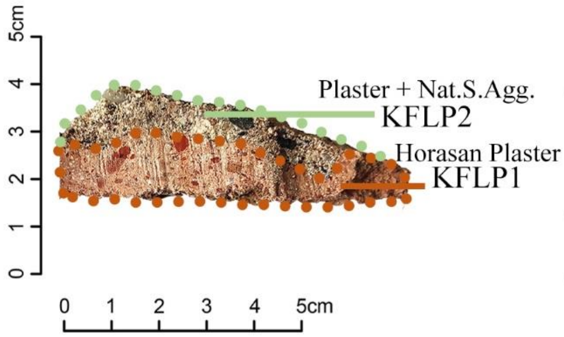
Color codes were chosen to identify each sample type and their layer and these color codes were used in graphs, figures, and tables when evaluating the results of the samples (Figure 4.1). The lower-level plasters typically consisted of two or three plaster with natural stone aggregate layers with a horasan plaster. The upper-level plasters consist of two or three layers of plaster with natural stone aggregate or lime plaster layers. The last layer of the upper level is lime plaster.



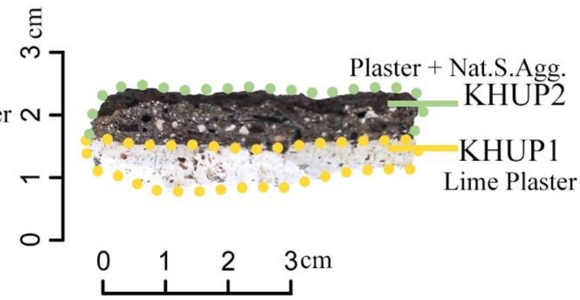
İsa Bey Bath
Lower-Level Plaster



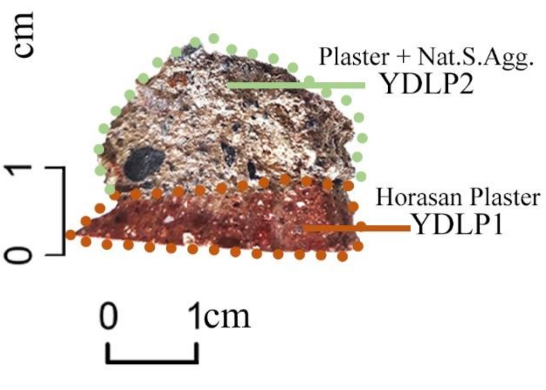
İsa Bey Bath
Upper-Level Plaster



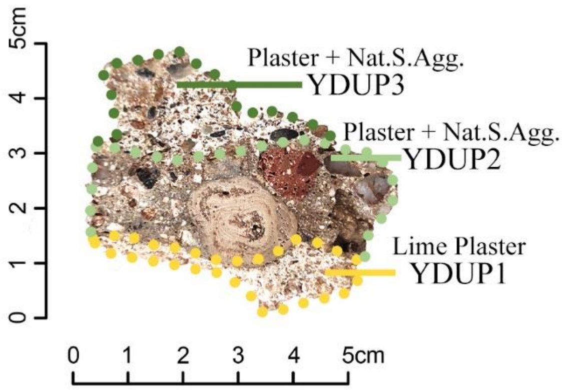
Kale Altı Bath
Lower-Level Plaster



Kale Altı Bath
Upper-Level Plaster



Yahşi Bey Bath
Lower-Level Plaster



Yahşi Bey Bath
Upper-Level Plaster

Figure 4.2. Interfaces between plaster layers from the lower-level and upper-level

In the İsa Bey Bath, seven samples were collected (Figure 4.4). Mortar was taken from the north side of the *Sıcaklık*; the plasters were taken from the *Halvet* (H) located in the southwest. In İsa Bey Bath there was a clear boundary between the lower and upper levels of plaster. The lower-level plaster of the southwest *Halvet* (H) consisted of three layers; one horasan plaster and two plaster with natural stone aggregate layers. The upper-level plaster of the southwest *Halvet* consisted of three layers of lime plaster.

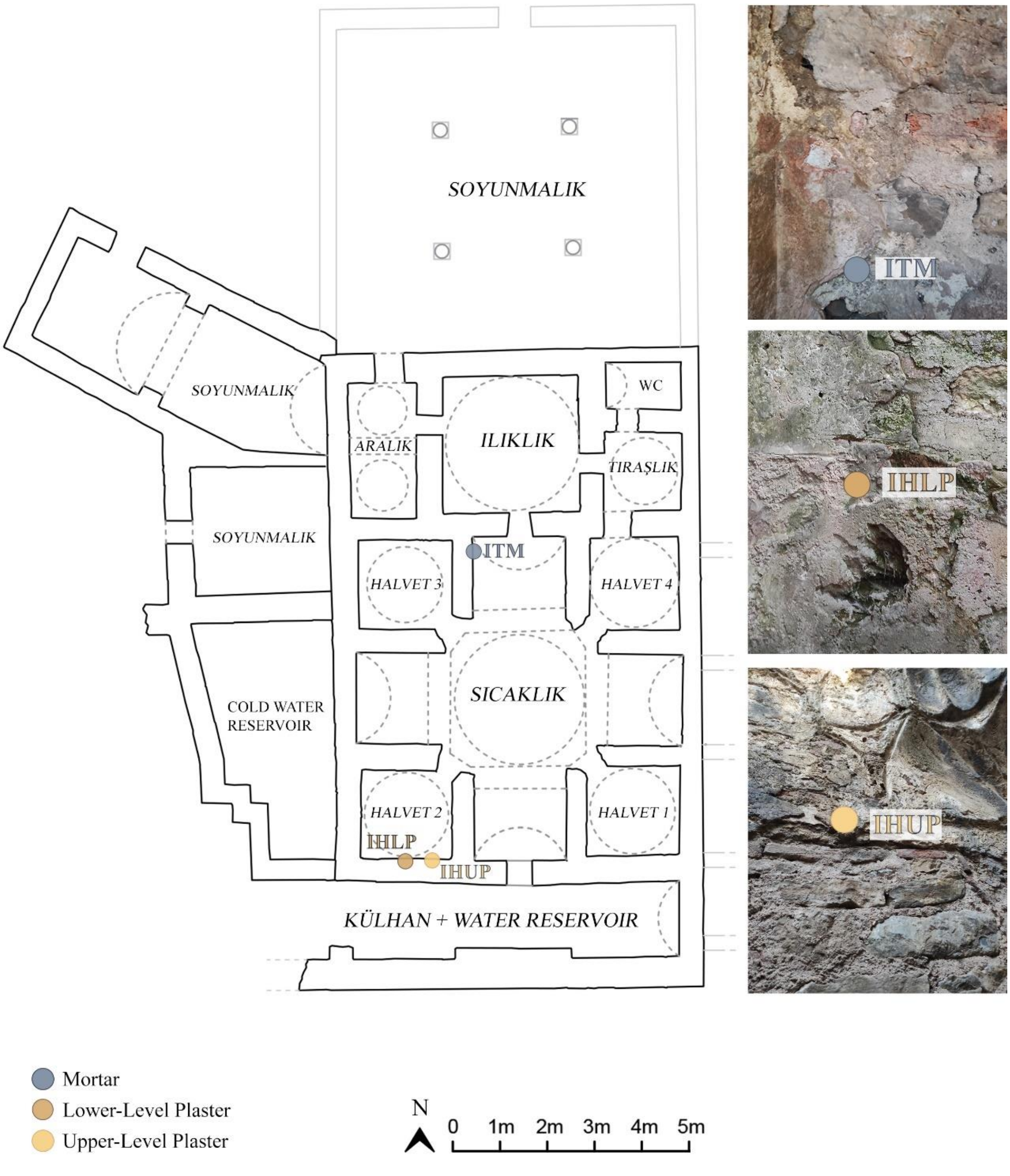
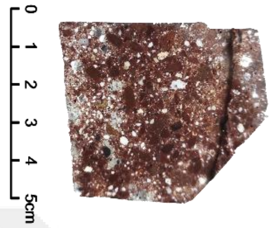
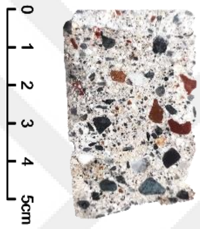
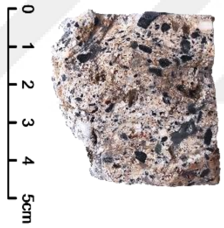
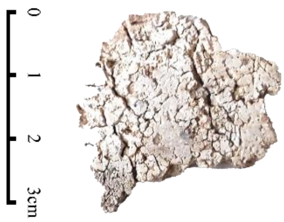


Figure 4. 3. İsa Bey Bath plan and collected samples' photos (Plan Source: Erat 1997)

Table 4.1. Definitions of the samples taken from the İsa Bey Bath

Sample		Definition
Code	**Photo**	
ITM		Mortar taken from the east wall of the *Sıcaklık's* north iwan.
IHLP1		Horasan plaster taken from the lower-level plaster of the southwest *Halvet* (C2).
IHLP2		The second layer of plaster taken from the lower-level plaster of the southwest *Halvet* (C2).
IHLP3		The third layer of plaster taken from the lower-level plaster of the southwest *Halvet* (C2).
IHUP1		Lime plaster taken from the upper-level plaster of the southwest *Halvet* (C2).
IHUP2		The second layer of lime plaster taken from the upper-level plaster of the southwest *Halvet* (C2).
IHUP3		The third layer of lime plaster taken from the upper-level plaster of the southwest *Halvet* (C2).

Five samples were collected from the Kale Altı Bath (Figures 4.4). The lower-level plaster was taken from the *Külhan* located in the south. Only the *Külhan* had lower-level plaster and it consisted of two layers; one Horasan, and plaster with natural stone aggregate. Mortar and upper-level plaster were collected from the southeast *Halvet.* The upper-level plaster consisted of two layers; one lime plaster and one plaster with natural stone aggregate.

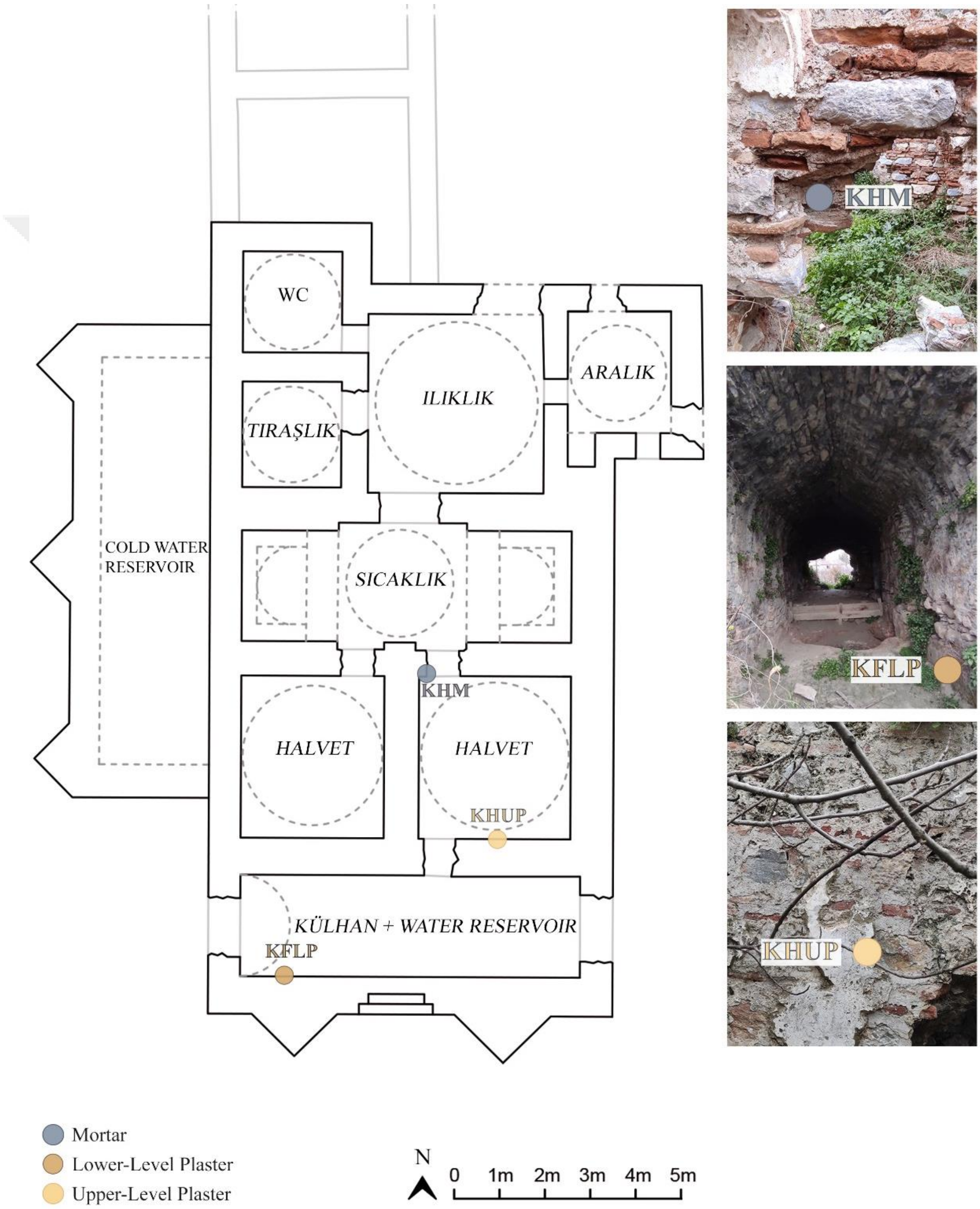
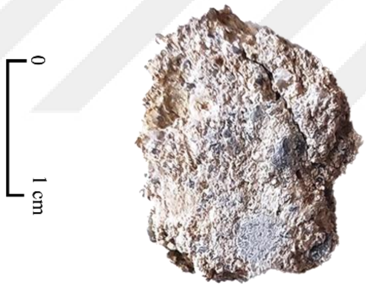
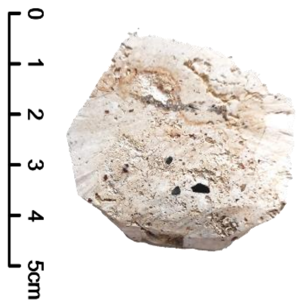
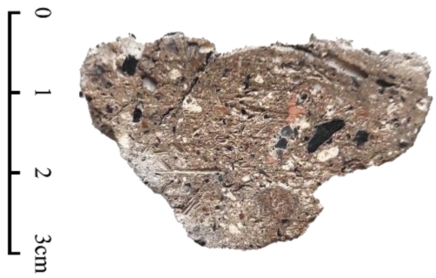


Figure 4.4. Kale Altı Bath Plan and collected sample photos
(Plan Source: Özeren and Büyükkolancı 1977b, 2; Daş 1997, 391; Öktem 2002, 227).

Table 4.2. Definitions of the samples taken from the from the Kale Altı Bath

Sample		Definition
Code	**Photo**	
KHM		Mortar taken from the north wall of the southeast *Halvet*.
KFLP1		Horasan plaster taken from the lower-level plaster of *Külhan's* south wall.
KFLP2		The second layer of plaster taken from the lower-level plaster of *Külhan's* south wall.
KHUP1		Lime plaster taken from the upper-level plaster of the southeast *Halvet's* south wall.
KHUP2		The second layer of plaster taken from the upper-level plaster of the southeast *Halvet's* south wall.

In the Yahşi Bey Bath, six samples were collected (Figure 4.5). Mortar, lower-level plaster, and upper-level plaster were taken from *Soyunmalık*. The lower-level plaster of the *Soyunmalık* consisted of two layers; Horasan plaster and plaster with natural stone aggregate layers. The upper-level plaster consisted of three layers; one lime plaster, and two plaster with natural stone aggregate.

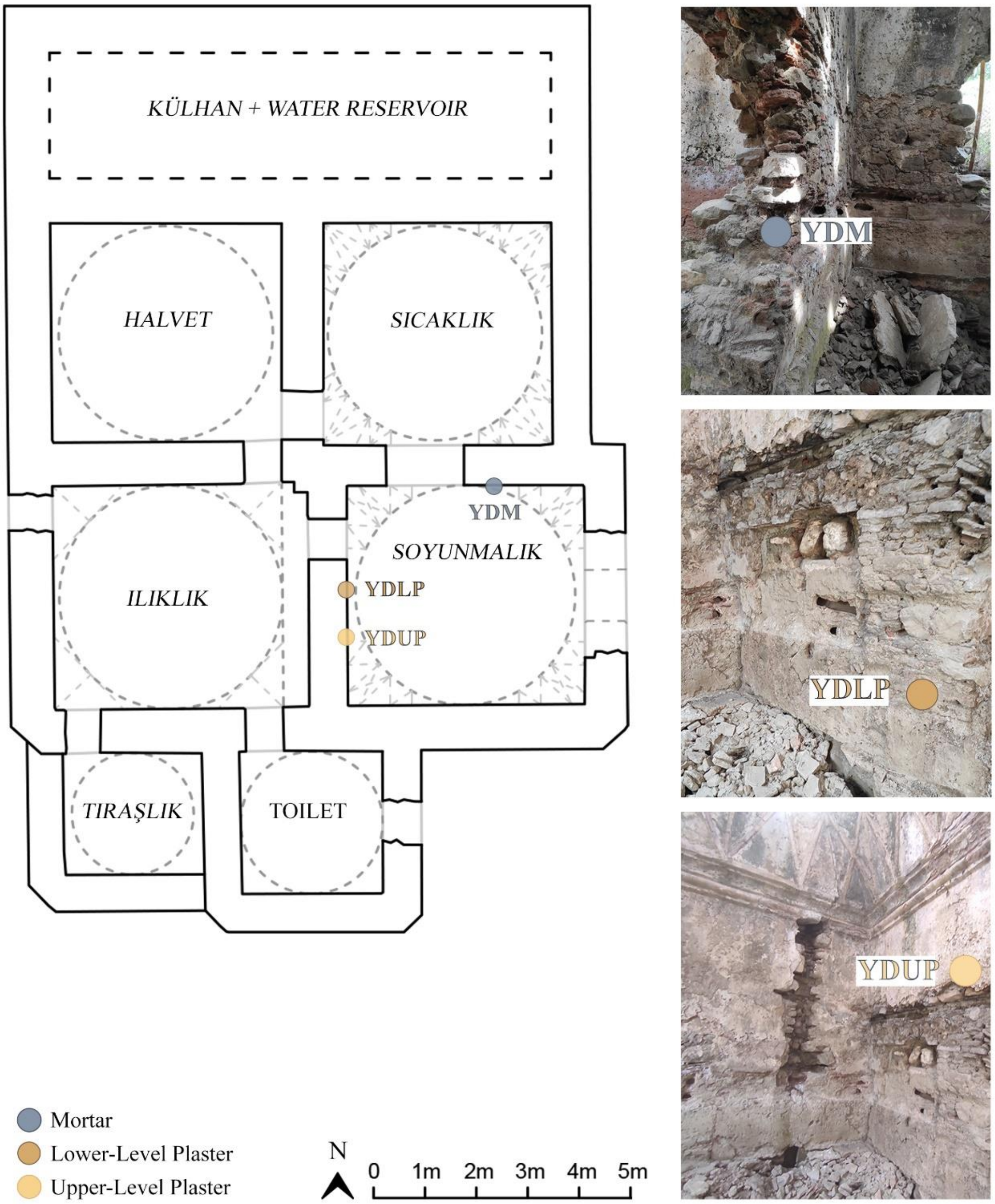
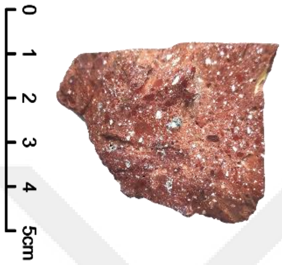
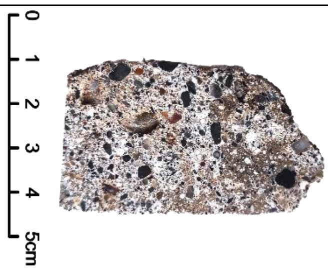


Figure 4.5. Yahşi Bey Bath Plan and collected samples' photos
(Plan Source: Daş 1997; Öktem 2002)

Table 4.3. Definitions of the samples taken from the Yahşi Bey Bath.

Sample		Definition
Code	Photo	
YDM		Mortar, taken from *Soyunmalık's* north wall
YDLP1		Horasan plaster taken from the lower-level plaster of *Soyunmalık*'s west wall.
YDLP2		The second layer of plaster taken from the lower-level plaster of *Soyunmalık's* west wall.
YDUP1		Lime plaster taken from the upper-level plaster of *Soyunmalık's* west wall.
YDUP2		The second layer of plaster taken from the upper-level plaster of *Soyunmalık's* west wall.
YDUP3		The third layer of plaster taken from the upper-level plaster of *Soyunmalık's* west wall.

4.2. Experimental Studies

The following properties of the collected mortars and plasters were examined by experimental studies:

- Basic physical properties:
  - Density
  - Porosity
- Raw material compositions:
  - Lime-aggregate ratio
  - Aggregate particle size distributions.
- Mineralogical and Chemical Compositions
- Pozzolanic Activity
- Thermogravimetric Analyses of Hydraulic Properties
- Microstructural properties

4.2.1. Determination of Basic Physical Properties

The basic physical properties of the samples were defined by their apparent density (g/cm^3) and total porosity (%) values. Standard test methods were used to determine the apparent densities and porosities of plaster and mortar samples (RILEM 1980).

