ARCHEOMETRIC ANALYSIS OF KIRŞEHİR- YASSIHÖYÜK MUDBRICKS MATERIALS

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1.0 INTRODUCTION

In this study, it is aimed to determine the properties of mudbricks dated to Early Bronze Age in Kırşehir-Yassıhöyük by archaeometric methods. The physical, chemical and petrographical properties of these mudbricks were determined by archaeometric analysis. Unit volume weight from physical tests, porosity, water absorption capacity, conductivity analysis from chemical tests and analyzes / water-soluble salt tests, acidic aggregate/binder analysis, granulometric sieve analysis-particle size distribution in aggregate, gravimetric analysis / glow loss analysis, X-Ray Fluorescence analysis (PED-XRF and point Micro-XRF) and petrographic thin section optical microscope analysis. Materials of Yassıhöyük mudbrick structures were photographed and documented and coded for analysis in the laboratory. The paste colors of the samples taken from adobe bricks and plasters were documented by CIE L * a * b * color system by chromometric analysis. It was determined that the physical properties of mudbricks were low and the chemical strengths were high strength. Structural deterioration due to ambient conditions was determined in mudbricks. The salinization problem that may arise from the close environment was not detected in mudbricks. The petrographic analysis revealed that the stones used as fillings on the walls of the mound were mostly of syenite rock type. The mudbricks sampled from different parts of the mound are classified in 6 different types of plasters. This indicates the use of different periods or raw materials. Petrographic analysis revealed that the structural materials are very compatible with the local rock structure.

2.0. MUDBRICKS OF YASSIHOYUK

Adobe; It is a mortar which is prepared as a slime / mud mixture with straw and dense clay content, dried by the sun by hand shaping or pouring into molds and used for masonry. In addition to the determination of the conservation status of Yassıhöyük mudbrick remains, archaeometric examination of the remains in the determined area, it has been evaluated whether the current status of the effective and preventive conservation studies performed before and whether it shows continuity. However, it is of great importance that the content of adobe bricks and plasters be determined for the future preservation works, since the use of preservatives compatible with the original material is required in these studies.

In this study, it is aimed to identify 24 mortar samples belonging to Yassıhöyük by archaeometric analysis and to establish an identity. Ankara Hacı Bayram Veli University-

Historical Materials Research and Protection Laboratory (MAKLAB) and Ankara University-Earth Sciences Application and Research Center (YEBİM).

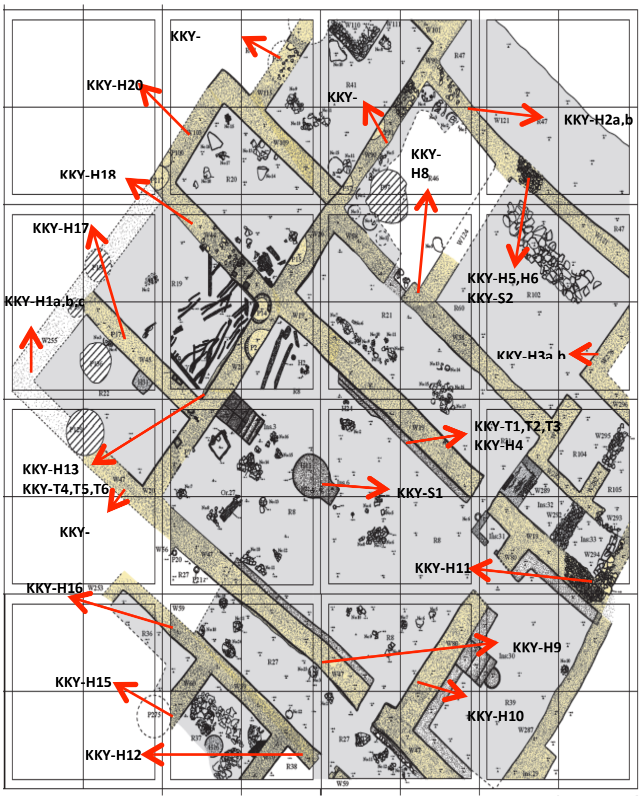


Figure 1

3.0. METHODS AND EXPERIMENTS

The structural and environmental materials collected from the mudbrick structures of Yassıhöyük were documented in the field and in the laboratory. The mudbrick walls that were taken as examples were given a code and processed into architectural drawing plans according to these codes (Figure 1). The mudbrick mortars, one of the building materials in Yassıhöyük, were examined and archaeometric analyzes were performed on 24 mortar samples. Conductivity analysis, salt type and pH tests, acidic aggregate / binder analysis, granulometric sieve analysis (aggregate grain size distribution), gravimetric analysis (weight loss analysis with glow), X-ray Fluorescence (PED-XRF) analysis and thin section optical microscope (petrographic analysis) analysis was applied. The mortar samples analyzed were marked in the architectural plan drawing (Table 1).