The densities and porosities were determined using two parallel specimens for each sample. First, the samples were dried in a 45ºC oven for at least 24 hours (Figure 4.8.a), then their dry masses (M_{dry}) were weighed using a precision balance (AND HF-3000G) (Figure 4.8.b). Subsequently, immerse the samples in distilled water in a vacuum oven (Lab-Line 3608-6CE Vacuum Oven) at -25 kPa for 24 hours. The water level should be approximately 2 cm above the samples (Figure 4.8.c). The saturated weights (M_{sat}) were then measured, and the Archimedes weights (M_{arch}) were determined through hydrostatic weighing in distilled water using a precision balance (Figure 4.8.d).

Figure 4. 6. a: Oven for drying samples b: Precision balance used to weigh dry and saturated samples c: Samples in vacuum oven d: Archimedes weight measurement

The dry, saturated, and Archimedes weights were used in the subsequent formulas to calculate the bulk densities (D) (4.1) and porosities (P) (4.2) of the plaster and mortar.

$$D\ (g/cm^3) = M_{dry} / (M_{sat} - M_{arch}) \quad (4.1)$$

$$P\ (\%) = [\ (M_{sat} - M_{dry}) / (M_{sat} - M_{arch})]\ x\ 100 \quad (4.2)$$

where:

D : Density (g/cm3)

P : Porosity (%)

M_{atm} : Saturated weight at atmospheric pressure (g)

M_{dry} : Dry weight (g)

M_{sat} : Saturated weight (g)

M_{arch} : Archimedes weight (g)

M_{sat}-M_{dry} : Pore volume (g)

M_{sat}- M_{arch} : Bulk volume (g)

4.2.2. Determination of Raw Material Compositions of Plasters and Mortars

The raw material compositions of mortar and plaster were identified by the lime-aggregate ratio and the particle size distribution of the aggregate. Carbonated lime ($CaCO_3$) was detected in both samples. A dilute hydrochloric acid solution was used to dissolve the $CaCO_3$ so that the lime content and particle size distribution of the samples could be determined.

The samples were dried at 45°C for 24 hours and weighed using a precision balance (M_{sam}). Subsequently, the samples were immersed in beakers filled with a diluted hydrochloric acid solution of 5% concentration until complete dissolution of the carbonated lime occurred (Figure 4.9.a). The insoluble portion was filtered and washed with distilled water to remove chlorine ions (Figure 4.9.b) and dried at room temperature for a day before being oven-dried at 45°C (Figure 4.9.c). Finally, the dried insoluble portion was weighed again using the precision balance (M_{agg}). The ratios of acid-soluble and insoluble components were then calculated using the formula below.

$$\text{Insoluble (\%)} = [(Msam - Magg) / (Msam)] \times 100 \qquad (4.3)$$

$$\text{Acid Soluble (\%)} = 100 - \text{Insoluble (\%)} \qquad (4.4)$$

where:

M_{sam} : Dry weight of the sample (g)

M_{agg} : Dry weight of the aggregates (g)

The lime ratio of mortars and plasters is calculated based on the lime ($Ca(OH)_2$) used in their preparation. The acid-soluble ratio is calculated using dissolved carbonated lime ($CaCO_3$), but the exact ratio was determined by considering the chemical formula for carbonation, which is given below.

$$\underbrace{Ca(OH)_2}_{74g} + \underbrace{CO_2}_{44g} \rightarrow \underbrace{CaCO_3 + H_2O}_{100g \text{ (Molecular weights)}} \qquad (4.5)$$

$$\text{Aggregate \%} = (100 \times \text{Insoluble}) / [((\text{Acid Soluble \%} \times M.W._{Ca(OH)2}) / M.W._{CaCO3}) + \text{Insoluble \%}] \qquad (4.6)$$

$$\text{Lime \%} = 100 - \text{Aggregate \%} \qquad (4.7)$$

where:

$M.W._{CaCO3}$: Molecular weight of $CaCO_3$ which is 100.

$M.W._{Ca(OH)2}$: Molecular weight of $Ca(OH)_2$ which is 74.

Sieve analysis was carried out to determine to ascertain the particle size distributions of the purified aggregates. The dried residue aggregates were sieved using a Retsch AS200 analytical sieve shaker with mesh sizes of 1180 µm, 500 µm, 250 µm, 125 µm, and 53 µm. The weight of particles retained on each sieve was measured and the corresponding percentages were calculated to determine the particle size distribution.

4.2.3. Determination of Mineralogical and Chemical Compositions

The mineralogical compositions of binders, white lumps, and aggregates with a particle size of less than 53 µm were determined through X-ray diffraction (XRD) analysis. The XRD analyses were conducted using a Philips X-Pert Pro X-Ray Diffractometer, operating at 40 kV and 40 mA, with CuKα radiation and a Ni filter. The range of analysis spanned from 5 to 60°, with a scan speed of 0.08°/s.

The chemical compositions of aggregates, binders, and white lumps were all determined by a Philips XL 30S-FEG Scanning Electron Microscope (SEM) coupled with an Energy Dispersive X-ray Spectroscopy (EDS). The chemical compositions of the samples were determined by SEM-EDS analysis of pellets prepared from fine powder samples with a particle size of less than 53 µm. The pellets were pressed with a pressure of 10 tons/cm^2. Each pellets samples were examined from three distinct areas and obtained via an X-ray detector.

4.2.4. Determination of Pozzolanic Activity of Aggregates

The pozzolanic activity of aggregates with a particle size of less than 53 µm was determined through the measurement of the electrical conductivity of the samples (Luxán, Madruga, and Saavedra 1989; Mccarter and Tran 1996). The electrical conductivity of a saturated solution of calcium hydroxide ($Ca(OH)_2$) was measured using a WTW Multiline P3 conductivity meter. Aggregates with a particle size of less than 53 µm were then added to the solution at a ratio of 1g/40ml. The mixture was stirred for 2 minutes using an IKAMAG RH magnetic stirrer, and the conductivity was then measured again. The pozzolanic activity values were determined by calculating the difference (ΔEC in mS/cm) between the two conductivity measurements. Aggregates were pozzolans if the ΔEC was greater than 1.2 mS/cm (Luxán, Madruga, and Saavedra 1989).

4.2.5. Determination of Hydraulic Properties

Thermogravimetric analysis (TGA) was used to evaluate the hydraulic properties of mortars and plasters. TGA measurements were conducted in the temperature range of 200 – 900 °C using a Shimadzu TGA-21. The weight loss between 200 – 600 °C was attributed to the release of chemically bound water (H_2O) from hydraulic compounds in the mortars and plasters. The weight loss between 600–900 °C was attributed to the release of CO_2 gas resulting from the decomposition of $CaCO_3$. The CO_2/H_2O ratio between 1 -10 revealed the hydraulic character of the mortars and plasters. (Bakolas et al. 1998; Antonia Moropoulou, Bakolas, and Bisbikou 2000).

The hydraulic indices of the binders were determined using also Boynton's formula (4.8). The hydraulic index (H.I.) value greater than 0.1 indicates hydraulic properties in the material (Eckel 2005; Boynton 1980).

$$H.I. = (SiO_2\ \% + Al_2O_3\ \% + Fe_2O_3\ \%) / (CaO\ \% + MgO\ \%) \tag{4.8}$$

4.2.6. Determination of Microstructural Properties

The micro-structural and morphological properties of the mortar and plaster were investigated using by Philips XL 30S FEG Scanning Electron Microscope (SEM) coupled with energy-dispersive X-ray Analysis (EDX).

The microstructure of plasters, mortars, aggregates, and white lumps was investigated by scanning electron detector (SE). The properties of the reaction rims at the interfaces between the binder and pozzolanic aggregates and around the limestone aggregates were investigated in detail using backscattered electron (BSE) detectors at various magnifications (100x, 250x, 500x, 1000x, 2500x, 5000x, 10000x). These analyses were conducted on thin sections and gold-coated broken surfaces of plasters, mortars, and white lumps.

CHAPTER 5

RESULTS AND DISCUSSION

In this chapter, the results of the experimental studies conducted on mortar and plaster samples taken from the Isa Bey, Kale Altı, and Yahşi Bey Baths are given and discussed. The basic physical, chemical, and mineralogical properties of mortars and plasters were determined. In addition, the raw material compositions of the mortars and plasters were analyzed and pozzolan activities of natural and brick aggregates were evaluated.

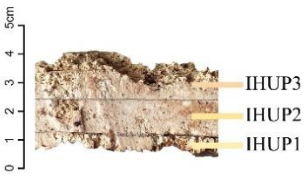
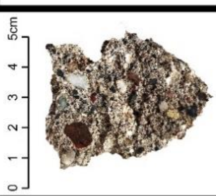
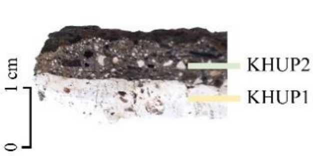
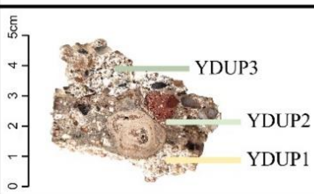
5.1. Basic Physical Properties

The basic physical properties of mortar and plasters were defined by their density (g/cm^3) and total porosity (%) values. The results are shown in Table 5.1 and Figure 5.1 – 5.2.

The total porosity values of mortar samples were found in the range of 17.35 – 31.46% with density values of 1.77 g/cm^3 – 2.02 g/cm^3 (Table 5.1.). The mortar sample taken from Yahşi Bey Bath (YDM) had the lowest porosity value (17.35%) and the highest density value (2.02 g/cm^3). The mortar sample taken from İsa Bey Bath (ITM) had the highest porosity value (31.46%) and the lowest density value (1.77 g/cm^3).

The density and porosity values of the mortar samples in this study are in a similar range to mortars from the Principalities (13^{th} - 14^{th} c.) (Solak 2016; Budak 2005), Ottoman (14^{th} -16^{th}c.) (Stefanidou et al. 2014; Gürhan 2018; Böke et al. 2006; Çizer, Böke, and İpekoğlu 2004), Seljuk (13^{th} c.) (Oğuz 2013; Oğuz, Türker, and Koçkal 2015), and Byzantine (13^{th}–14^{th} c.) (Işık 2022; Stefanidou et al. 2014) periods structures (Table 5.2).

Table 5.1. Density and porosity values of mortars and plasters

		Sample Photos		Sample Types	Density (g/cm^3)	Porosity (%)
İsa Bey Bath			ITM	Mortar	1.77	31.46
İsa Bey Bath	Lower-Level		IHLP1	Horasan Plaster	1.9	22.57
			IHLP2	Plaster with Natural Stone Arggregate	1.65	32.85
			IHLP3	Plaster with Natural Stone Arggregate	1.51	37.89
İsa Bey Bath	Upper-Level		IHUP1	Lime Plaster	1.54	24.73
			IHUP2	Lime Plaster	1.56	31.93
			IHUP3	Lime Plaster	1.25	41.35
Kale Altı Bath			KHM	Mortar	1.81	22.35
Kale Altı Bath	Lower-Level		KFLP1	Horasan Plaster	1.6	36.08
			KFLP2	Plaster with Natural Stone Arggregate	1.31	25.34
Kale Altı Bath	Upper-Level		KHUP1	Lime Plaster	1.83	23.66
			KHUP2	Plaster with Natural Stone Arggregate	1.34	38.69
Yahşi Bey Bath			YDM	Mortar	2.02	17.36
Yahşi Bey Bath	Lower-Level		YDLP1	Horasan Plaster	1.65	21.84
			YDLP2	Plaster with Natural Stone Arggregate	1.47	26.74
Yahşi Bey Bath	Upper-Level		YDUP1	Lime Plaster	1.32	32.42
			YDUP2	Plaster with Natural Stone Arggregate	1.56	35.82
			YDUP3	Plaster with Natural Stone Arggregate	1.77	26.64

Mortar   Horasan Plaster   Natural Stone Aggregated Plaster   Lime Plaster

The density values of the horasan plasters of the baths were found in the range of 1.6 g/cm^3 – 1.9 g/cm^3 with porosity values of 21.84 – 36.08% (Table 5.2).

Density and porosity values of lower-level plasters with natural stone aggregate were between 1.31 – 1.65 g/cm^3 and 25.34 – 37.89%, respectively (Table 5.2). Density and porosity values of the upper-level lime plasters with natural stone aggregates were in the range of 1.34 – 1.77 g/cm^3 and 26.64 – 38.69 % (Table 5.2).

Lime plasters of the baths had density and porosity values ranging between 1.25 – 1.88 g/cm3 and 23.66 – 41.35 % respectively (Table 5.2).

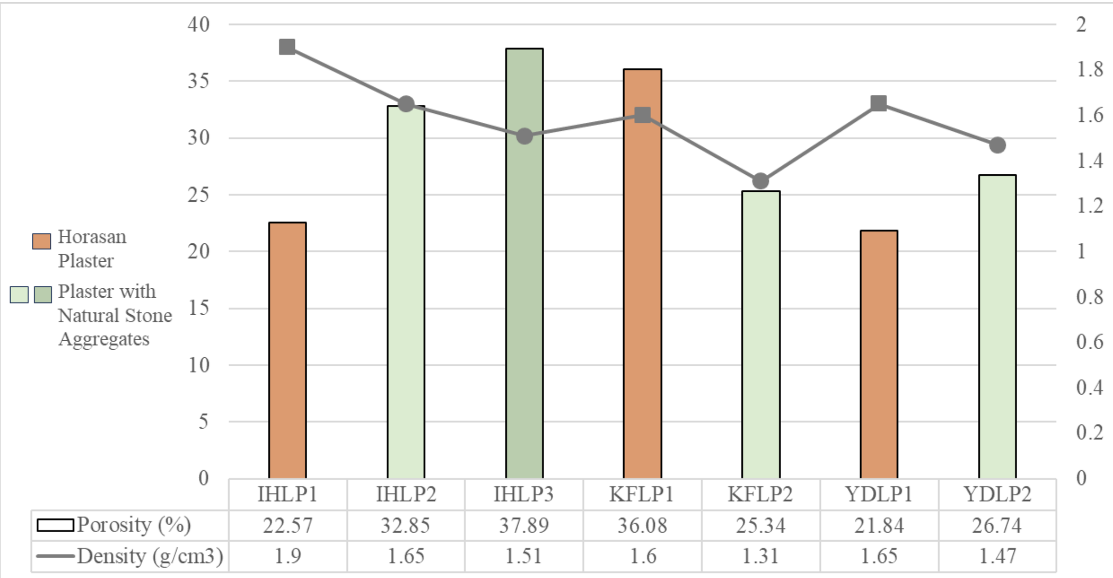


Figure 5. 1. Density and porosity values of lower-level plaster samples

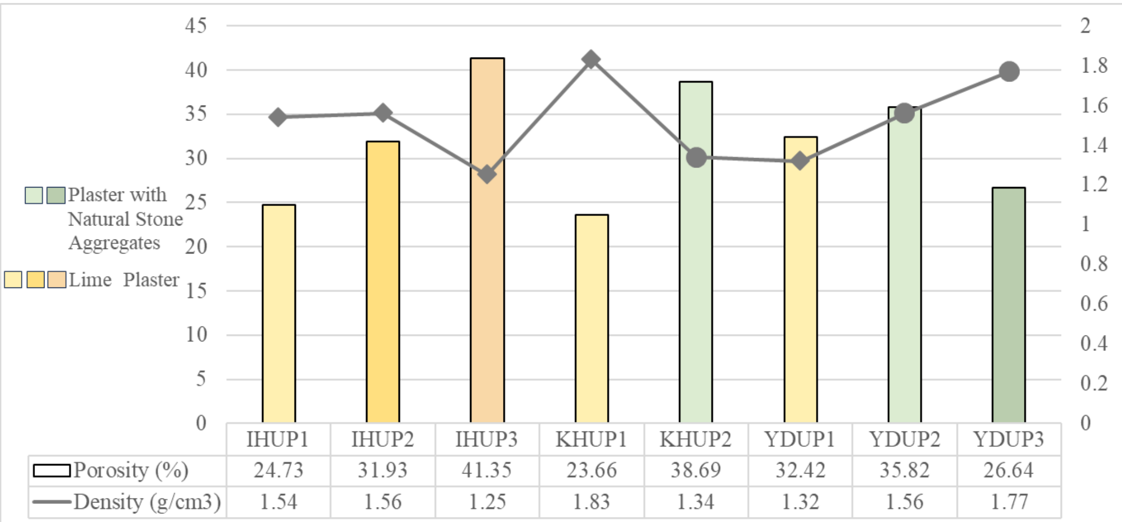


Figure 5. 2. Density and porosity values of upper-level plaster samples

All plaster types and mortars in the studied bath buildings have similar physical properties. The number of plaster layers differs. The average density and porosity values for lower-level and upper-level plaster are in the range of 1.45-1.69 g/cm³ and 24.29 - 31.10% respectively. The number of layers and types of plaster have changed, but the average density and porosity values are close to each other (Figure 5.1 – 5.2).

Horasan plaster layers had higher density and lower porosity values than lime plasters with natural stone aggregates. The low porosity values indicate that the horasan layers provide a waterproof surface that prevents water from reaching the structure of the bath. The use of horasan plaster as a finishing layer was observed in some of the baths examined in previous studies (Uğurlu 2005; Gürhan 2018; Böke, Akkurt, and Ipekoğlu 2004), however, this finishing layer was not found in the studied bath buildings in this study. The density and porosity values of lime plaster and lime plasters with natural stone aggregates were similar to each other, and they were less dense and more porous.

The porosity and apparent density values of the horasan plasters, lime plasters with natural stone aggregates, and lime plasters of the Isa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath are in similar ranges to those of various monuments from the Byzantine Period in Ayasuluk (6th century) (Işık 2022), the Principalities Period (14th century) in Manisa (Esen et al. 2004; Budak 2005), Ottoman Period (14th – 15th century) baths in Bursa, Edirne, İzmir, Aydın (Böke, Akkurt, and Ipekoğlu 2004; Uğurlu 2005; Gürhan 2018) (Table 5.2).

Table 5.2. The density and porosity of mortar and plaster investigated by recent studies (H: Horosan Plaster L: Lime Plaster)

Location - Referances		Period of Samples	Sample Types	Basic Physical Properties	
				Density (g/cm^3)	Porosity (%)
Çukur Bath - Manisa (Esen et al. 2004; Budak 2005)		14^{th} century	Mortar (Esen et al. 2004)	1.62 - 2.22	29 - 32
			Plaster (H) (Budak 2005)	0.97 - 1.84	29 - 59
Hacet Mescidi - Manisa (Budak 2005)		14^{th} century	Mortar	1.89	29.3
Böke, Akkurt, and Ipekoğlu 2004	Ördekli Bath - Bursa	14^{th} century	Plaster (H)	avg. 1.4	avg. 43
	Beylerbeyi Bath - Edirne	15^{th} century	Plaster (H)	avg. 1.7	avg. 26
	Saray Bath - Edirne		Plaster (H)	avg. 1.6	avg. 32
Çizer, Böke, and İpekoğlu 2004; Uğurlu 2005	Düzce Bath - Urla, İzmir	15^{th} century	Mortar (Çizer et al. 2004)	1.50	38.6
			Plaster (H_L) (Uğurlu 2005)	1.3 - 1.6 _ 1.3 - 1.7	31 - 48 _ 24 - 41
	Herekzade Bath - Urla, İzmir		Mortar (Çizer et al. 2004)	1.50	36.9
			Plaster (H_L) (Uğurlu 2005)	1.1 - 1.5 _ 1.3	40 - 57 _ 40
	Kamanlı Bath - Urla, İzmir		Mortar (Çizer et al. 2004)	1.70	32.8
			Plaster (H_L) (Uğurlu 2005)	1.1 - 1.7 _ 1.3 - 1.8	32 - 54 _ 29 - 45
Işık 2022	Kadıkalesi Anaia (K) - Kuşadası	13^{th} - 14^{th} century	Mortar	1.54	33.71
			Plaster (Brick Agg.)	1.30 - 1.42	43.62 - 47.49
	Ayasuluk Hill (A) - Selçuk, Izmir	6^{th} century	Mortar	1.45	42.60
Taşdibi Andriake Port- Antalya (Oğuz, Türker, and Koçkal 2015; Oğuz 2013)		13^{th} century	Mortar	1.57 - 1.85	30 - 40
Solak 2016	Kızıl Han - Muğla	14^{th} century	Mortar	2.67 - 2.74	30 - 38
	Karapaşa Madrasah - Muğla		Mortar	2.70 - 2.71	29 - 33
	Yelli Mosque - Muğla		Mortar	2.78 - 2.80	35 - 36
Stefanidou et al. 2014	Byzantine Bath - Greece	13^{th} - 14^{th} century	Mortar	-	11.8 - 18.6
	Pazar Bath - Greece	13^{th} - 14^{th} century	Mortar	-	18 - 25
Eski Bath - Efeler, Aydın Gürhan 2018		15^{th} - 16^{th} century	Plaster (H_L)	1.2 - 1.4 _ 1.3 - 1.7	47 - 53 _ 29 - 52

5.2. Raw Material Compositions

The raw material compositions of mortars and plasters were defined by lime/aggregate ratios and particle size distributions of aggregates.

The mortar samples with natural aggregates had a percentage of lime and aggregate values varying between 24.6 – 37.77% and 62.23 – 75.4% respectively. Lime/aggregate ratios of the mortars were found in the range of 0.33 – 0.61 (Table 5.3).

Horasan plasters with brick aggregates were found to be composed of 54.76 – 72.24% lime and 27.76 – 45.24% aggregate by weight. Lime/aggregate ratios of horasan plasters were in the range of 1.21 to 2.6 (Table 5.3).

The percentage of lime and aggregate values of lime plasters with natural stone aggregates of the baths ranged between 43.2 – 79.93% and 20.07 – 53.32% by weight. Their lime/aggregate ratios were found in the range of 0.76 – 3.98 (Table 5.3).

The lime plasters had lime/aggregate ratios ranging from 10.64 to 99, with the percentage of lime and aggregate values ranging from 91.41% to 99.03% and 0.87% to 8.59% by weight, respectively (Table 5.3). This indicated that a high amount of lime was used in their production.

According to the results, the lime/aggregate ratio of the horasan plaster of the Kale Altı Bath is higher than the other baths (Table 5.3). The average lime/aggregate ratios of the lime plasters with natural stone aggregates collected from the upper and lower levels of all baths were analyzed. The results indicated a significant difference in the lime content between the two levels, with the lower-level lime plasters with natural stone aggregates exhibiting a lower lime/aggregate ratio (avg. 0.82) compared to those from the upper levels (avg. 2.05). (Table 5.3). In addition, these values indicate that the average lime/aggregate ratios of Horasan plaster (avg. 1.56) and lime plasters with natural stone aggregates (avg. 1.55) were in close ranges (Table 5.3).

The lime/aggregate ratios of the mortars, horasan plaster, plasters with natural stone aggregates, and lime plaster are in a similar range to previous studies (Esen et al. 2004; Çizer, Böke, and İpekoğlu 2004; Budak 2005; Böke, Akkurt, and Ipekoğlu 2004; Stefanidou et al. 2014; Işık 2022; Solak 2016; Oğuz, Türker, and Koçkal 2015; Uğurlu 2005) (Table 5.4).

Table 5. 3. Lime and aggregate percentages and lime/aggregate ratios of samples

Name	Sample	Sample Types	Lime (%)	Aggregate (%)	Lime / Agg.
İsa Bey Bath	**ITM**	Mortar	37.15	62.85	0.59
	IHLP1	Horasan P.	64.44	35.56	1.81
	IHLP2	Nat. S.Agg P.	43.2	56.8	0.76
	IHLP3	Nat. S.Agg.P.	46.68	53.32	0.88
	IHUP1	Lime P.		***Insufficient sample size*	
	IHUP2	Lime P.	91.41	8.59	10.64
	IHUP3	Lime P.	99.03	0.87	99
Kale Altı Bath	**KHM**	Mortar	37.77	62.23	0.61
	KHLP1	Horasan P.	72.24	27.76	2.6
	KHLP2	Nat. S.Agg.P.		***Insufficient sample size*	
	KHUP1	Lime P.	94.53	5.47	17.29
	KHUP2	Nat. S.Agg.P.	56.88	43.12	1.32
Yahşi Bey Bath	**YDM**	Mortar	24.6	75.4	0.33
	YDLP1	Horasan P.	54.76	45.24	1.21
	YDLP2	Nat. S.Agg.P.		***Insufficient sample size*	
	YDUP1	Lime P.		***Insufficient sample size*	
	YDUP2	Nat. S.Agg.P.	79.93	20.07	3.98
	YDUP3	Nat. S.Agg.P.	46.07	53.93	0.85

(**: Can not be determined due to insufficient sample size)

Table 5.4. Lime and aggregate percentages and lime/aggregate ratios investigated by recent studies (H: Horasan Plaster L: Lime Plaster)

Location - Referances		Period of Samples	Sample Types	Raw Material Compositions		
				Lime (%)	Aggregate (%)	Lime / Agg.
Çukur Bath - Manisa (Esen et al. 2004; Budak 2005)		14th century	Mortar (Esen et al. 2004)	avg. 40	avg. 60	avg. 0.66
			Plaster (H) (Budak 2005)	avg. 52.5	avg. 47.5	avg. 1.1
Hacet Mescidi - Manisa (Budak 2005)		14th century	Mortar	35 - 38	62 - 65	0.53 - 0.61
Böke, Akkurt, and Ipekoğlu 2004	Ördekli Bath - Bursa	14th century	Plaster (H)	avg. 49	avg. 51	avg. 0.96
	Beylerbeyi Bath - Edirne	15th century	Mortar	-	-	0.35
			Plaster (H)	avg. 45	avg. 55	avg. 0.81
	Saray Bath - Edirne		Mortar	-	-	0.35
			Plaster (H)	avg. 58	avg. 42	avg. 1.3
Çizer, Böke, and İpekoğlu 2004; Uğurlu 2005	Düzce Bath - Urla, İzmir	15th century	Mortar (Çizer et al. 2004)	-	-	0.66
			Plaster (H_L) (Uğurlu 2005)	avg. 51_-	avg. 49_-	0.66-1.5_17-32
	Herekzade Bath - Urla, İzmir		Mortar (Çizer et al. 2004)	-	-	0.5
			Plaster (H_L) (Uğurlu 2005)	avg. 51_-	avg. 49_-	0.8-1.25_24
	Kamanlı Bath - Urla, İzmir		Mortar (Çizer et al. 2004)	-	-	0.66
			Plaster (H_L) (Uğurlu 2005)	avg. 46_-	avg. 54_-	0.5-1_32-99
Işık 2022	Kadıkalesi Anaia (K) - Kuşadası	13th - 14th century	Mortar	57.59	42.41	1.36
			Plaster	27.20 - 57.91	42.09 - 72.80	0.37 - 1.38
	Ayasuluk Hill (A) - Selçuk, Izmir	6th century	Mortar	56.16	43.84	1.28
Taşdibi Andriake Port- Antalya (Oğuz, Türker, and Koçkal 2015; Oğuz 2013)		13th century	Mortar	-	-	0.25
Solak 2016	Kızıl Han - Muğla	14th century	Mortar	-	-	0.5-1
	Karapaşa Madrasah - Muğla		Mortar	-	-	1
	Yelli Mosque - Muğla		Mortar	-	-	0.5-1
Stefanidou et al. 2014	Byzantine Bath - Greece	13th - 14th century	Mortar	-	-	0.5
	Pazar Bath - Greece	13th - 14th century	Mortar	-	-	0.5-0.66
Eski Bath - Efeler, Aydın Gürhan 2018		15th - 16th century	Plaster (H_L)	32-47_93-97	53-68_3-7	0.6-0.7_14-31

The results of sieve analysis to determine the particle size and distribution of aggregates in mortar and plaster samples are shown in Figure 5.3, Figure 5.4, Figure 5.5, and Table 5.5. The particle size distribution of the mortars with natural aggregate indicated that aggregates with particle sizes greater than 1180 µm were a major fraction of the total aggregates, ranging from 24.52% to 47.35% by weight (Figure 5.3-5.5, Table. 5.5).

Horasan plasters with brick aggregates exhibit a particle size distribution with a range of 17.76-28.45% by weight for particles between 1180 and 250 µm. Fine aggregates (<125 µm) comprise 12.89-16.16% of the horasan plaster. (Figure 5.3-5.5, Table. 5.5).

The particle size distribution of the lime plasters with natural stone aggregates indicated that aggregates with particle size greater than 1180µm were a major fraction (9.97 % - 29.75% by weight) of the total aggregates (except for YDUP2). However, the aggregate of the YDUP2 sample was mostly composed of particle sizes less than 250µm.

Analysis of the lime plasters revealed that aggregates less than 125 µm are the main fraction, ranging from 0.77 to 3.82% of the total aggregate (Figure 5.3-5.5, Table. 5.5).

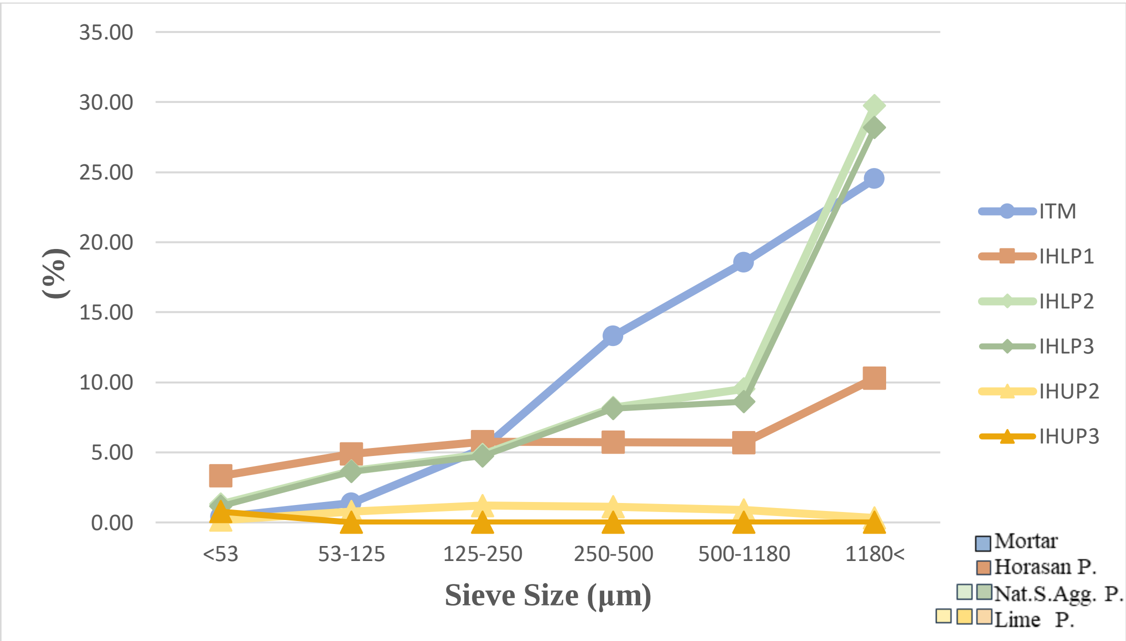


Figure 5. 3. Particle size distributions of aggregates from İsa Bey Bath

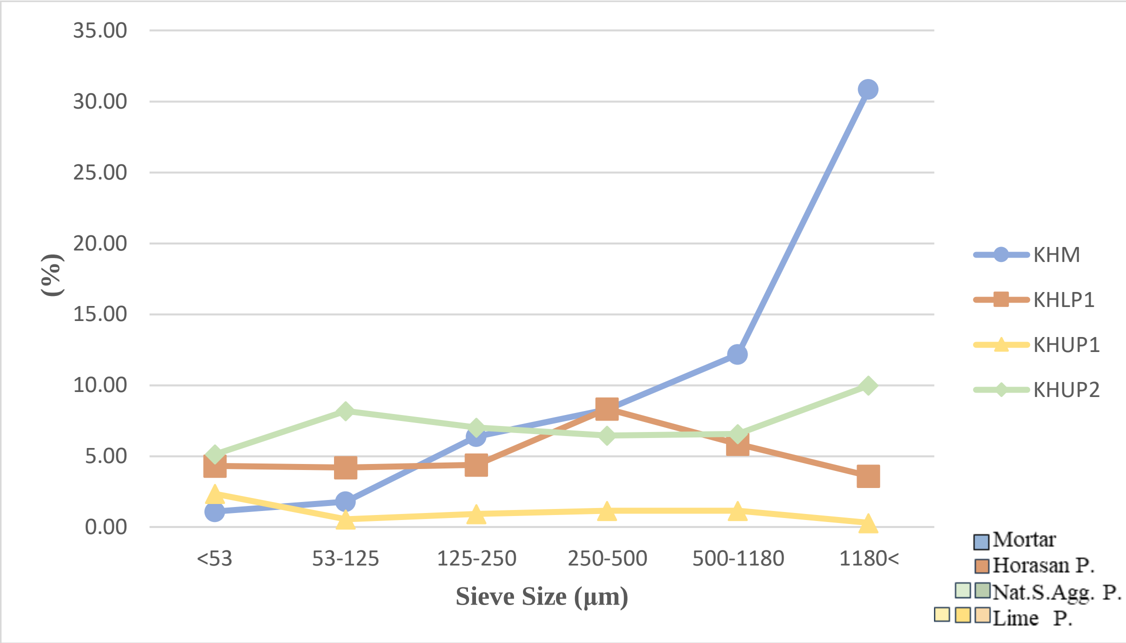


Figure 5. 4. Particle size distributions of aggregates from Kale Altı Bath

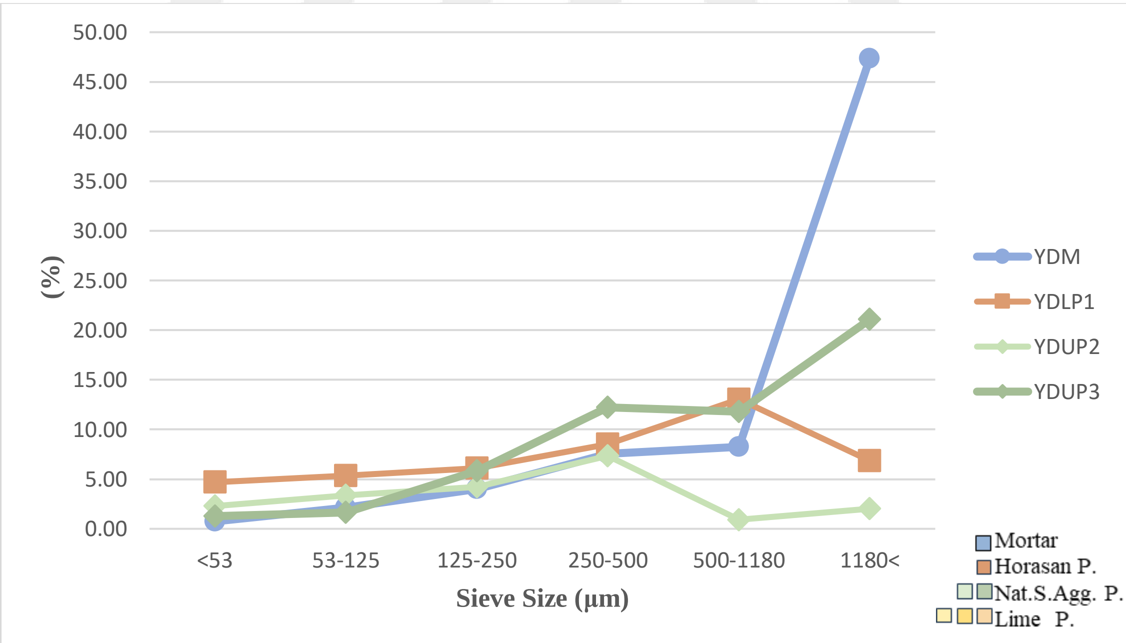


Figure 5. 5. Particle size distributions of aggregates from Yahşi Bey Bath

Table 5.5. Particle size distributions of the aggregates

Sample	Sample Types	Sieve Size (µm)					
		<53	53-125	125-250	250-500	500-1180	1180<
ITM	Mortar	0.38	1.39	5.26	13.31	18.57	24.52
IHLP1	Horasan P.	3.31	4.90	5.78	5.71	5.69	10.30
IHLP2	Nat.S.Agg. P.	1.30	3.66	4.85	8.21	9.51	29.75
IHLP3	Nat.S.Agg. P.	1.16	3.64	4.75	8.13	8.60	28.17
IHUP1	Lime P.	** *insufficient sample size*					
IHUP2	Lime P.	0.16	0.76	1.21	1.12	0.88	0.33
IHUP3	Lime P.	0.77	0.00	0.00	0.00	0.00	0.00
KHM	Mortar	1.09	1.79	6.37	8.31	12.15	30.86
KHLP1	Horasan P.	4.30	4.20	4.39	8.32	5.84	3.60
KHLP2	Nat.S.Agg. P.	** *insufficient sample size*					
KHUP1	Lime P.	2.34	0.55	0.93	1.15	1.15	0.30
KHUP2	Nat.S.Agg. P.	5.12	8.16	7.01	6.43	6.57	9.97
YDM	Mortar	0.75	2.10	3.98	7.54	8.27	47.35
YDLP1	Horasan P.	4.68	5.37	6.10	8.54	13.05	6.86
YDLP2	Nat.S.Agg. P.	** *insufficient sample size*					
YDUP1	Lime P.	** *insufficient sample size*					
YDUP2	Nat.S.Agg. P.	2.30	3.38	4.23	7.34	0.92	2.00
YDUP3	Nat.S.Agg. P.	1.28	1.65	5.84	12.23	11.78	21.08

(**: Can not be determined due to insufficient sample size)

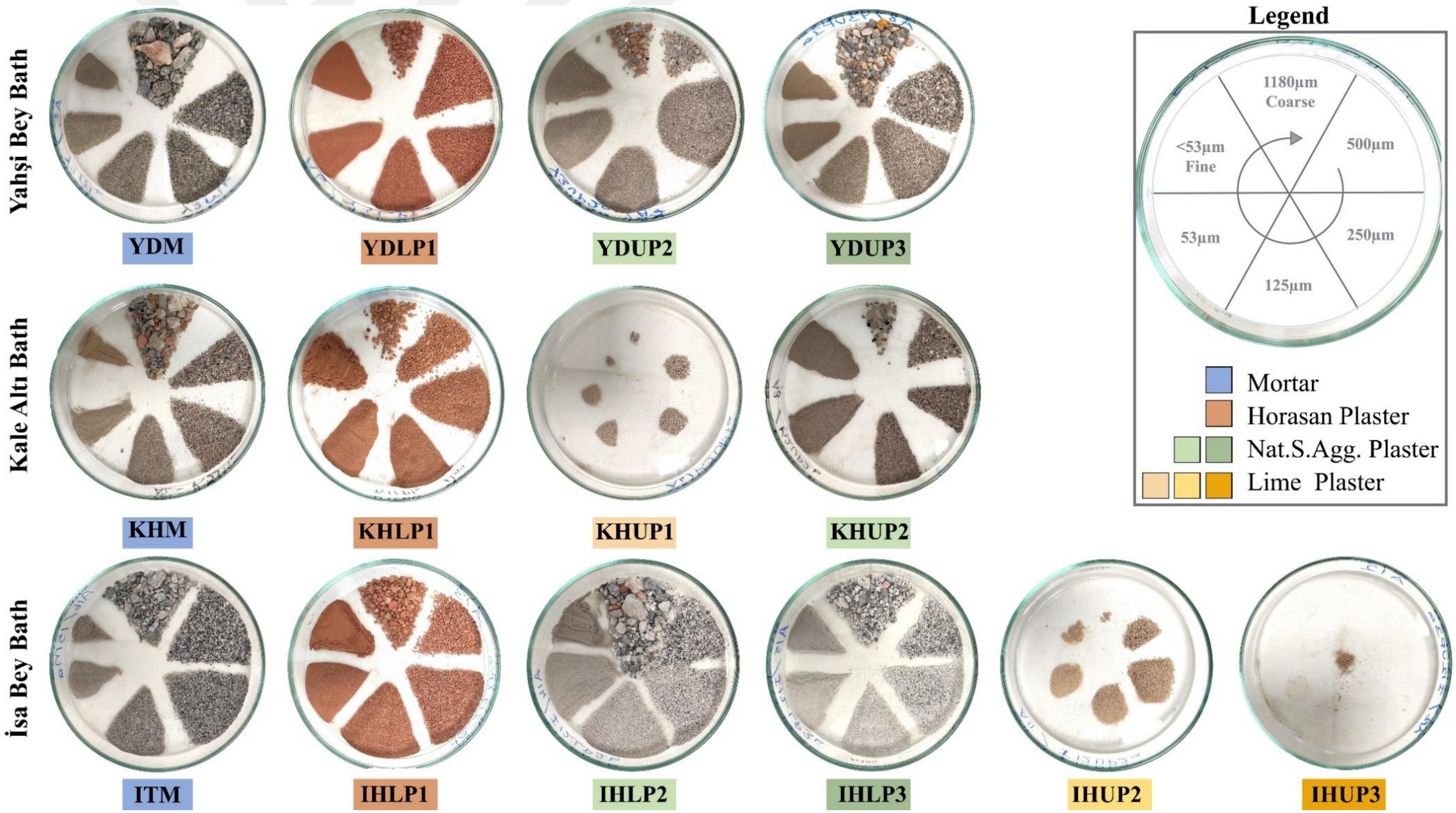


Figure 5.6. Distribution of aggregates in samples from coarse to fine

5.3. Characteristics of Lime (White) Lumps

In lime mortars and plasters, small white nodules ranging in size from a few millimeters to 2 cm in size were described as white (lime) lumps (Bakolas et al. 1995). These white lumps indicate the presence of lime used as a binder. The chemical and mineralogical composition of the lumps is generally accepted to be identical to that of the raw material (Bakolas et al. 1995). The chemical compositions of the white lumps were determined via scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS) analysis.

Table 5.6. Chemical compositions of the white (lime) lumps in studied bath buildings

Sample	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	Fe_2O_3
IHLP3	0.16	1.09	3.52	0.89	0.23	93.90	0.08	0.13
KHUP2	1.03	1.53	1.70	1.98	1.16	92.14	0.06	0.18
YDUP2	0.37	1.03	1.25	0.46	0.57	96.08	0.07	0.17

The results of SEM-EDS analysis indicated that white (lime) lumps in lime plasters with natural stone aggregates from the İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath were mainly composed of large amounts of CaO (92.14 – 96.08%), moderate amounts of Al_2O_3 (1.25 – 3.52%), and smaller amounts of MgO (1.03 – 1.53%), SiO_2 (0.46 – 1.98 %), K_2O (0.23 – 1.16 %), Na_2O (0.16 – 1.03%), Fe_2O_3 (0.13 – 0.18%), and TiO_2 (0.06 – 0.08%) (Table 5.6). The chemical compositions of the white lumps in lime plasters with natural stone aggregates from studied bath buildings were found to be similar to each other. The high CaO content in the white lumps suggests the use of air lime in the production of mortars and plasters (Cowper 1998).

Mineralogical compositions of lime lumps from İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath were determined via XRD. On the XRD patterns, lime lump samples from plaster with natural stone aggregates (IHLP3, KHUP2, YDUP2) were composed of only calcite (CaCO3) mineral which was derived from carbonated lime (Figure 5.7.). In the buildings of similar periods, it was determined that the lime lumps were non-hydraulic, air lime was used in their production, and composed only of calcite (Budak 2005; Uğurlu 2005; Işık 2022; Çizer, Böke, and İpekoğlu 2004).

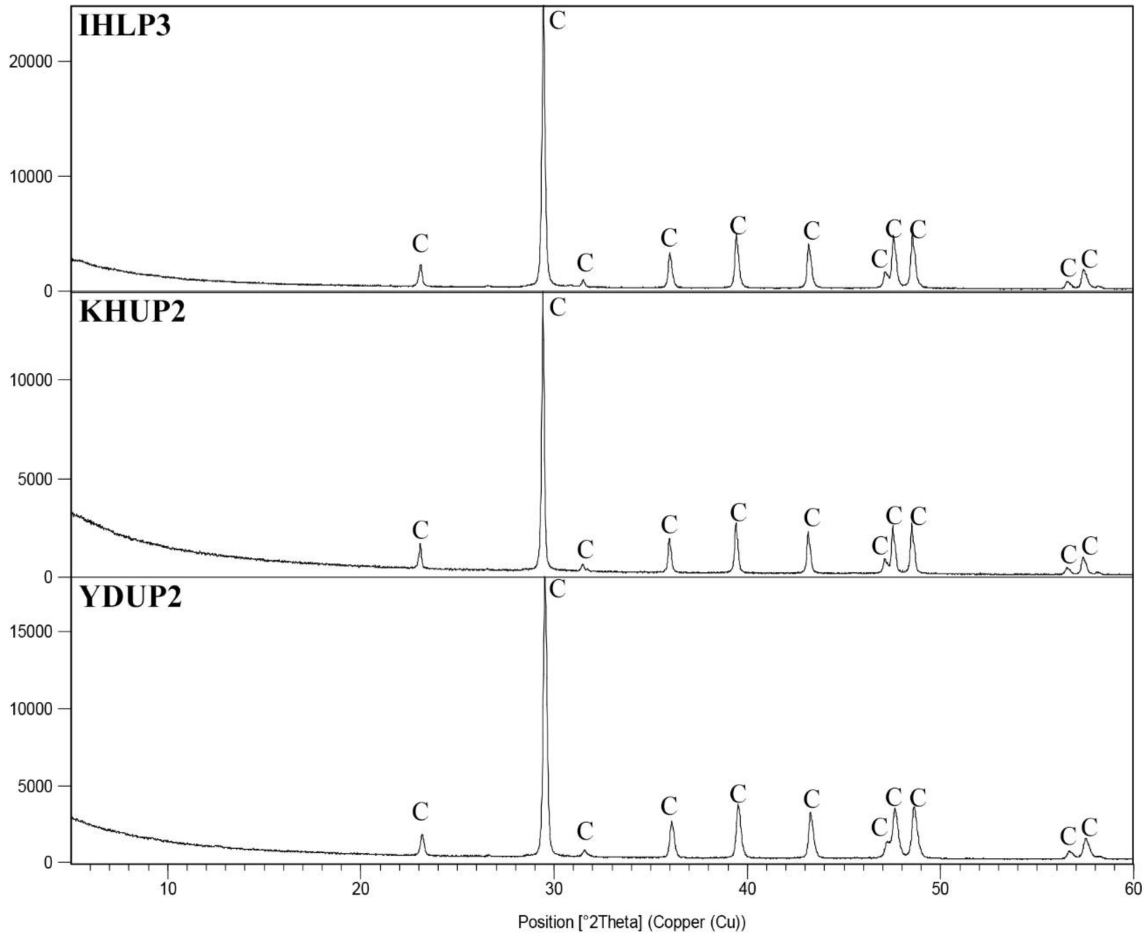


Figure 5. 7. XRD patterns of lime lumps (C: Calcite 86-2334)

5.4. Chemical, Mineralogical Compositions and Pozzolanic Activities of Aggregates

The chemical compositions of aggregates in mortars and plasters were specified via SEM-EDS. Mineralogical compositions of the aggregates were determined by XRD analysis. Electrical conductivity differences (ΔEC) of a calcium hydroxide solution were used to evaluate the pozzolanic activity of fine aggregates.

5.4.1. Characteristics of Natural Aggregates

The chemical compositions of natural aggregates in mortars and plasters were determined with pellets prepared from fine powder samples with a particle size of less than 53 µm by a Philips XL 30S-FEG Scanning Electron Microscope (SEM) coupled with an Energy Dispersive X-ray Spectroscopy (EDS).

The results of SEM-EDS analysis indicated that fine natural aggregates in the mortars from the İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath were mainly composed of large amounts of SiO_2 (74.85%, 70.16%, and 64.42% respectively), moderate amounts of Al_2O_3 (11.69%, 13.49%, and 14.72 %) and smaller amounts of MgO (4.18%, 3.96% and 7.21%), Fe_2O_3 (3.90%, 5.31%, and 7.02 %), K_2O (2.15%, 1.38%, and 2.44 %), CaO (1.19%, 1.98 %, and 1.48%), TiO_2 (1.19%, 1.25 %, and 1.17%), and Na_2O (0.86%, 2.45%, and 1.56%) (Table 5.8). The research conducted in Aysuluk (Işık 2022) found that the average SiO_2 and Al_2O_3 contents in the chemical composition of natural aggregates used in mortar production are in a similar range with studied bath buildings (Table 5.9).

Lime plasters with natural stone aggregates used in İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath were mainly comprised of large amounts of SiO_2 (86.48 – 89.70%, 81.79%, and 70.50 – 73% respectively), moderate amounts of Al_2O_3 (5.52 – 5.75%, 7.87%, and 12.38 – 14.25 %), and smaller amounts of Fe_2O_3 (1.55 – 2.60%, 1.45%, and 4.88 – 5.33 %), MgO (1.06 – 2.38%, 0.95%, and 3.46 – 5.02%), K_2O (0.61 – 1.15%, 1.77%, and 1.55 – 3.37%), CaO (0.59 – 0.77%, 4.38 %, and 0.76 – 1.79%), TiO_2 (0.38 – 0.77%, 0.72%, and 0.86 – 0.90%) and Na_2O (0.11 – 0.38%, 1.08, and 0.35 - 1.60%) (Table 5.8). The studied bath buildings revealed that the mortar, and lime plaster with natural stone aggregate samples of all baths exhibit similar chemical compositions and they were found to contain high levels of silica and alumina, but low levels of carbonate and alkaline phases.

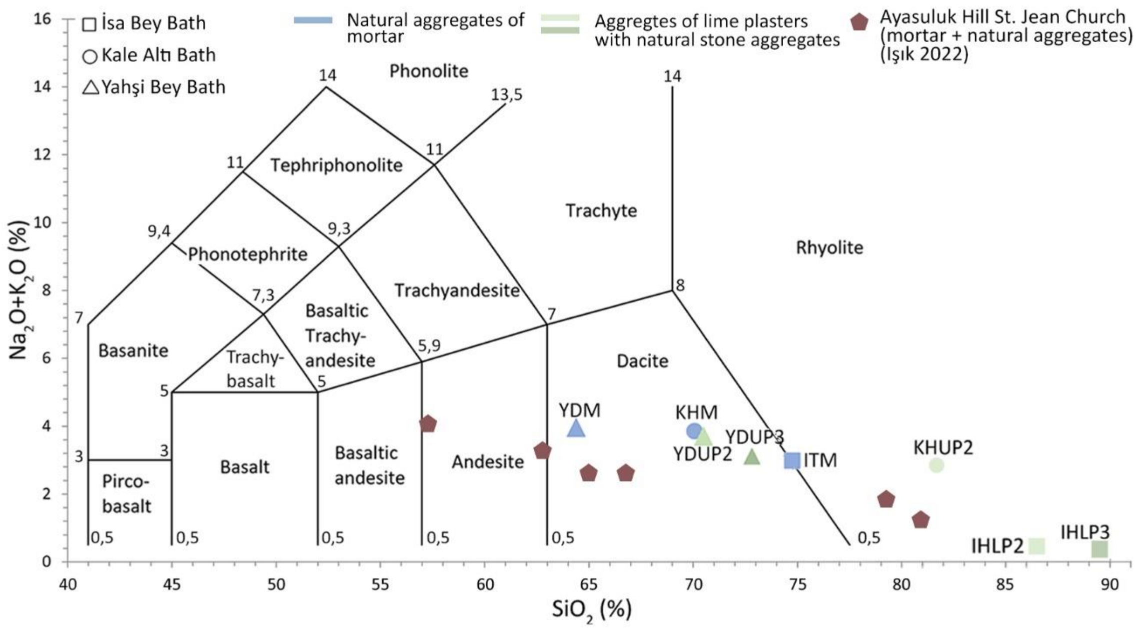


Figure 5. 8. TAS (Total Alkali-Silica) diagram showing the geochemical sources of fine natural aggregates of mortars and lime plasters with natural stone aggregates (Le Maitre et al. 2003)

To identify the potential geochemical sources of fine natural aggregates of mortar and lime plasters with natural stone aggregates the chemical compositions were considered using the Total Alkali-Silica (TAS) diagram (Le Maitre et al. 2003). The silica (SiO_2) content of the natural aggregates of mortar and plaster in Isa Bey Bath varied between 74.85% and 89.70%, and the alkali (Na_2O+K_2O) contents were between 0.99% and 3.01% (Figure 5.8). Natural aggregates of Kale Altı Bath exhibited a silica content ranging from 70.16% to 81.79%, and an alkali content between 2.85% and 3.83%. In Yahşi Bey Bath natural aggregates, silica content varied between 64.42 – 73.00%, alkali content varied between 3.15 – 4.00%. The dominant igneous rocks found in the aggregates of the studied bath buildings are dacite and rhyolite. However, the natural aggregates of the plaster samples (KHUP2, IHLP2, IHLP3) of the Isa and Kale Altı baths were in the rhyolite class (like mortar with natural aggregate samples from Ayasuluk Hill (Işık 2022)), while the natural aggregates of plaster (YDUP2) from the Yahşi bey baths were in dacite class. The natural aggregates used in the mortars which are ITM, KHM, and YDM of studied bath buildings were in the dacite class like some of the mortars with natural aggregate samples from Ayasuluk Hill (Işık 2022) (Figure 5.8). It is not possible to make a definitive interpretation of the origin of fine aggregates using TAS diagrams alone. Therefore, future studies should search for detailed chemical analyses and the geology of the region.

Mineralogical compositions of the natural aggregates in mortars were determined by XRD analysis. XRD results revealed that the aggregates of Isa Bey Bath, Kale Altı Bath and Yahşi Bey Bath were composed of albite ($Na(AlSi_3O_8)$), clinochlore ($(Mg,Fe)_5Al(Si_3Al)O_{10}(OH)_8$), muscovite ($KAl_2(Si_3Al)O_{10}(OH, F)_2$), orthoclase ($K(AlSi_3O_8)$), quartz ($SiO_2$), phillipsite ($(KCa(Si_5Al_3)O_{16}.6H_2O)$, oligoclase $(Na,Ca)[Al(Si,Al)Si_2O_8]$ and hornblende ($(Ca,Na)_2(Mg,Fe,Al)_5(Al,Si)_8O_{22}(OH)_2$) (Figure 5.10). Due to the location of the baths close to volcanic areas such as Seferihisar-Doğanbey, the hornblende and phillipsite minerals found in the natural aggregates may have originated from these volcanic units (Işık 2022). Previous studies have revealed that the natural aggregates in the mortars are mainly composed of quartz, albite, and muscovite minerals, while anorthite was found in Çukur Hamam and Hacet Mescidi; clinochlore, hornblende, orthoclase, and phillipsite in Ayasuluk Hill (Çizer, Böke, and İpekoğlu 2004; Budak 2005; Işık 2022) (Table 5.7). The XRD results of the mortar natural aggregates used in the Ayasuluk Hill and Isa Bey Baths were found similar compositions, and both structures could use materials from the same sources.

The lime plaster with natural stone aggregates was mainly composed of quartz, albite, graphite (C), clinochlore, muscovite, phillipsite ($(KCa(Si_5Al_3)O_{16}.6H_2O)$, plagioclase feldspar $(Na,Ca)[(Si,Al)AlSi_2]O_8$ minerals (Figure 5.10). Phillipsite minerals found in the natural aggregates may have originated from these volcanic units from Seferihisar- Doğanbey volcanic areas.

On the XRD pattern of the aggregates from lime plasters albite, quartz, clinochlore, and muscovite minerals were found in İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath (Figure 5.11.) Similarly, in Eski Bath, albite, quartz, and calcite minerals were found in aggregates of lime plasters (Gürhan 2018).

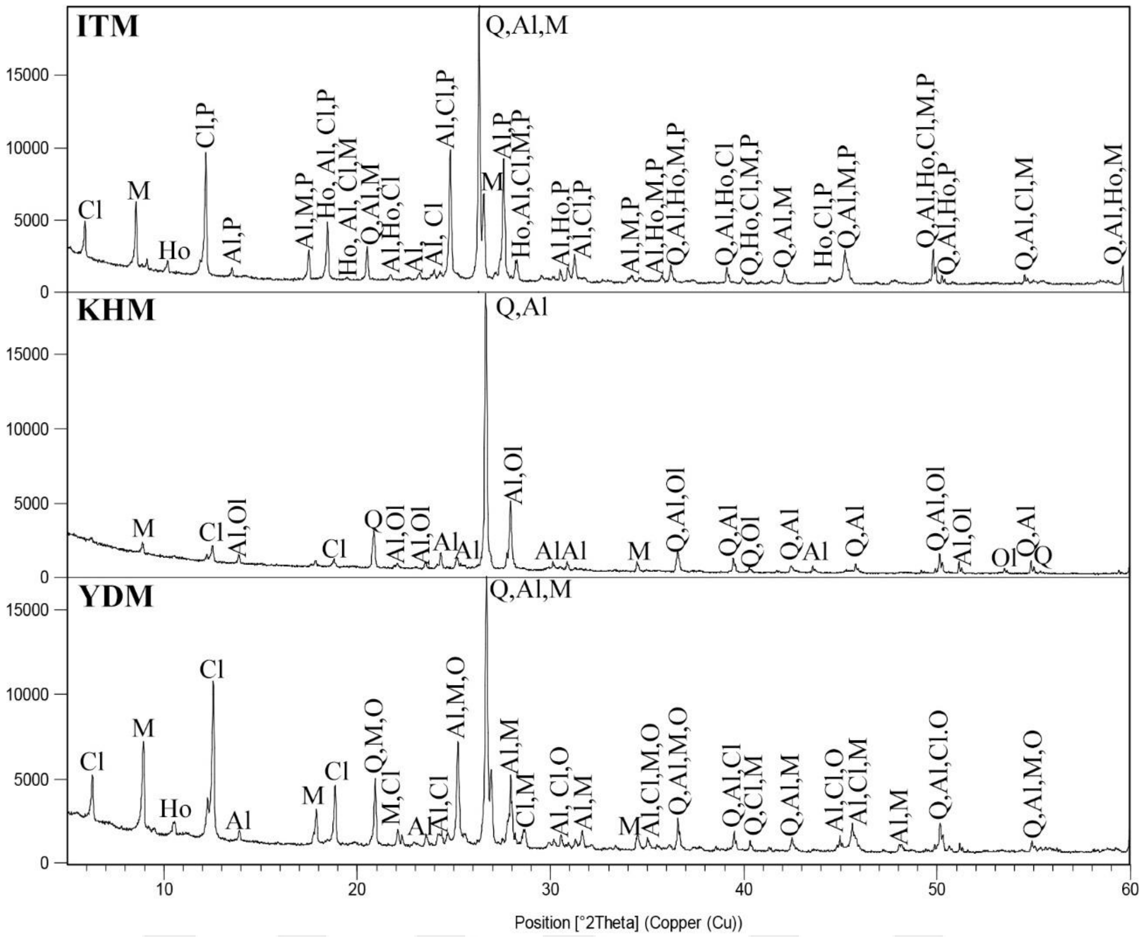


Figure 5. 9 XRD patterns of natural aggregates in mortars from İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath (A: Albite, Q: Quartz, M: Muscovite, Cl: Clinochlore, Ho: Hornblende, O: Orthoclase, P: Phillipsite, Ol: Oligaclase)

Table 5.7. Mineralogical composition of natural aggregates in mortar in previous studies

Location - References		**Q**	**O**	**P**	**Al**	**A**	**Cl**	**M**	**Ho**
Budak 2005	Çukur Bath	+			+	+		+	
	Hacet Mescidi	+			+	+		+	
Işık 2022	Kadıkalesi Anaia	+			+		+	+	
	Ayasuluk Hill	+	+	+	+		+	+	+
Çizer, Böke, and İpekoğlu 2004)	Düzce Bath	+			+			+	
	Herekzade Bath	+			+			+	
	Kamanlı Bath	+			+			+	

Q: Quartz, O: Orthoclase, P: Phillipsite, Al: Albite, A: Anorthite, Cl: Clinochlore, M: Muscovite, Ho: Hornblende

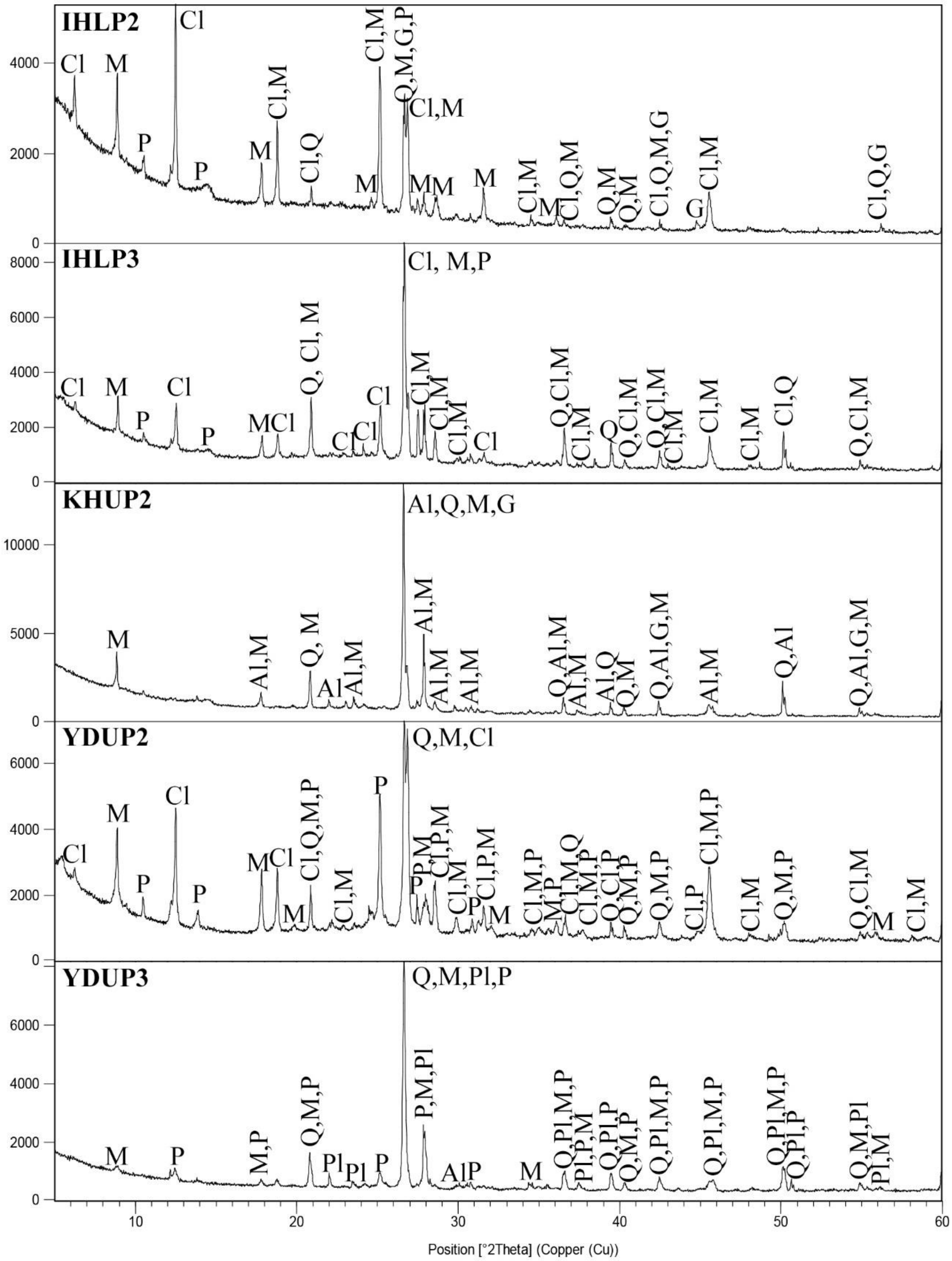


Figure 5. 10. XRD patterns of natural aggregates in plaster with natural stone aggregates from İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath (G: Graphite Al: Albite Q: Quartz Cl: Clinochlore M: Muscovite P: Phillipsite Pl: Plagioclase Feldspar)

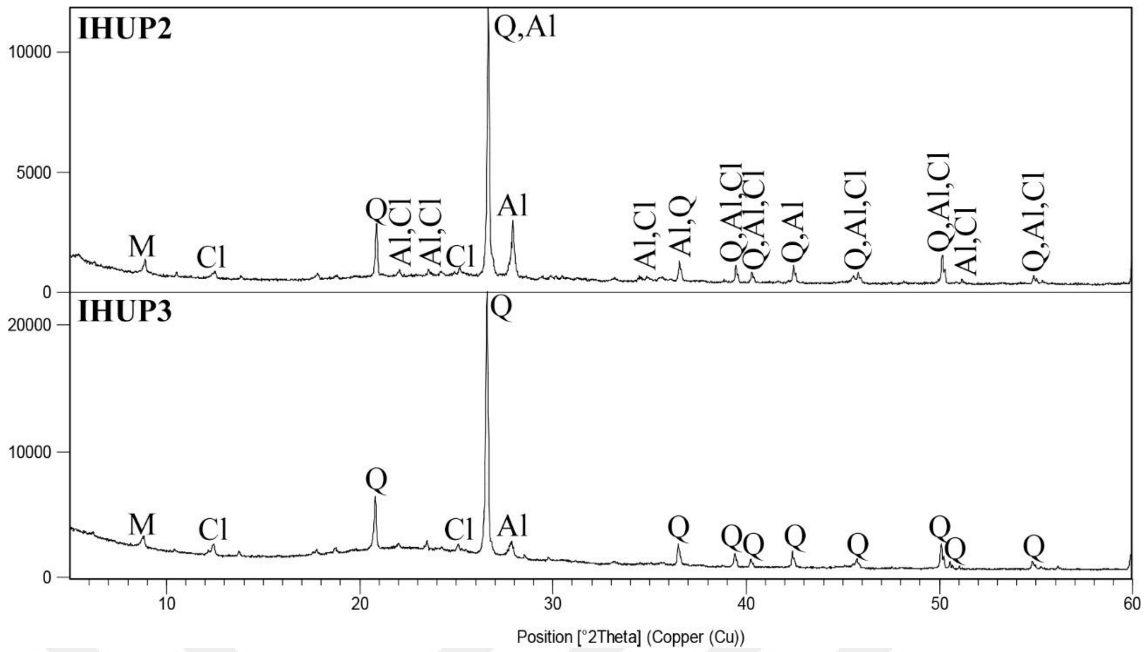


Figure 5. 11. XRD patterns of natural aggregates in lime plaster from İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath (Al: Albite, Cl: Clinochlore, M: Muscovite, Q: Quartz)

Table 5.8. Chemical compositions of the fine aggregates in Isa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath

Name	Sample	Sample Types	Aggregate types	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	Fe_2O_3
İsa Bey Bath	**ITM**	Mortar	Natural	0.86	4.18	11.69	74.85	2.15	1.19	1.19	3.90
	IHLP1	Horasan P.	Brick	0.42	4.06	14.14	69.66	2.08	0.96	0.94	7.75
	IHLP2	Nat.S.Agg P.	Natural	0.11	2.38	5.75	86.48	1.15	0.77	0.77	2.60
	IHLP3	Nat.S. Agg P.	Natural	0.38	1.06	5.52	89.70	0.61	0.59	0.38	1.55
	IHUP1	Lime P.	-	**: *Can not be determined due to insufficient sample size*							
	IHUP2	Lime P.	Natural	0.59	2.85	8.65	79.01	1.40	1.88	0.72	4.89
	IHUP3	Lime P.	Natural	**: *Can not be determined due to insufficient sample size*							
Kale Altı Bath	**KHM**	Mortar	Natural	2.45	3.96	13.49	70.16	1.38	1.98	1.25	5.31
	KHLP1	Horasan P.	Brick	0.55	5.92	17.74	61.01	2.33	1.96	0.97	9.53
	KHLP2	Nat.S. Agg P.	Natural	**: *Can not be determined due to insufficient sample size*							
	KHUP1	Lime P.	Natural	0.33	1.36	4.10	90.52	0.58	0.73	0.41	1.97
	KHUP2	Nat. Agg P.	Natural	1.08	0.95	7.87	81.79	1.77	4.38	0.72	1.45
Yahşi Bey Bath	**YDM**	Mortar	Natural	1.56	7.21	14.72	64.42	2.44	1.48	1.17	7.02
	YDLP1	Horasan P.	Brick	0.94	4.84	14.08	69.65	1.59	1.01	0.79	7.09
	YDLP2	Nat. S.Agg P.	Natural	**: *Can not be determined due to insufficient sample size*							
	YDUP1	Lime P.	-	**: *Can not be determined due to insufficient sample size*							
	YDUP2	Nat.S.Agg P.	Natural	0.35	5.02	14.25	70.50	3.37	0.76	0.86	4.88
	YDUP3	Nat.S.Agg P.	Natural	1.60	3.46	12.38	73.00	1.55	1.79	0.90	5.33

Table 5.9. Chemical Compositions of the aggregates in previous studies

Location - Referances		**Period of Samples**	**Sample Types**	**Chemical Composition of Fine Aggregates (%)**							
				Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	Fe_2O_3
Çukur Bath - Manisa (Budak 2005)		14th century	Mortar	2 - 4	2 - 3	6 -10	73-85	1-3	2-3	1	1-3
Işık 2022	Ayasuluk Hill (A) - Selçuk, Izmir	6th century	Mortar	0.50 - 1.05	3.57 - 8.81	7.48 - 18.34	57.22 - 80.85	1.09 - 3.18	0.82 - 1.84	0.75 - 1.28	3.70 - 9.13
			Plaster	0.51 - 0.55	4.82 - 6.58	12.91 - 16.06	61.96- 69.24	2.04 - 2.47	1.51 - 1.65	0.92 - 1.54	7.29 - 9.97
	Kadıkalesi Anaia (K) - Kuşadası	13th - 14th century	Mortar	0.37	2.24	15.27	71.14	3.27	0.85	1.46	5.40
Böke, Akkurt, and Ipekoğlu 2004	Ördekli Bath - Bursa	14th century	Horasan Plaster	1.53 - 5.83	1.71 - 4.44	8.36 - 14.28	58.29 - 80.73	1.24 - 8.21	-	-	5.88 - 8.99
	Beylerbeyi Bath - Edirne	15th century	Horasan Plaster	0.92 - 2.97	2.92 - 5.52	9.00 - 21.36	52.37 - 76.15	0.68 - 3.43	-	-	10.33 - 14.85
	Saray Bath - Edirne		Horasan Plaster	3.21	6.22	15.24	53.01	12.35	-	-	9.97
Uğurlu 2005	Düzce Bath - Urla, İzmir	15th century	Horasan Plaster	1.50 - 3.10	1.50 - 2.90	59.00 - 82.10	7.10 - 22.70	1.30 - 4.20	1.00 - 1.60	-	4.70 - 8.30
	Herekzade Bath - Urla, İzmir		Horasan Plaster	1.20 - 2.30	0.60 - 0.90	4.20 - 8.80	81.30 - 92.60	0.80 - 2.10	1.10 - 0.80	-	2.30 - 3.40
	Kamanlı Bath - Urla, İzmir		Horasan Plaster	1.60 - 3.50	1.70 - 1.80	6.80 - 14.20	68.50 - 83.40	1.00 - 3.10	1.20 - 1.70	-	4.10 - 7.20
Eski Bath - Efeler, Aydın (Gürhan 2018)		15th - 16th century	Horasan Plaster	0.68	1.51 - 3.36	10.15 - 19.36	63.50 - 81.51	1.72 - 3.95	0.66 - 1.96	0.81 - 1.00	3.77 - 8.75
			Lime Plaster	0.44 - 0.64	0.65 - 0.76	4.82 - 6.81	86.37 - 90.92	0.93 - 1.48	0.86 - 1.57	0.66 - 0.87	1.91 - 2.38

5.4.2. Characteristics of Brick Aggregates

Fine brick aggregates of horasan plaster from studied bath buildings (İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath) were comprised of mostly large amounts of SiO_2 (69.66%, 61.01%, and 69.65% respectively), moderate amounts of Al_2O_3 (14.14%, 17.74%, and 14.08%), and smaller amounts of Fe_2O_3 (7.75%, 9.53%, and 7.09%), MgO (4.06%, 5.92%, and 4.84%), K_2O (2.08%, 2.33%, and1.59%), CaO (0.96%, 1.96 %, and 1.01%), TiO_2 (0.94%, 0.97 %, and 0.79%), and Na_2O (0.42%, 0.55%, and 0.94%) (Table 5.8). According to previous studies of baths, the percentages of the chemical composition of the brick aggregates in the horasan plasters were found similar (Table 5.9).

The mineralogical compositions of brick aggregates in horasan plasters from İsa bey Bath, Kale Altı Bath, and Yahşi Bay Bath had albite, clinochlore, dolomite $CaMg(CO_3)_2$, hematite (Fe_2O_3), muscovite, and quartz on their XRD patterns (Figure 5.12). Previous studies revealed that brick aggregates in plaster mainly consist of quartz and albite minerals. Also, hematite and muscovite were identified in the plasters of Kadıkalesi Anaia (Işık 2022) and Eski Bath; plagioclase feldspar minerals were found in Ördekli, Beylerbeyi, and Saray Bath (Böke, Akkurt, and Ipekoğlu 2004); potassium feldspar minerals were in the plasters in previous studies (Uğurlu 2005; Gürhan 2018) (Table 5.10). XRD patterns provide information about the mineralogical composition and firing temperatures of brick aggregates used in Horasan plaster (Uğurlu 2005). In the XRD patterns of the brick aggregates, minerals that require high firing temperatures such as wollastonite (rich clays) were not detected; this indicates that the bricks were fired at a temperature below 850 ºC (Cardiano et al. 2004). Additionally, the presence of hematite (no rich clays) and dolomite (rich clays) minerals suggest a firing temperature of 850 ºC (Cardiano et al. 2004). Additionally, XRD patterns provide information on the pozzolanic properties of the brick aggregates, in the pattern 20-30 degrees 2θ indicates the presence of pozzolanic amorphous materials, most likely originating from highly heated clay minerals (Lee, Kim, and Moon 1999).

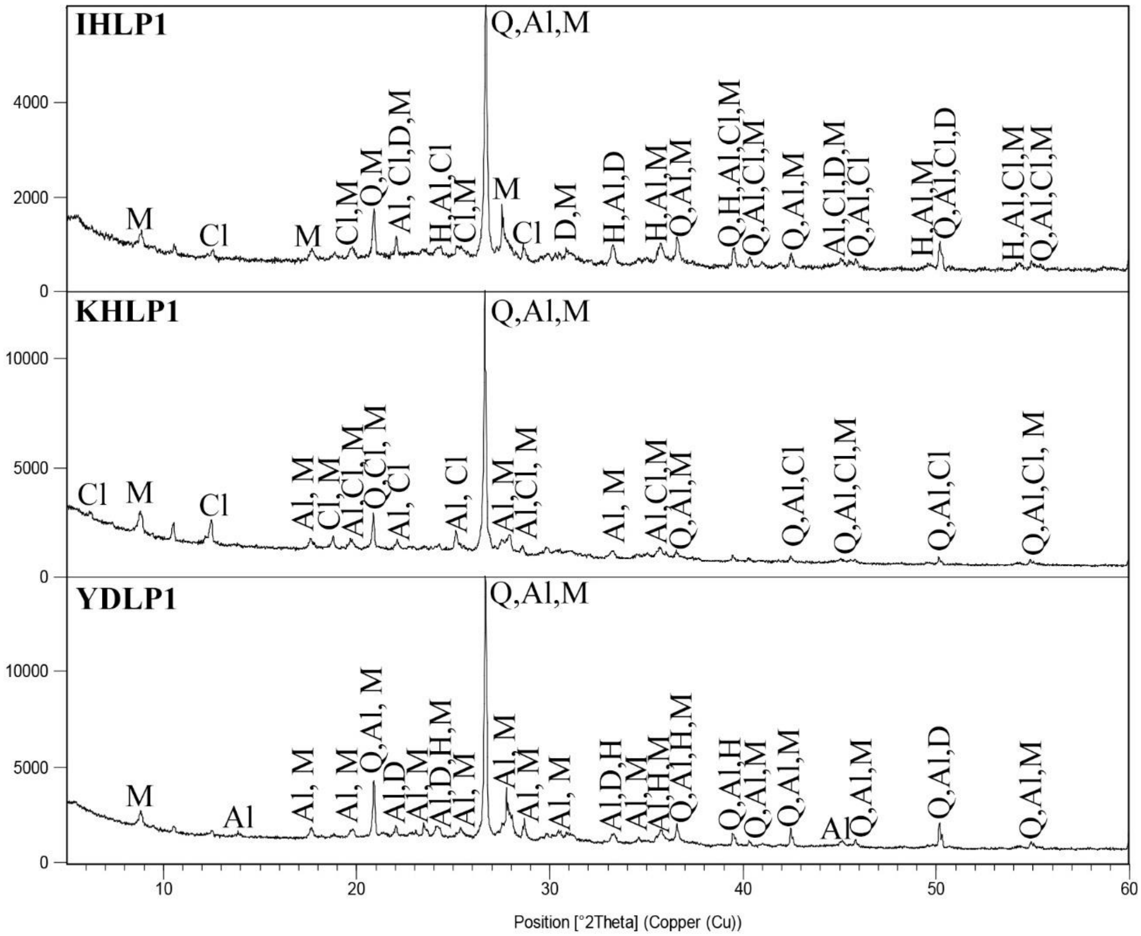


Figure 5. 12. XRD patterns of brick aggregates in horasan plasters from İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath (A: Albite, Q: Quartz, M: Muscovite, Cl: Clinochlore, D: Dolomite H: Hematite)

Table 5.10.Mineralogical composition of brick aggregates in plasters in previous studies

Locations - References		Al	Q	H	M	Pl	Pf	C
Kadıkalesi Anaia (Işık 2022)		+	+	+	+			
Böke, Akkurt, and Ipekoğlu 2004	Ördekli Bath	+	+			+		
	Beylerbeyi Bath	+	+			+		
	Saray Bath	+	+			+		
Uğurlu 2005	Düzce Bath	+	+				+	
	Herekzade Bath	+	+				+	
	Kamanlı Bath	+	+				+	
Eski Bath (Gürhan 2018)		+	+	+	+		+	+

Q: Quartz, Al: Albite, Cl: Clinochlore, H: Hematite, M: Muscovite, Pf: Potassium Feldspar Pl: Plagioclase Feldspar C: Calcite

5.4.3. Pozzolanic Activities of Fine Aggregates

Electrical conductivity differences (ΔEC) of a calcium hydroxide solution were used to evaluate the pozzolanic activity of fine aggregates (less than 53 μm). Aggregates with electrical conductivity differences greater than 1.2 mS/cm indicated pozzolanic properties (Luxán, Madruga, and Saavedra 1989). In addition, the ASTM C618-03 Standard was used to determine pozzolanic properties. The standard specifies that a material is considered pozzolanic if its total content of silicon dioxide (SiO_2), aluminum oxide (Al_2O_3), and iron oxide (Fe_2O_3) is higher than 70% (ASTMC618-03 2003).

Table 5.11. Pozzolanic activities of samples
(ΔEC (mS/cm): Electrical Conductivity Difference)

Name	Sample	Sample Types	Aggregate Types	ΔEC (mS/cm)	SiO_2+ Al_2O_3+ Fe_2O_3 (%)
İsa Bey Bath	**ITM**	Mortar	Natural	6.64	90.44
	IHLP1	Horasan P.	Brick	6.98	91.54
	IHLP2	Nat. S. Agg P.	Natural	7.37	94.82
	IHLP3	Nat. S. Agg P.	Natural	7.53	96.78
	IHUP1	Lime P.	-	***Insufficient sample size*	
	IHUP2	Lime P.	Natural	6.35	92.55
	IHUP3	Lime P.	Natural	***Insufficient sample size*	
Kale Altı Bath	**KHM**	Mortar	Natural	6.00	88.96
	KHLP1	Horasan P.	Brick	5.96	88.28
	KHLP2	Nat. S. Agg P.	Natural	***Insufficient sample size*	
	KHUP1	Lime P.	Natural	**	96.59
	KHUP2	Nat. S. Agg P.	Natural	7.45	91.11
Yahşi Bey Bath	**YDM**	Mortar	Natural	2.82	86.16
	YDLP1	Horasan P.	Brick	6.50	90.82
	YDLP2	Nat. S. Agg P.	Natural	***Insufficient sample size*	
	YDUP1	Lime P.	-	***Insufficient sample size*	
	YDUP2	Nat. S. Agg P.	Natural	5.40	90.50
	YDUP3	Nat. S. Agg P.	Natural	4.32	90.71

**: Can not be determined due to insufficient sample size

The electrical conductivity (ΔEC) of the aggregates of mortar samples ranged between 2.82 - 6.64 mS/cm. The contents of $SiO_2 + Al_2O_3 + Fe_2O_3$ varied from 86.16% to 90.44% (Table 5.11).

The Horasan plasters brick aggregates showed electrical conductivity differences ranging from 5.96 to 6.98 mS/cm and the SiO_2, Al_2O_3, and Fe_2O_3 contents varied between 88.28% -91.54% (Table 5.11). The lime plasters with natural stone aggregate samples exhibited electrical conductivity differences ranging between 4.32 and 7.53 mS/cm. The contents of SiO_2, Al_2O_3, and Fe_2O_3 range between 90.50% - 96.78% (Table 5.11). Horasan plaster and lime plasters with natural stone aggregates have similar electrical conductivity differences and SiO_2, Al_2O_3, and Fe_2O_3 content.

Based on the results of the electrical conductivity difference and ASTM C618-03 Standard, it was found that aggregates of mortars, horasan plasters, and lime plasters with natural stone aggregates exhibit highly active pozzolanic properties (ASTMC618-03 2003; Luxán, Madruga, and Saavedra 1989). However, the pozzolanic activities of the mortar samples were found lower than Horasan and plaster with natural stone aggregate. In both methods, the samples with the highest and lowest pozzolanic activity were consistent.

The results showed that the average values of pozzolanic activity of brick aggregates in horasan plaster were in a similar range to previous studies (Budak 2005; Böke, Akkurt, and Ipekoğlu 2004; Uğurlu 2005; Gürhan 2018). In addition, the average pozzolanic activity values of natural aggregates used in mortars and plasters were in a close range with previous studies (Table 5.12) (Budak 2005; Böke, Akkurt, and Ipekoğlu 2004; Çizer, Böke, and İpekoğlu 2004; Işık 2022; Oğuz, Türker, and Koçkal 2015; Oğuz 2013; Solak 2016; Stefanidou et al. 2014).

Table 5.12. Recent studies investigated the pozzolanic activities of fine aggregates and the hydraulicity of binders

Location - Referances		Period of Samples	Sample Types	Pozzolanic Activities of Fine Aggregates		Hydraulicity of Binders
				ΔEC (mS/cm)	SiO_2+ Al_2O_3+ Fe_2O_3(%)	$\frac{CO_2}{H_2O}$
Budak 2005	Çukur Hamam - Manisa	14th century	Mortar	2.71 - 5.67	84 - 92*	4.76 - 7.31
			Plaster (H)	7.47- 7.54	-	5.94
	Hacet Mescidi - Manisa		Mortar	1.71 - 7.23	91*	5.08 - 5.68
Böke, Akkurt, and Ipekoğlu 2004	Ördekli Bath - Bursa	14th century	Plaster (H)	avg. 8	88.08 - 95.48*	-
	Beylerbeyi Bath - Edirne	15th century	Mortar	7	92.47*	-
			Plaster (H)	avg. 6.1	78.22*	-
	Saray Bath - Edirne		Mortar	8	92.4*	-
			Plaster (H)	avg. 4.4	81.56 - 95.34*	-
Çizer, Böke, and İpekoğlu 2004; Uğurlu 2005	Düzce Bath - Urla, İzmir	15th century	Mortar (Çizer et al. 2004)	1.3 - 6.2	91.9 - 97.9*	2.5 - 6
			Plaster (H_L) (Uğurlu 2005)	3.1 - 7.8 _ -	90 - 94.4 _ 4 - 13.4*	3.21-9.01_12.51-20.2
	Herekzade Bath - Urla, İzmir		Mortar (Çizer et al. 2004)	6.3 - 6.2	96.3 - 98.2*	4.5 - 6.5
			Plaster (H_L) (Uğurlu 2005)	4.3 - 7.3 _ -	93.5 - 96.8 9*	0.64-2.91_12.5
	Kamanlı Bath - Urla, İzmir		Mortar (Çizer et al. 2004)	1.3 - 6.0	94.4 - 97.9*	8.5 - 10.5
			Plaster (H_L) (Uğurlu 2005)	1.6 - 6.5 _ -	89.9 - 94.3 _ 5.8 - 10.5*	1.12-8.39_11.99-12.8
Işık 2022	Kadıkalesi Anaia (K) - Kuşadası	13th - 14th c.	Mortar	3.8	91.81	8.4
	Ayasuluk Hill (A) - Selçuk, Izmir	5th- 6th c.	Mortar	6.13	87.49	3.2 - 6.2
Taşdibi Andriake Port- Antalya (Oğuz, Türker, and Koçkal 2015; Oğuz 2013)		13th century	Mortar	0.4	21*	14
Solak 2016	Kızıl Han - Muğla	14th century	Mortar	0.5 - 0.7	51.8 - 20.9*	4.51 - 4.65
	Karapaşa Madrasah - Muğla		Mortar	0.6 - 0.7	20.2 - 28.9*	6.02 - 6.31
	Yelli Mosque - Muğla		Mortar	0.4	38.6*	4.81 - 5.16
Stefanidou et al. 2014	Byzantine Bath - Greece	13th - 14th c.	Mortar	-	44 - 51.4	2.88 - 3.08
	Pazar Bath - Greece	16th c.	Mortar	-	22.15 - 33.2	-
Eski Bath - Efeler, Aydın (Gürhan 2018)		15th - 16th c.	Plaster (H_L)	6.61 - 7.8 _ 6.95 - 7.09	88.57 - 95.33 _ -*	1.15 - 4.93 _ 2.47 - 8.09

*: Calculated by the author based on data in the publication

5.5. Chemical, Mineralogical Compositions and Hydraulic Properties of Binders

The binder is defined as a fine mortar and plaster matrix consisting of calcium carbonate (CaCO3) and small grain-size aggregates (Bakolas et al. 1995). The chemical compositions of binders in mortars and plasters were specified via SEM-EDS. Mineralogical compositions of the binders were determined by XRD analysis. Thermogravimetric analysis (TGA) was used to determine the hydraulic properties of mortars and plasters.

5.5.1. Characteristics of Binders with Natural Aggregates

The results of SEM-EDS analysis indicated that binders in the mortars with natural aggregates from the İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath were mainly composed of large amounts of CaO (60.64 – 71.28%), moderate amounts of SiO_2 (15.04 – 21.13 %) and smaller amounts of Al_2O_3 (4.64 – 6.71 %), MgO (4.01 – 6.06%), Fe_2O_3 (2.10 – 3.12 %), K_2O (1.38 – 2.44 %), Na_2O (0.39–2.20%), and TiO_2 (0.1 – 0.27 %) (Table 5.15).

Binders of lime plasters with natural stone aggregates from studied bath buildings were comprised of mostly large amounts of CaO (41.42 – 66.81 %), moderate amounts of SiO_2 (18.6 – 37.03 %), and smaller amounts of Al_2O_3 (4.85 – 8.96 %), MgO (2.40 – 9.58 %), Fe_2O_3 (1.62 – 3.31 %), K_2O (0.48 – 3.30 %), Na_2O (0.25 – 1.20 %), and TiO_2 (0.14 – 0.37 %) (Table 5.15).

Binders in the lime plaster samples of baths mainly consisted of large amounts of CaO (79.66 – 88.68%), moderate amounts of SiO_2 (3.61 – 9.02 %), and smaller amounts of MgO (2.31 – 6.75 %), Al_2O_3 (1.19 – 4.69 %), Fe_2O_3 (0.35 – 5.03 %), K_2O (0.24 – 0.5%), Na_2O (0.14 – 0.98 %), and TiO_2 (0.04 – 0.11%) (Table 5.15). This indicates that a substantial quantity of pure lime was utilized in the production of lime plasters.

The binders in mortars from İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath consisted of calcite ($CaCO_3$), quartz, clinochlore, dolomite and muscovite minerals

(Figure 5.13). In previous studies mainly calcite, and quartz minerals were found, additionally albite, dolomite, hornblende, and plagioclase feldspar minerals were detected on the XRD patterns (Budak 2005; Işık 2022; Solak 2016; Oğuz, Türker, and Koçkal 2015) (Table 5.13). Calcite was derived from carbonated lime, whereas quartz, albite, muscovite, clinochore, dolomite, hornblende, and plagioclase feldspar were from aggregates.

Binder of lime plaster with natural stone aggregates was mainly composed of calcite, and quartz minerals at studied bath buildings. The clinochlore minerals were found in all samples except KHUP2, additionally, muscovite minerals were found in all samples except IHLP2 on their XRD patterns (Figure 5.14). On the other hand, the XRD pattern of lime plasters mainly consists of calcite minerals (Figure 5.15).

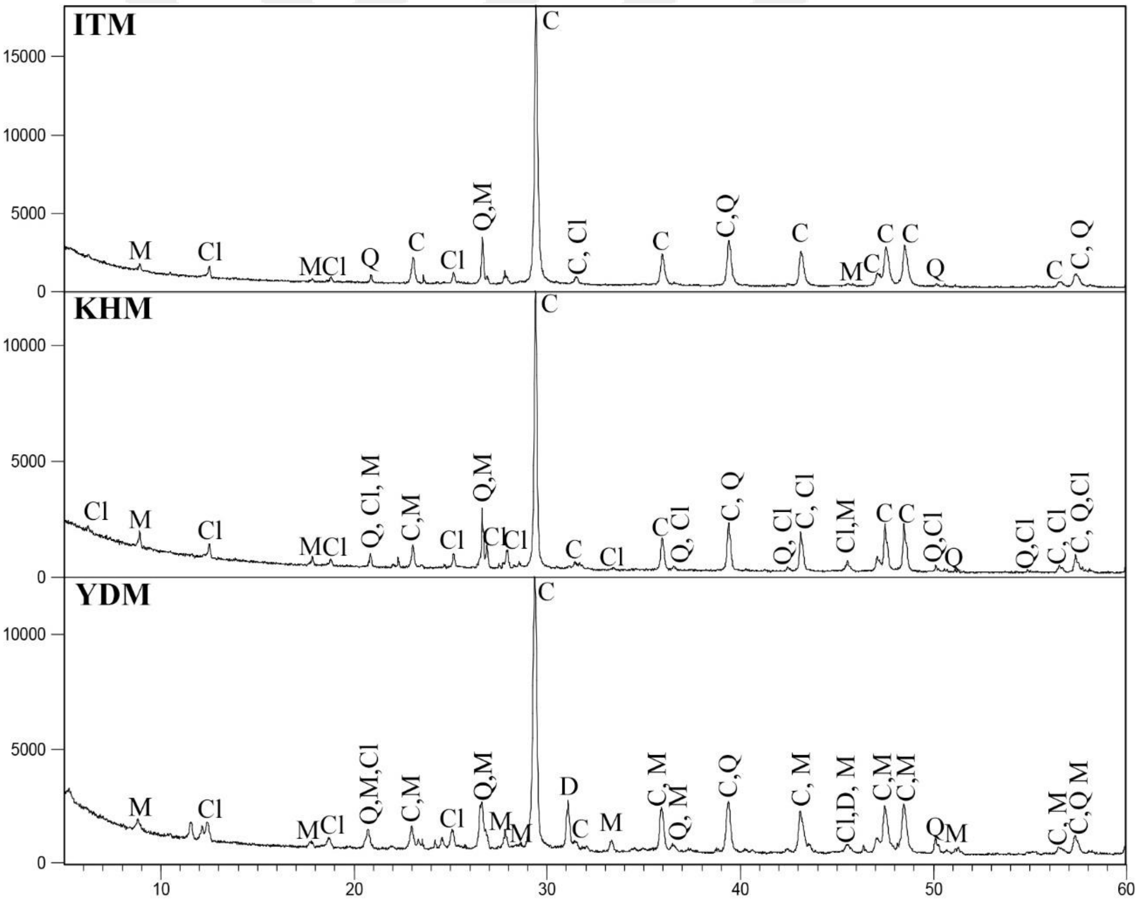


Figure 5. 13. XRD patterns of binders in mortar with natural aggregates from İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath (C: Calcite D: Dolomite Q: Quartz Cl: Clinochlore M: Muscovite)

Table 5.13. The mineralogical composition of binders from mortar in previous studies

Locations - References		**C**	**Q**	**Al**	**D**	**Ho**	**Pl**
Budak 2005	Çukur Bath	+	+	+			
	Hacet Mescidi	+	+	+			
Işık 2022	Ayasuluk Hill	+		+		+	
	Kadıkalesi Anaia	+	+	+			
Solak 2016	Kızıl Han	+	+		+		+
	Karapaşa Madrasah	+	+		+		+
	Yelli Mosque	+	+		+		+
Oğuz, Türker, and Koçkal 2015	Taşdibi Andriake Port	+	+		+		

C: Calcite Q: Quartz, Al: Albite, Ho: Hornblende, D: Dolomite, Pl: Plagioclase Feldspar

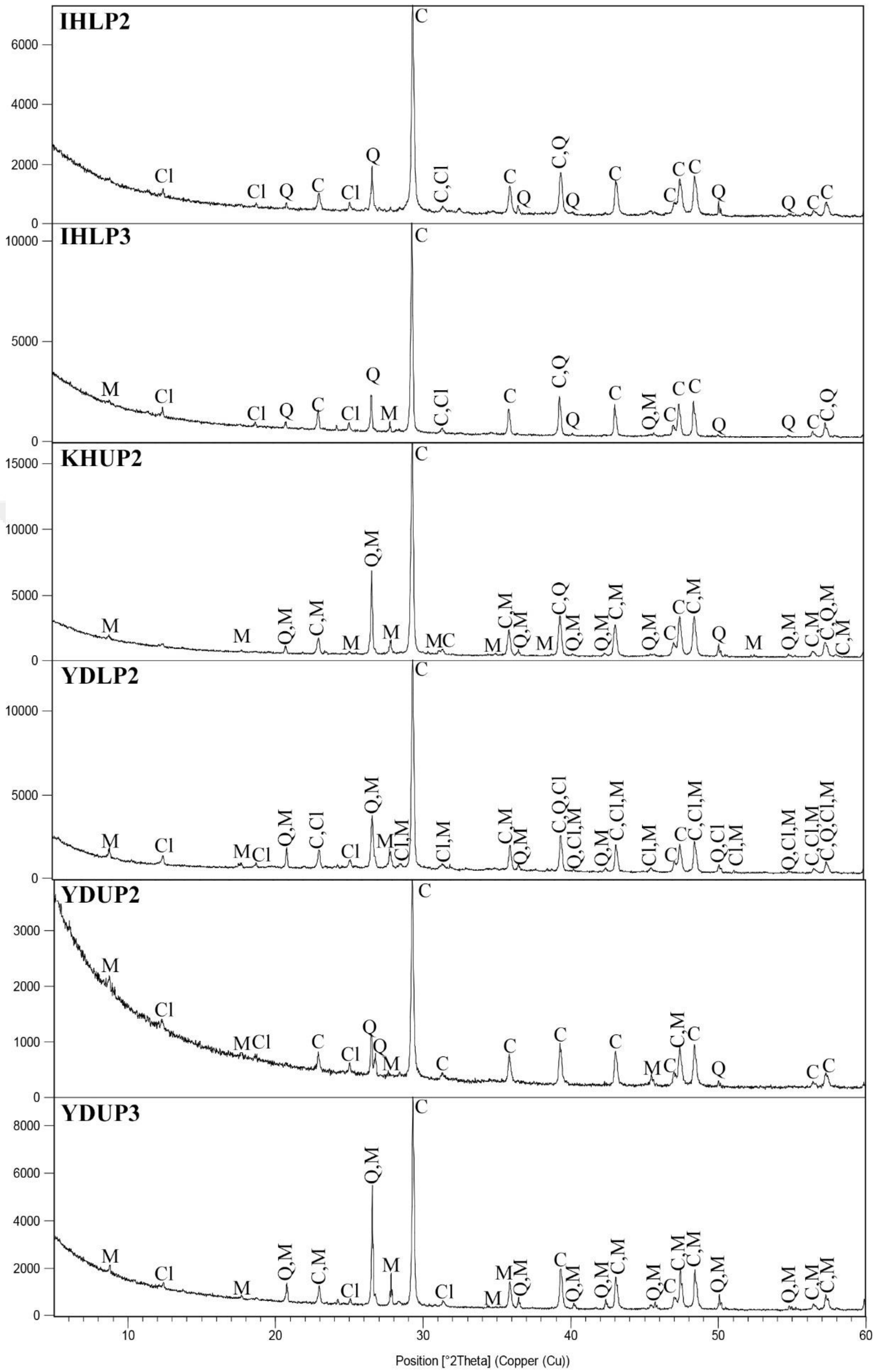


Figure 5. 14. XRD patterns of binder in plaster with natural stone aggregates from İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath (C: Calcite Q: Quartz Cl: Clinochlore M: Muscovite)

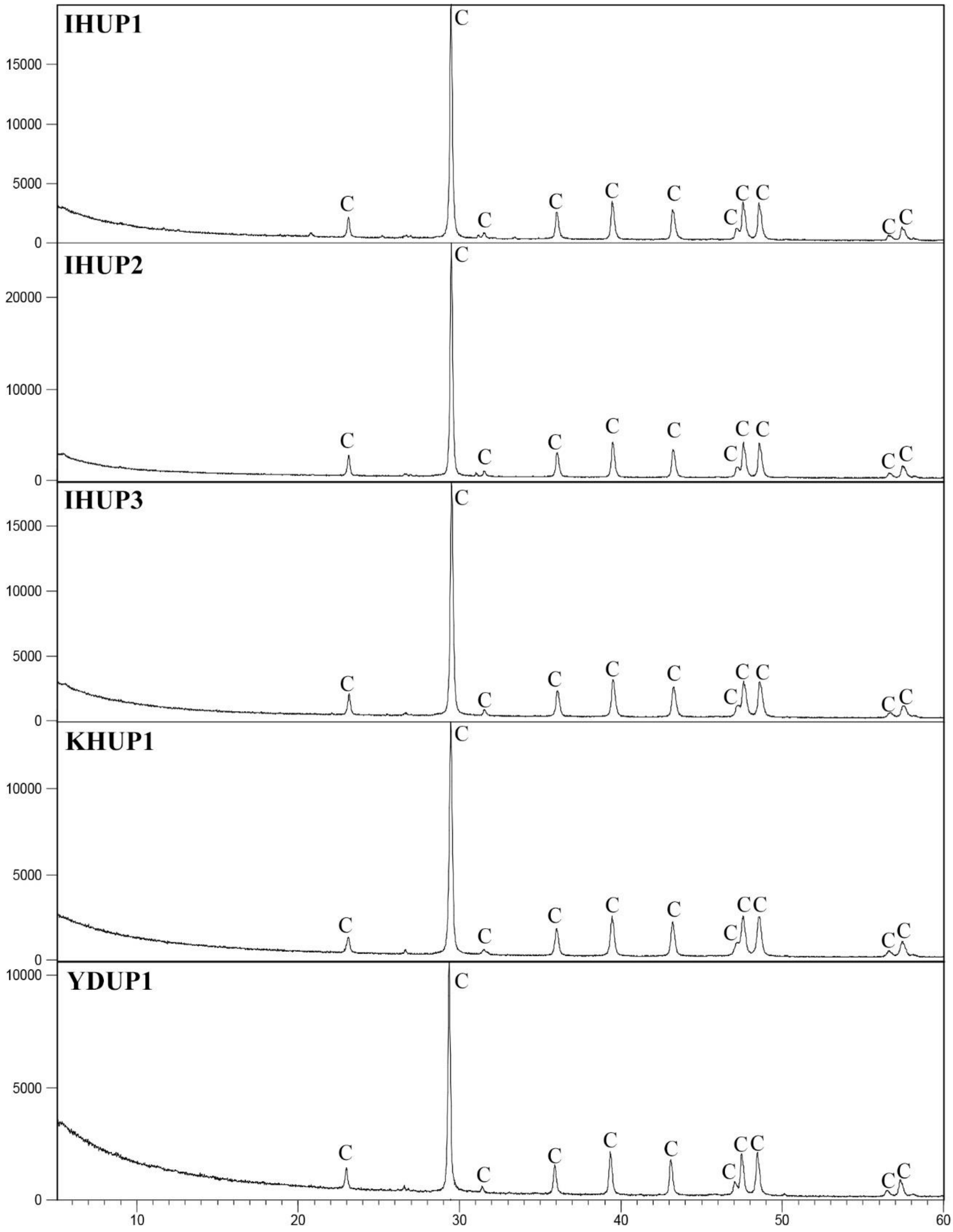


Figure 5. 15. XRD patterns of binder in lime plaster from İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath (C: Calcite)

5.5.2. Characteristics of Binders with Brick Aggregates

Horasan plaster used in İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath were mainly comprised of large amounts of CaO (39.67 – 66.99%), moderate amounts of SiO_2 (16.66 – 32.54 %), and smaller amounts of Al_2O_3 (7.59 – 11.76 %), MgO (3.58 – 6.66 %), Fe_2O_3 (2.96 – 5.62 %), K_2O (1.27 – 2.48 %), Na_2O (0.31 – 0.65 %), and TiO_2 (0.29 – 0.37%) (Table 5.15). The calcium oxide (CaO) was obtained from the carbonated lime, and the silica (SiO_2) and aluminum oxide (Al_2O_3) were sourced from the brick powder.

The mineralogical compositions of binders in horasan plasters with brick aggregates from İsa bey Bath, Kale Altı Bath, and Yahşi Bay Bath had only calcite and quartz minerals on their XRD patterns (Figure 5.16). The calcite was sourced from carbonated lime, while the quartz minerals were obtained from brick powder. Previous studies revealed that the binders of plaster with brick aggregates mainly consist of calcite, quartz, and albite minerals (Table 5.14) (Işık 2022; Uğurlu 2005; Gürhan 2018).

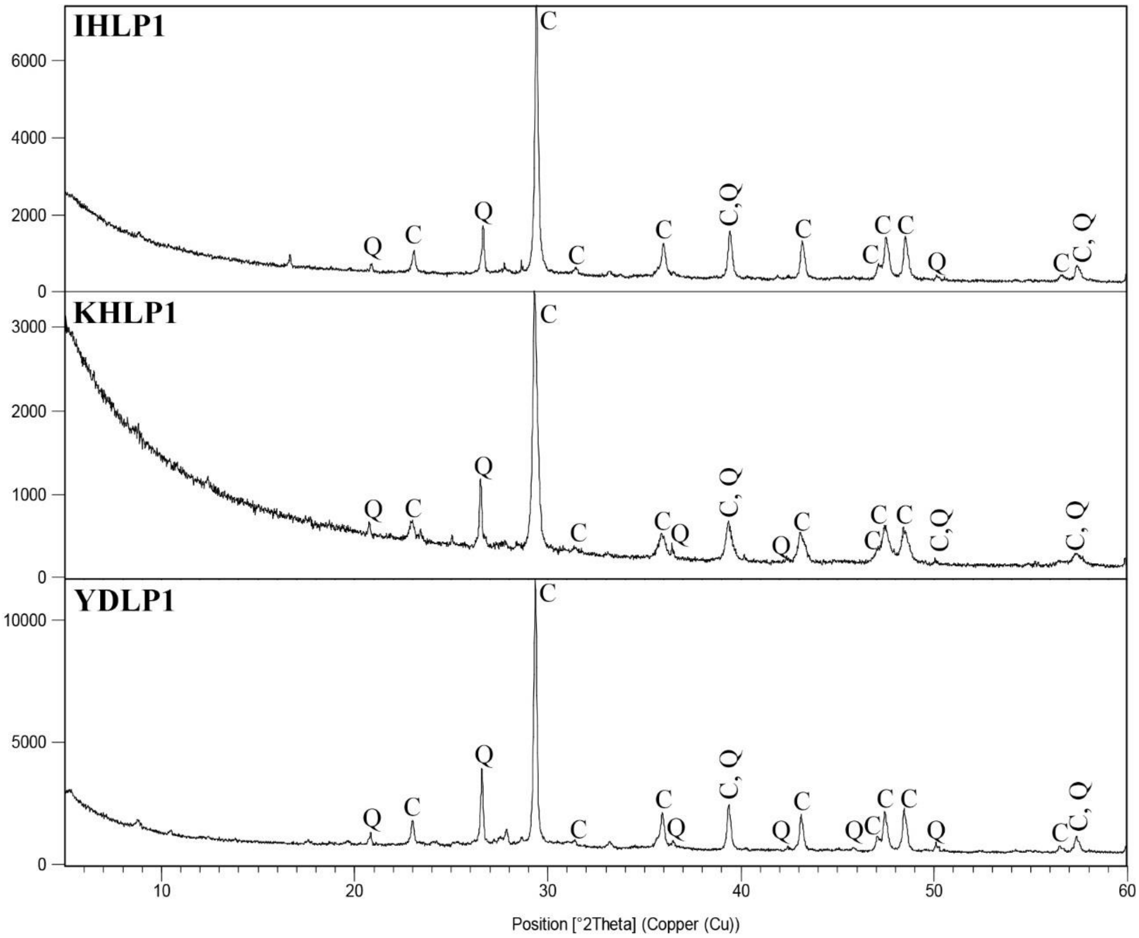


Figure 5. 16. XRD patterns of binders in horasan plaster from İsa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath (C: Calcite Q: Quartz)

Table 5.14. The mineralogical composition of binders from plaster with brick aggregates in previous studies

Locations - References		C	Q	Al	M	Pf	H	V
Kadıkalesi Anaia (Işık 2022)		+	+	+	+			
Uğurlu 2005	Düzce Bath	+	+	+				
	Herekzade Bath	+	+	+				
	Kamanlı Bath	+	+	+				
Eski Bath (Gürhan 2018)		+	+	+	+	+	+	+

C: Calcite Q: Quartz Al: Albite M: Muscovite Pf: Potassium Feldspar H: Hematite V: Vaterite

Table 5.15. Chemical Compositions of the binders in Isa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath

Name	Sample	Sample Types	Aggregate Types	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	Fe_2O_3
İsa Bey Bath	**ITM**	Mortar	Natural	0.39	5.66	4.64	15.04	0.62	71.28	0.10	2.26
	IHLP1	Horasan P.	Brick	0.31	4.89	10.61	23.64	1.27	54.57	0.34	4.37
	IHLP2	Nat.S.Agg P.	Natural	0.25	4.85	6.97	36.53	0.48	48.96	0.25	1.71
	IHLP3	Nat.S.Agg P.	Natural	0.35	5.49	8.26	34.42	0.66	49.07	0.14	1.62
	IHUP1	Lime P.	-	0.21	5.28	1.19	9.02	0.27	83.57	0.11	0.35
	IHUP2	Lime P.	Natural	0.26	3.82	4.69	7.09	0.31	83.01	0.08	0.73
	IHUP3	Lime P.	Natural	0.17	2.62	2.04	4.59	0.24	85.26	0.04	5.03
Kale Altı Bath	**KHM**	Mortar	Natural	2.20	4.01	6.24	19.36	1.56	64.31	0.23	2.10
	KHLP1	Horasan P.	Brick	0.65	3.58	7.59	16.66	1.28	66.99	0.29	2.96
	KHLP2	Nat.S.Agg P.	Natural	***: Can not be determined due to insufficient sample size*							
	KHUP1	Lime P.	Natural	0.98	2.31	3.46	3.61	0.50	88.68	0.11	0.36
	KHUP2	Nat.S.Agg P.	Natural	0.68	4.11	5.65	18.60	1.45	66.81	0.27	2.42
Yahşi Bey Bath	**YDM**	Mortar	Natural	0.68	6.06	6.71	21.13	1.39	60.64	0.27	3.12
	YDLP1	Horasan P.	Brick	0.57	6.66	11.76	32.54	2.48	39.67	0.37	5.62
	YDLP2	Nat.S.Agg P.	Natural	1.20	4.65	8.96	37.03	3.30	41.42	0.25	3.19
	YDUP1	Lime P.	-	0.14	6.75	3.27	8.49	0.40	79.66	0.05	1.25
	YDUP2	Nat.S.Agg P.	Natural	0.36	9.58	7.77	26.48	1.56	50.57	0.37	3.31
	YDUP3	Nat.S.Agg P.	Natural	0.74	2.40	4.85	28.43	0.76	60.85	0.19	1.76

5.5.3. Hydraulic Properties of Binders

Thermogravimetric analysis (TGA) was used to determine the hydraulic properties of mortars and plasters. Weight loss between 200-600°C indicated water release (H_2O), while loss between 600-900°C indicated CO_2 release. The CO_2/H_2O ratio between 1-10 indicated the hydraulic character of the samples (Bakolas et al. 1998; Antonia Moropoulou, Bakolas, and Bisbikou 2000).

The hydraulic indices of the binders were determined using Boynton's formula in addition to the TGA analysis. The hydraulic index (H.I.) value greater than 0.1 indicates hydraulic properties in the material (Eckel 2005; Boynton 1980).

The CO_2/H_2O ratios ranged from 4.95 to 6.99 for mortars, from 3.80 to 6.77 for horasan plasters, from 1.55 to 13.24 for lime plasters with natural stone aggregates, and from 7.35 to 12.08 for lime plasters (Table 5.7). H.I. values of the binders were in the ranges of 0.3–0.5 for mortars, 0.4–1.1 for horasan plasters, 0.6–1.1 for lime plasters with natural stone aggregates, and 0.1 for lime plasters (Table 5.16).

The binders of mortar and horasan plaster samples exhibited hydraulic properties, while the binders of lime plasters are considered non-hydraulic. Lime plasters with natural stone aggregate samples exhibited hydraulic properties on both levels but the lower-level plaster samples had higher hydraulic properties. Both methods mostly gave the same results for the hydraulic properties of the samples except lime plasters IHUP1, KHUP1, YDUP1, and plaster with natural stone aggregate YDUP3. According to TGA results these lime plasters exhibited hydraulic character, but their hydraulic properties were low, and their hydraulic index showed that they were non-hydraulic.

The hydraulic properties could be due to the pozzolanic properties of the aggregates used (Navrátilová and Rovnaníková 2016; Böke, Akkurt, and Ipekoğlu 2004). The average hydraulic properties and pozzolanic activities of horasan plasters and plaster with natural stone aggregate were found in close ranges. It was observed that brick and natural aggregates used in the plasters could provide similar hydraulic and pozzolanic properties to the materials.

The hydraulic properties are due to their pozzolanic properties of aggregates; however, there is no simple correlation relationship was found between the pozzolanic activity values of aggregates and the hydraulic properties of mortars and plaster with natural stone aggregates. The heterogeneous structure of mortars and plasters, and the lime/aggregate ratios can influence the hydraulic properties, so a simple correlation could not be stated (Arizzi and Cultrone 2021).

In previous studies, it has been observed that mortars and plasters with natural aggregates exhibit hydraulic properties and these hydraulic properties are caused using pozzolanic aggregates. (Budak 2005; Çizer, Böke, and İpekoğlu 2004; Işık 2022; Solak 2016; Stefanidou et al. 2014; Uğurlu 2005; Gürhan 2018) (Table 5.12).

Table 5.16. Hydraulic properties of the binders in Isa Bey Bath, Kale Altı Bath and Yahşi Bey Bath

Name	Sample	Sample Types	Aggregate type	200-600°C (%) H_2O	600-900°C (%) CO_2	$\frac{CO_2}{H_2O}$	$SiO_2 + Al_2O_3 + Fe_2O_3$ (%)	CaO + MgO (%)	H. I
İsa Bey Bath	**ITM**	Mortar	Natural	4.52	31.63	6.99	21.94	76.94	0.3
	IHLP1	Horasan P.	Brick	3.29	22.31	6.77	38.62	59.46	0.6
	IHLP2	Nat. Agg P.	Natural	10.52	16.36	1.55	45.22	53.81	0.8
	IHLP3	Nat. Agg P.	Natural	4.89	22.96	4.70	43.92	54.55	0.8
	IHUP1	Lime P.	-	4.51	36.97	8.20	10.56	88.85	0.1
	IHUP2	Lime P.	Natural	3.06	36.90	12.08	12.51	86.84	0.1
	IHUP3	Lime P.	Natural	3.72	39.33	10.57	11.66	68.31	0.1
Kale Altı Bath	**KHM**	Mortar	Natural	5.40	26.75	4.95	27.70	68.31	0.4
	KHLP1	Horasan P.	Brick	4.90	27.11	5.53	27.21	70.57	0.4
	KHLP2	Nat. Agg P.	Natural	***: Can not be determined due to insufficient sample size*					
	KHUP1	Lime P.	Natural	4.09	37.61	9.19	7.43	90.99	0.1
	KHUP2	Nat. Agg P.	Natural	4.29	30.04	7.01	26.67	70.92	0.4
Yahşi Bey Bath	**YDM**	Mortar	Natural	4.12	21.53	5.22	30.96	66.70	0.5
	YDLP1	Horasan P.	Brick	4.76	18.09	3.80	49.92	46.33	1.1
	YDLP2	Nat.Agg P.	Natural	6.74	15.40	2.29	49.18	46.08	1.1
	YDUP1	Lime P.	-	4.82	35.44	7.35	12.98	86.19	0.1
	YDUP2	Nat. Agg P.	Natural	4.47	24.74	5.54	37.56	60.15	0.6
	YDUP3	Nat. Agg P.	Natural	2.10	27.76	13.24	35.05	63.25	0.6

CHAPTER 6

CONCLUSION

In this study, Isa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath, the rare buildings from the 14^{th}-15^{th} century Principalities Period that have survived to the present day by preserving their authentic material properties, were investigated.

The lime mortars used in all studied bath buildings consisted of natural aggregates. The interior walls of the baths had two distinct layers of plaster of different colors on the upper and lower levels. The lower-level plasters typically consisted of one or two layers of plaster with natural stone aggregates and one layer of horasan plaster with brick aggregates. The upper-level plasters consist of two or three layers; the first layer is lime plaster, while the other layers consist of plaster with natural stone aggregate or lime plaster.

The average density and porosity values of the Horasan plasters and plaster with natural stone aggregates of the baths were found 1.71 g/cm³ to 1.48 g/cm³ and 26.83% to 30.70%, respectively. The low porosity values indicate that the horasan layers provide a waterproof surface, preventing water from reaching the structure of the baths.

The lime/aggregate ratios of Horasan plaster in the baths were in the range of 1.21 to 2.6 which were almost similar to each other. According to the lime/aggregate ratios of plaster with natural stone aggregate, there was a significant difference in the lime content between the upper (avg. 2.05) and lower-level (avg. 0.82) lime plasters with natural stone aggregates. Additionally, it was determined that mortars with natural aggregates exhibited a higher aggregate content, while plaster with natural stone aggregate exhibited a higher lime content and a higher lime/aggregate ratio. The lime plasters had high lime/aggregate ratios in the range of 10.64 – 99, which indicated that a high amount of lime was used in their production. In addition, it was revealed that there was a wide range of particle sizes for both the natural and brick aggregates.

The studied bath buildings revealed that the mortar, and lime plaster with natural stone aggregate samples of all baths exhibit similar chemical compositions and they were found to be rich in silica and alumina, but poor in carbonate and alkaline phases.

Mineralogical compositions of fine natural aggregates in mortars consisted of albite, clinochlore, muscovite, orthoclase, quartz, phillipsite, oligoclase, and hornblende minerals; likewise, lime plaster with natural stone aggregates mainly consisted of quartz, albite, graphite, clinochlore, muscovite, and phillipsite minerals. The hornblende and phillipsite minerals found in natural aggregates may have originated from volcanic units.

The binder is defined as a mortar and plaster matrix composed of calcium carbonate (CaCO3) and fine aggregates. The binders in the lime mortars and plasters with natural aggregates were mainly composed of large amounts of CaO, moderate amounts of SiO_2, and smaller amounts of Al_2O_3, MgO, Fe_2O_3, K_2O, Na_2O, and TiO_2. The higher CaO content of lime plaster than mortar and plaster with natural stone aggregate indicates that a significant amount of pure lime was used in the production of lime plaster.

The binders in mortars with natural aggregates consisted of calcite, quartz, clinochlore, dolomite, and muscovite minerals; plaster with natural stone aggregates was composed of calcite, quartz, clinochlore, and muscovite. The binders of lime plasters mainly consist of calcite minerals. Calcite is derived mainly from lime, while quartz, dolomite, clinochlore, and muscovite are derived from aggregates.

Brick aggregates in Horasan plaster were comprised of mostly large amounts of SiO_2, moderate amounts of Al_2O_3, and smaller amounts of Fe_2O_3, MgO, K_2O, CaO, TiO_2, and Na_2O. They had albite, clinochlore, dolomite, hematite, muscovite, and quartz on their XRD patterns. In the XRD pattern, the absence of wollastonite minerals and the presence of hematite and dolomite indicate that the brick aggregates have a firing temperature around or below 850 ºC.

The binders of Horasan plasters with brick aggregates were mainly comprised of large amounts of CaO, moderate amounts of SiO_2, and smaller amounts of Al_2O_3, MgO, Fe_2O_3, K_2O, Na_2O, and TiO_2. The calcium oxide (CaO) was obtained from the carbonated lime, and the silica (SiO_2) and aluminum oxide (Al_2O_3) were sourced from the brick powder. They were mostly composed of calcite which originated from carbonated lime and quartz minerals that were obtained from brick powder.

The aggregates of mortars, horasan plasters, and lime plasters with natural stone aggregates exhibit highly active pozzolanic properties. The binders of lime mortar, plaster with natural stone aggregate, and horasan plaster samples exhibited hydraulic properties, while the binders of lime plasters are considered non-hydraulic. The hydraulic properties are due to their pozzolanic properties of aggregates; however, the heterogeneous structure of mortars and plasters, and the lime/aggregate ratios can influence the hydraulic properties.

Consequently, this study revealed that despite variations in the number of layers and plaster types, the average basic physical properties of upper and lower-level plasters are similar to each other in studied bath buildings. The chemical and mineralogical composition and hydraulic properties of mortar and plaster types vary according to the aggregate type. The finding of suitable raw material resources for hydraulic mortar and plaster production and the use of mortars and plasters with similar properties in different baths show that local knowledge of raw material resources and lime mortars and plasters production techniques was used for years in these historical bath structures. In addition, this study shows that the mortar and plaster of the baths from the Principalities period in Selçuk were similar to other nearby examples built in recent times.

The characterization of the mortars and plasters used in the İsa Bey Bath, the Kale Altı Bath, and the Yahşi Bey Bath has provided an understanding and documentation of the authentic material properties, manufacturing, and craftsmanship techniques used during the 14^{th}-15^{th} century Principalities period. The lime mortars and plasters to be used in future conservation projects for Isa Bey Bath, Kale Altı Bath, and Yahşi Bey Bath should be compatible with the original mortar and plaster properties determined in this study. Considering the basic physical, chemical, and mineralogical properties, raw material compositions, and hydraulic properties, materials with high porosity, hydraulic features, and pozzolanic aggregates should be preferred. Future research should include detailed chemical analyses and regional geology to identify potential geochemical sources of fine natural aggregates. Research can be carried out in the Seferihisar-Doğanbey volcanic areas to examine the mineral resources thought to originate from volcanic units.

REFERENCES

Adam, J. P. 2001. *Roman Building: Materials and Techniques*. London: Routledge Publishing. https://www.routledge.com/Roman-Building-Materials-andTechniques/Adam/p/book/9780415208666.

Ahunbay, M. 2006. "Bizans Yapım Teknikleri." In *Geçmişten Geleceğe Anadolu'da Malzeme ve Mimarlık Sempozyumu, 4-5 Temmuz 2005*, 68–78. İstanbul: Çizgi Basım Yayın.

Akın, H. 1968. "Aydınoğulları Tarihi Hakkında Bir Araştırma." Doctorate Thesis, Ankara: Dil ve Tarih Coğrafya Fakültesi .

Arizzi, A., and G. Cultrone. 2021. "Mortars and Plasters-How to Characterise Hydraulic Mortars." *Archaeological and Anthropological Sciences* 13 (144): 2–22. https://doi.org/10.1007/s12520-021-01404-2/Published.

Arıkan, Z. 1990. "XIV-XVI. Yüzyıllarda Ayasuluğ." *Belleten* 209:121–77.

Arıoğlu, N., and S. Acun. 2006. "A Research about a Method for Restoration of Traditional Lime Mortars and Plasters: A Staging System Approach." *Building and Environment* 41 (9): 1223–30. https://doi.org/10.1016/J.BUILDENV.2005.05.015.

Ashurst, J., and N. Ashurst. 1990. *Mortars, Plasters and Renders, Practical Building Conservation, English Heritage Technical Handbook*. Vol. 3. Gower Technical Press. https://www.scrbd.com/document/61516068/Practcal-Buldng-Conservaton.

ASTMC618-03. 2003. "Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete." *ASTM International*. https://doi.org/https://doi.org/10.1520/C0618-03.

Bakolas, A., G. Biscontin, A. Moropoulou, and E. Zendri. 1995. "Characterization of the Lumps in the Mortars of Historic Masonry." *Thermochimica Acta* 269 (270): 809–16. https://doi.org/https://doi.org/10.1016/0040-6031(95)02573-1.

Bakolas, A., G. Biscontin, A. Moropoulou, and E. Zendri. 1998. "Characterization of Structural Byzantine Mortars by Thermogravimetric Analysis." *Thermochimica Acta* 321 (1–2): 151–60. https://doi.org/10.1016/S0040-6031(98)00454-7.

Balen, K. V., and D. V. Gemert. 1994. "Modelling Lime Mortar Carbonation." *Materials and Structures* 27 (7): 393–98. https://doi.org/10.1007/BF02473442.

Balen, K. V. 2003. "Understanding the Lime Cycle and Its Influence on Historical Construction Practice." In *First International Congress on Construction History*, edited by S Huerta, 2042–55. Madrid: Sociedad Española de Historia de la Construcción.

Barca, D., D. Miriello, A. Pecci, L. Barba, A. Ortiz, L. R. Manzanilla, J. Blancas, and G. M. Crisci. 2013. "Provenance of Glass Shards in Archaeological Lime Plasters by LA-ICP-MS: Implications for the Ancient Routes from the Gulf of Mexico to Teotihuacan in Central Mexico." *Journal of Archaeological Science* 40 (11). https://doi.org/10.1016/j.jas.2013.05.016.

Başgelen, N. 1993. *Çağlar Boyunca Anadolu'da Duvar*. İstanbul: Arkeoloji ve Sanat Yayınları. https://www.nadirkitap.com/caglar-boyunca-anadolu-da-duvar-basgelen-nezih-kitap27568331.html.

Battuta, İ. 2004. *İbn Battûta Seyahatnamesi*. Edited by Sait Aykut. İstanbul: Yapı Kredi Yayınları.

Baykara, T. 1990. *Aydınoğlu Gazi Umur Paşa*. Ankara: Başbakanlık Basımevi.

Bellibaş, E. S. 2015. "Efes Artemisionu'ndaki Anonim Türbe." Master, İstanbul: Fen Bilimleri Enstitüsü.

Bellibaş, E. S., S. Ladstätter, and M. Kürüm. 2013. "Sığla Türbesi (İzmir - Selçuk) Ve Restorasyon Yaklaşımları." In *Uluslararası Batı Anadolu Beylikleri Tarih, Kültür ve Medeniyeti Sempozyumu-I, Aydınoğulları Tarihi*, edited by Mehmet Şeker, Cüneyt Kanat, and Mehmet Ersan, 275–307. Ankara: Türk Tarih Kurumu.

Binici, H., J. Arocena, S. Kapur, O. Aksogan, and H. Kaplan. 2010. "Investigation of the Physico-Chemical and Microscopic Properties of Ottoman Mortars from Erzurum (Turkey)." *Construction and Building Materials* 24 (10): 1995–2002. https://doi.org/10.1016/J.CONBUILDMAT.2010.03.013.

Böke, H., S. Akkurt, and B. Ipekoğlu. 2004. “Investigation of the Pozzolanic Properties of Bricks Used in Horasan Mortars and Plasters in Historic Buildings.” *Key Engineering Materials* 264–268 (III): 2399–2402. https://doi.org/10.4028/www.scientific.net/kem.264-268.2399.

Böke, H., S. Akkurt, B. Ipekoğlu, and Elif Uğurlu. 2006. “Investigation of the Pozzolanic Properties of Bricks Used in Horasan Mortars and Plasters in Historic Buildings.” *Cement and Concrete Research* 36 (March):1115–22. https://doi.org/10.4028/www.scientific.net/kem.264-268.2399.

Borrelli, E. 1999. “Binders - ARC Laboratory Handbook.” In. Vol. 4. Rome: ICCROM. S.p.A Pres. https://www.iccrom.org/sites/default/files/publications/2021-04/iccrom_14_arclabhandbook00_en.pdf.

Boynton, R. S. 1980. *Chemistry and Technology of Lime and Limestone*. 2nd ed. John Wiley & Sons.

Buch, W. 1982. “14.- 15. Yüzyılda Kudüs’e Giden Alman Hacılarının Türkiye İzlenimleri.” *Belleten* 46 (183): 509–34. https://doi.org/10.37879/BELLETEN.1982.509.

Budak, M. 2005. “Characterization of Selected Beylikler Period Mortars And Processing Of New Intervention Mortars For Restoration Purposes.” Master of Science Thesis, İzmir: İzmir Institute of Technology.

Büyükkolancı, M. 1998. “Apasas, Eski Efes ve Ayasuluk.” In *Birinci Uluslararası Geçmişten Günümüze Selçuk Sempozyumu*, 1st ed., 31–39. İzmir, Selçuk: Selçuk Belediyesi Kültür Yayınları.

Büyükkolancı, M. 2023. “Ayasuluk’ta Yeni Keşfedilen İki Yapı: Zaviyeli (Tabhaneli) Camiler.” *Türkiye Bilimler Akademisi Arkeoloji Dergisi*, June. https://doi.org/10.22520/tubaar.1275350.

Büyükkolancı, M., S Erdemgil, and F. Heuber. 1997. *Ephesos; Gebaute Geschichte Verlag Phillipp Von Zabern*. Bonn, Germany: Antiquariat Bücheretage Press.

Cardiano, P., S. Ioppolo, C. D. Stefano, A. Pettignano, S. Sergi, and P. Piraino. 2004. “Study and Characterization of the Ancient Bricks of Monastery of ‘“San Filippo Di Fragala”’ in Frazzano (Sicily).” *Analytica Chimica Acta* 1 (519): 103–11. https://doi.org/10.1016/J.ACA.2004.05.042.

Çizer, Ö., H. Böke, and B. İpekoğlu. 2004. “Bazı Osmanlı Dönemi Hamam Yapılarının Kubbe ve Duvarlarında Kullanılan Kireç Harçların Özellikleri.” In *II. Ulusal Yapı Malzemesi Kongresi*, 1–13. İstanbul: TMMOB Mimarlar Odası Büyükkent Şubesi.

Cowper, A. D. 1998. *Lime and Lime Mortars. Lime and Lime Mortars*. 1st ed. Routledge, Building Research Establishment.

Daş, E. 1997. “Selçuk’daki Türk Hamamları.” In *Birinci Uluslararası Geçmişten Günümüze Selçuk Sempozyumu*, 385–97. İzmir,Selçuk: Selçuk Municipality.

Davey, N. 1961. “A History of Building Materials.” *Technology and Culture* 3 (3). https://doi.org/10.2307/3100831.

Eckel, E. C. 2005. *Cements, Limes and Plasters: Their Materials, Manufacture and Properties*. 1st ed. New York.: John Wiley & Sons.

Erat, B. 1997. “Anadolu’da XIV. Yüzyıl Türk Hamam Mimarisi_Birsen Erat_1997_Ankara_Doktora_sosyal Bilimler Enstitüsü_arkeoloji ve Sanat Tarihi Anabilim Dalı_ankara Üni.” Doctoral Thesis, Ankara: Ankara University.

Erdemgil, S., and M. Büyükkolancı. 1991. “1990 Yılı Efes - Ayasuluk Tepesi Prehistorik Kazısı.” In *T.C Kültür Bakanlığı Anıtlar ve Müzeler Genel Müdürlüğü XIII. Kazı Sonuçları Toplantısı : Çanakkale, 27-31 Mayıs 1991.*, 275–82. Çanakkale: Ankara Üniversitesi Basımevi.

Ertuğrul, A. 2009. “Hamam Yapıları ve Literatürü.” *Türkiye Araştırmaları Literatür Dergisi* 7 (13).

Ertuğrul, S. 1995. “Anadolu’da Aydınoğulları Dönemi Mimarisi.” Doctorate, İstanbul: Sosyal Bilimler Enstitüsü.

Esen, S., N. Tunç, S. Telatar, A. Tavukçuoğlu, E. N. Caner Saltik, and Ş. Demirci. 2004. “Manisa Çukur Hamam’ın Onarımına Yönelik Malzeme Çalışmaları.” In *2nd National Congress on Construction Materials*. İstanbul: 2nd National Congress on Construction Materials.

Evliya Çelebi. 2011. *Günümüz Türkçesiyle Evliya Çelebi Seyahatnamesi*. Edited by Seyit Ali Kahraman. 9th ed. Vol. 9. Kitap 1. Cilt. İstanbul: Yapı Kredi Yayınları.

Evren, A., and Cengiz İ. 1998. “Efes Çukuriçi ve Arvalya (Gül Hanım) Höyükleri.” In *VIII. Müze Kurtarma Kazıları Semineri 7-9 Nisan 1997 (Kuşadası)*, 111–33. Ankara: T.C. Kültür Bakanlığı Anıtlar ve Müzeler Genel Müdürlüğü.

Foss, C. 1979. *Ephesus After Antiquity A Late Antique, Byzantine and Turkish City*. Cambridge : Cambridge University Press.

Gibbons, P. 1997. “Pozzolans for Lime Mortars.” *The Conservation and Repair of Ecclesiastical Buildings*. https://www.buldingconservation.com/articles/pozzo/lime-pozzolans.htm.

Gürhan, F. 2018. “Plaster Characteristics of Historic Eski Hamam in Aydın.” Master of Science, İzmir: İzmir Institute of Technology.

Gürhan, F., E. Uğurlu Sağin, and H. Böke. 2017. “Plaster Characteristics of Aydin Old Bath.” In *Uluslararası Katılımlı 6. Tarihi Yapıların Korunması ve Güçlendirilmesi Sempozyumu*.

Haga, K., M. Shibata, M. Hironaga, S. Tanaka, and S. Nagasaki. 2002. “Silicate Anion Structural Change in Calcium Silicate Hydrate Gel on Dissolution of Hydrated Cement.” *Journal of Nuclear Science and Technology* 39 (5): 540–47. https://doi.org/10.1080/18811248.2002.9715232.

ICOMOS. 1964. “International Charter For The Conservation And Restoration Of Monuments And Sites (The Venice Charter 1964).” In *IInd International Congress of Architects and Technicians of Historic Monuments.*

ICOMOS. 1993. “THE NARA DOCUMENT ON AUTHENTICITY.”. Accessed April 16, 2023 http://whc.unesco.org/events/gt-zimbabwe/nara.htm

ICOMOS. 1999. *The Burra Charter : The Australia ICOMOS Charter for Places of Cultural Significance*. Accessed April 16, 2023 https://openarchive.icomos.org/id/eprint/2145/

İpekci, E., E. Uğurlu Sağın, and H. Böke. 2019. “Interior Plastering of Ottoman Bath Buildings.” *Case Studies in Construction Materials* 11 (December). https://doi.org/10.1016/J.CSCM.2019.E00295.

Işık, T. 2022. “Lime Mortar and Plaster Characteristics of Some Byzantine Period Buildings in Kadikalesi (Anaia) And Ayasuluk Hill.” Msc, Izmir: Izmir Institute of Technology.

Kalfazade, S. 2013. “Aydınoğlu Cami Mimarisine Genel Bir Bakış.” In *Uluslararası Batı Anadolu Beylikleri Tarih, Kültür ve Medeniyeti Sempozyumu-I, Aydınoğulları Tarihi*, edited by Mehmet Kanat, Cüneyt Ersan, Mehmet Şeker, 249–74. Ankara: Türk Tarih Kurumu.

Kılıç, A. 1998. “Selçuk (Ayasluğ) Kültür Tarihine Genel Bir Bakış.” In *Birinci Uluslararası Geçmişten Günümüze Selçuk Sempozyumu 4-6 Eylül 1997*, 3–17. İzmir: Ege Üniversitesi Basımevi.

KOERI Boğaziçi University, Kandilli Observatory and Earthquake Research Institute, Regional Earthquake-Tsunami Monitoring Center. 2017. “Tarihsel Depremler.” Boğaziçi University, Kandilli Observatory and Earthquake Research Institute, Regional Earthquake-Tsunami Monitoring Center. 2017. Accessed January 1, 2024 http://www.koeri.boun.edu.tr/sismo/2/deprem-bilgileri/tarihsel-depremler/.

KOERI Boğaziçi University, Kandilli Observatory and Earthquake Research Institute, Regional Earthquake-Tsunami Monitoring Center. 2024. “Deprem Sorgulama.” Boğaziçi University, Kandilli Observatory and Earthquake Research Institute, Regional Earthquake-Tsunami Monitoring Center. 2024. Accessed January 1, 2024 http://www.koeri.boun.edu.tr/sismo/zeqdb/.

Kolay, İ. A. 1999. *Batı Anadolu 14. Yüzyıl Beylikler Mimarisinde Yapım Teknikleri*. Ankara: Atatürk Kültür Merkezi BaĢkanlığı Yayınları.

Kozlu, H, and A. Ersen. 2011. “Characterization of Historical Mortars in Roman, Byzantine, Seljuk and Ottoman Buildings Located in Kayseri and the Design of Restoration Mortars.” *İTÜ Dergisi / Mimarlık, Planlama, Tasarım* 10 (March):125–36.

Kuban, D. 1982. *Türk ve İslam Sanatı Üzerine Denemeler*. İstanbul: Arkeoloji ve Sanat Yayınları.

Kuban, D. 2002. *Selçuklu Çağında Anadolu Sanatı*. İstanbul: Yapı Kredi Yayınları. https://kitap.ykykultur.com.tr/kitaplar/selcuklu-caginda-anadolu-sanati.

Kuban, D. 2009. *Çağlar Boyunca Türkiye Sanatının Anahatları*. İstanbul: YEM Yayınları.

Külzer, A. 2011. *Bizans Dönemi Ephesos"u Tarihine Bir Genel Bakış*. İstanbul: Ege Yayınları.

Ladstaetter, S., M. Büyükkolancı, E. Ulusan, U. Özdemir, and B. Sayar. 2015. "Unesco Nomination File." Https://Whc.Unesco.Org/Uploads/Nominations/1018rev.Pdf. 2015.

Ladstätter, S. 2011. "Bizans Döneminde Ephesos Büyük Bir Antik Kentin Tarihinde Son Sayfa." In *Bizans Döneminde Ephesos*, edited by Falko Daim and Sabine Ladstätter, 3–28. İstanbul: Ege Yayınları. https://doi.org/10.13114/mjh/20122760.

Lee, S., Y. J. Kim, and H. S. Moon. 1999. "Phase Transformation Sequence from Kaolinite to Mullite Investigated by an Energy-Filtering Transmission Electron Microscope." *Journal of the American Ceramic Society* 82 (10): 2841–48. https://doi.org/10.1111/J.1151-2916.1999.TB02165.X.

Luxán, M. P., F. Madruga, and J. Saavedra. 1989. "Rapid Evaluation of Pozzolanic Activity of Natural Products by Conductivity Measurement." *Cement and Concrete Research* 19 (1): 63–68. https://doi.org/10.1016/0008-8846(89)90066-5.

Lynch, G. 1998. "Lime Mortars for Brickwork: Traditional Practice and Modern Misconceptions—Part One." *Journal of Architectural Conservation* 4 (1): 7–20. https://doi.org/10.1080/13556207.1998.10785204.

Maitre, R. W. Le, M. Bas, B. Bonin, and S. Gibson. 2003. "Igneous Rocks. A Classification and Glossary of Terms. Recommendations of the International Union of Geological Sciences Subcommission on the Systematics of Igneous Rocks." *Cambridge University Press*, 2003. https://doi.org/https://doi.org/10.1017/S0016756803388028.

Mccarter, W. J., and D. Tran. 1996. "Monitoring Pozzolanic Activity by Direct Activation with Calcium Hydroxide." *Consrruction and Euilding Materials*. Vol. 10.

Miriello, D., A. Bloise, G. M. Crisci, C. Apollaro, and A. L. Marca. 2011a. "Characterisation of Archaeological Mortars and Plasters from Kyme (Turkey)." *Journal of Archaeological Science* 38 (4): 794–804. https://doi.org/10.1016/J.JAS.2010.11.002.

Miriello, D., A. Bloise, G. M. Crisci, C. Apollaro, and A. L. Marca. 2011b. “Characterisation of Archaeological Mortars and Plasters from Kyme (Turkey).” *Journal of Archaeological Science* 38 (4): 794–804. https://doi.org/10.1016/J.JAS.2010.11.002.

Miriello, D., A. Bloise, G. M. Crisci, R. D. Luca, B. D. Nigris, A. Martellone, M. Osanna, R. Pace, A. Pecci, and N. Ruggieri. 2018. “New Compositional Data on Ancient Mortars and Plasters from Pompeii (Campania – Southern Italy): Archaeometric Results and Considerations about Their Time Evolution.” *Materials Characterization* 146 (December):189–203. https://doi.org/10.1016/J.MATCHAR.2018.09.046.

Moropoulou, A., A. Bakolas, and K. Bisbikou. 1995. “Characterization of Ancient, Byzantine and Later Historic Mortars by Thermal and X-Ray Diffraction Techniques.” *Thermochimica Acta* 269–270 (C): 779–95. https://doi.org/10.1016/0040-6031(95)02571-5.

Moropoulou, A., A. S. Cakmak, G. Biscontin, A. Bakolas, and E. Zendri. 2002. “Advanced Byzantine Cement Based Composites Resisting Earthquake Stresses: The Crushed Brick/Lime Mortars of Justinian’s Hagia Sophia.” *Construction and Building Materials* 16 (8): 543–52. https://doi.org/10.1016/S0950-0618(02)00005-3.

Moropoulou, A., A. Bakolas, and E. Aggelakopoulou. 2001. “The Effects of Limestone Characteristics and Calcination Temperature to the Reactivity of the Quicklime.” *Cement and Concrete Research* 31 (4): 633–39. https://doi.org/10.1016/S0008-8846(00)00490-7.

Moropoulou, A., A. Bakolas, and K. Bisbikou. 2000. “Investigation of the Technology of Historic Mortars.” *Journal of Cultural Heritage* 1 (1): 45–58. https://doi.org/10.1016/S1296-2074(99)00118-1.

Navrátilová, E., and P. Rovnaníková. 2016. “Pozzolanic Properties of Brick Powders and Their Effect on the Properties of Modified Lime Mortars.” *Construction and Building Materials*. Elsevier Ltd. https://doi.org/10.1016/j.conbuildmat.2016.05.062.

Oates, J.A.H. 1998. *Lime and Limestone*. *Lime and Limestone*. Wiley. https://doi.org/10.1002/9783527612024.

Oğuz, C. 2013. “Myra Ve Limanı Andriake’de Kullanılan Özgün Malzeme Özelliklerinin Araştırılması.” Doctorate Thesis, Antalya: Akdeniz Üniversitesi Fen Bilimleri Enstitüsü.

Oğuz, C., F. Türker, and N. U. Koçkal. 2015. “Andriake Limanı’nda Roma, Bizans ve Selçuklu Dönemi Harçların Özellikleri.” *İMO Teknik Dergi*, no. 429.

Öktem, G. 2002. “Selçuk Kent Alanı İçinde Bizans, Selçuklu ve Osmanlı Yapıları Koruma ve Onarım Sorunları.” Master Thesis, İzmir: Dokuz Eylül University.

Özdoğan, A. 2007. *12000 Yıl Önce Uygarlığın Anadolu’dan Avrupa’ya Yolculuğunun Başlangıcı Neolitik Dönem*. Edited by Nezih Başgelen. İstanbul: Yapı Kredi Kültür Sanat Yayıncılık. https://www.researchgate.net/publication/291334031_Ozkaya_V_Coskun_A_Kortik_Tepe_12000_Yil_Once_Uygarligin_Anadolu’dan_Avrupa’ya_Yolculugunun_Baslangici_Neolitik_Donem_2007_86-88_143-156.

Özeren, Ö., and M. Büyükkolancı. 1977a. “Conservation Inventory of Natural and Cultural Assets İsa Bey Bey Bath Registration Document.” Selçuk, İzmir.

Özeren, Ö., and M. Büyükkolancı. 1977b. “Conservation Inventory of Natural and Cultural Assets Kale Altı Bath Registration Document.” İzmir.

Özeren, Ö., and M. Büyükkolancı. 1977c. “Conservation Inventory of Natural and Cultural Assets Yahşi Bey Bath Registration Document.” İzmir,Selçuk.

Öztan, A. 2009. *Arkeoloji ve Sanat Tarihi Eski Anadolu Uygarlıkları Neolitik Çağ (Yeni Taş/ Cilalı Taş Çağı). T.C. Kültür ve Turizm Bakanlığı Türkiye Kültür Portalı Projesi*. Ankara: T.C. Kültür ve Turizm Bakanlığı Türkiye Kültür Portalı Projesi.

Palomo, A., M. Blanco-Varela, S. Martínez-Ramírez, F. Puertas, and C. Fortes. 2002. “Historic Mortars : Characterization and Durability . New Tendencies for Research.”

Pekak, M. S., and S. Aydın. 1998. “Selçuk ve Çevresinde Osmanlı İdaresindeki Gayrimüslim Tebaanın İmar Faaliyetleri.” *Hacettepe Üniversitesi Edebiyat Fakültesi Dergisi* 15 (2): 125–55.

Pülz, A. 2011. “Bizans Döneminde Ephesos’un Görünümü.” In *Bizans Döneminde Ephesos*, edited by Falko Daim and Sabine Ladstätter, 47–75. İstanbul: Ege Yayınları.

RILEM. 1980. “Tests Defining the Structure -Materials and Construction.”

Scherrer, P. 1995. “The City of Ephesos From the Roman Period to Late Antiquity.” In *Ephesos Metropolis of Asia: An Interdisciplinary Approach to Its Archaeology, Religion and Culture* , edited by Helmut Koester, 1–25. Pennsylvania: Trinity Press International.

Scherrer, P. 2000. *Efes Rehberi*. İzmir: Ege Yayınları.

Şeker, M. 1998. “Aydınoğulları Beyliği ve Ayasuluğ.” In *Birinci Uluslararası Geçmişten Günümüze Selçuk Sempozyumu 4-6 Eylül 1997*, 265–75. İzmir: Ege Üniversitesi Basımevi.

Solak, A. 2016. “Experimental Investigation of Lime Mortar Used in Historical Buildings in Becin, Turkey.” *Medziagotyra* 22 (1): 105–12. https://doi.org/10.5755/j01.ms.22.1.9022.

Stefanidou, M., V. Pachta, S. Konopissi, F. Karkadelidou, and I. Papayianni. 2014. “Analysis and Characterization of Hydraulic Mortars from Ancient Cisterns and Baths in Greece.” *Materials and Structures/Materiaux et Constructions* 47 (4): 571–80. https://doi.org/10.1617/s11527-013-0080-y.

Strabon. 1993. *Antik Anadolu Coğrafyası (Geographika: XII-XIII-XIV)*. Edited by Adnan Pekman. İstanbul: Arkeoloji ve Sanat Yayınları.

Taşcı, B., and H. Böke. 2018. “Properties of Roman Lime Mortars in Ancient Lycia Region.” *AIP Conference Proceedings* 2022 (November). https://doi.org/10.1063/1.5060692.

Telci, C. 2010. *Ayasuluğ (Selçuk) XV-XVI. Yüzyıllar*. 1st ed. İzmir: Selçuk Belediyesi.

Texier, C. 2002. *Küçük Asya Coğrafyası, Tarihi ve Arkeolojisi*. Edited by Ahmet Suat. Vol. 9. Ankara: Ankara Enformasyon ve Dokümantasyon Hizmetleri Vakfı.

Torraca, G. 1996. “Lime Technology in Architectural Conservation.” *Lime News* 4 (1): 34–41.

Tournefort, J. 2008. *Tournefort Seyahatnamesi*. Edited by Stefanos Yerasimos. Kitap Yayınevi.

Uğurlu, E. 2005. “Characterization Of Horasan Plasters From Some Ottoman Baths in İzmir.” Master of Science Thesis, İzmir: Izmir Institute of Technology.

Uğurlu, E., and H. Böke. 2009. “The Use of Brick-Lime Plasters and Their Relevance to Climatic Conditions of Historic Bath Buildings.” *Construction and Building Materials* 23 (6): 2442–50. https://doi.org/10.1016/j.conbuildmat.2008.10.005.

Uğurlu Sağın, E., H. E. Duran, and H. Böke. 2021. “Lime Mortar Technology in Ancient Eastern Roman Provinces.” *Journal of Archaeological Science: Reports* 39 (October):103132. https://doi.org/10.1016/J.JASREP.2021.103132.

Uzunçarşılı, İ. H. 1969. *Anadolu Beylikleri ve Akkoyunlu, Karakoyunlu Devletleri*. Ankara: Türk Tarih Kurumu Basımevi.

Vaschalde, C., A. Durand, and I. Figueiral. 2008. “Charcoal Analysis of Lime Kiln Remains in Southern France : An Original process of Mediaeval and Modern Traditional Lime Burning.” In *Proceedings of the Fourth International Meeting of Anthracology: Brussels, 8–13 September 2008, Royal Belgian Institute of Natural Sciences*, edited by Freddy Damblon, 258. Oxford, England: British Archaeological Reports.

Vicat, L.-J. (Louis-Joseph). 1837. *Mortars and Cements*. 1997 Editon. Donhead. https://doi.org/https://doi.org/10.1038/scientificamerican12021905-25016bsupp.

Vitruvius. 1914. *The Ten Books of Architecture Translated by Morris Hieky Morgon*. Edited by Morris Hieky Morgon. London: Harvard University Press,.

Ward-Perkins, J.B. 1970. *Roman Imperial Architecture*. 1st ed. Vol. 1. New Haven, Connecticut: Yale University Press. https://www.slideshare.net/slideshow/roman-imperial-architecture-wardperkins/252026946.

Watts, A. 2013. *Modern Construction Handbook*. *Modern Construction Handbook*. https://doi.org/10.1515/9783990434550.

Wood, J. T. 2014. *John Turtle Woodun Anıları Efes Kazıları*. Edited by Ali Tamer Ürüm. İstanbul: Arkeoloji ve Sanat Yayınları.

Yavuz, A. T. 2006. “Selçuklu Döneminde Malzeme ve Mimarlık İlişkisi.” In *Geçmişten Geleceğe Anadolu’da Malzeme ve Mimarlık Sempozyumu*, 79–142. İstanbul: Çizgi Basım Yayın.

Zimmermann, N., and S. Ladstatter. 2011. *Wall Painting in Ephesos*. İstanbul: Ege Yayınları.