Group Code	Photo	Physical Tests (Unit Volume Weight, Porosity, Water Absorption Capacity	Petrographic Analysis	PED-XRF	Aggregate / Binder Ratio Analysis	Gravimetric Analysis (Loss on Ignition Analysis)	Water-soluble salt tests (Conductivity Analysis)	Color Measurement
Mudbrick Plaster KKY-H	24	18	12	24	24	24	24	24

Table 1

The physical properties of Yassıhöyük mudbricks can be explained by the states within the specified standard limits. The structural materials that can be perceived as very robust (durable) visually are climatic (such as temperature and humidity change in the seasonal cycle, freezing and thawing process), environmental (proximity to industrial zones, waste areas, emission of exhaust gases, vegetation etc.) and human (destructive / vandalist). They may be subjected to sudden or medium / long term salinization, cracking, rupture and lichenization. Unit volume weights (saturated-dry BHA, g / cm^3), water absorption capacity (SEK%) by means of direct dry weight of samples, archimedes (in water) and saturated weights (aqueous weight provided to reach pores under 50 torr pressure in pure water) and porosity (P%) values were determined (Ulusay, 2005: 58; RILEM 1980: 73). The amount of sample required to carry out standard physical tests is 5-10 cm3.

Salts, which are naturally present in the adobe mortar or dissolved in water and subsequently carried by the capillary effect to the surface or pores of the materials, provide information about the chemical changes that may occur in the structure of the material itself and in the structure of the other materials to which they relate. The amount of water-soluble salt (total) and types and pH values of the structures were determined. For the determination of total salt measurement in the samples; 5 grams of sample taken in 25 ml of water was centrifuged for 1 hour and filtered and then standard sodium hexametaphosphate was added. Total salt contents of the prepared samples were recorded with conductivity meter (Neukum Series 3001 pH-temperature-conductivity meter) (Means and Parcher, 1963).

Spot salt tests are pre-tests or process anion / cation tests applied in the field during sampling or in the laboratory. It is applied in order to determine the properties of the materials which constitute the natural content or subsequently acquired by external / environmental effects (rain, snow, day-night temperature differences, air pollution, traffic, etc.). These salts are dissolved in water and carried to the material; sulfate, phosphate, nitrate, nitrite, chloride and carbonate and the like. groups. For the determination of salt (cation / anion) species in stone / rock, ceramic and soil samples, spot salt tests were applied and pH distributions of the samples were determined. Tests were carried out either by adding reagents or using strip according to spot test type. Anion assays used standard Merck chloride (Cl^-; 110079), Merck sulfate (SO_4^{2-}; 114789), Merck phosphate (PO_4^{3-}; 114846), Merck nitrite (NO_2^-; 108025) and Merck nitrate (NO_3^-; 111170) test kits (Feigl 1966).

In order to determine aggregate and binder ratios of adobe mortar and plaster samples, firstly dry weighed samples were then treated with dilute acid (5% HCl) to remove binder (total carbonate content; CO32-) contents. Mortar and plaster samples separated from lime and all carbonate contents (binder) by filtration, washing and drying processes were re-weighed after drying at room temperature and total binder and aggregate amounts (% w / w) were obtained. Aggregate particle size distributions (granulometric analysis) were determined by applying systematic sieving (sieves between 63-1000 μm) to non-carbonate aggregates (TS 3530 EN 933-1 / April 1999-February 2007; Tests for Geometric Properties of Aggregates; Part 1: Particle Size) Distribution Determination - Screening Method). It is not possible to reach the aggregate and binder ratios of the mortars only by acidic aggregate / binder analysis. Because of acidic process, in addition to lime, the carbonate-containing materials in the structure are purified, whereas insoluble binders can be treated as aggregates. Acidic aggregate and binder analysis of all the carbonate-containing material purified in the mortar, is expressed as “binder.

Thin sections of all samples of Yassıhöyük mudbricks were prepared and examined under optical microscope. Thin sections; all samples were prepared to show all layers from outside to inside, hardening of mortar samples, soil samples were prepared by sprinkling on a suitable resin. LEICA Research Polarizing DMLP Model was used in the investigations. Photographs were made with a Leica DFC280 digital camera connected to a microscope and evaluations were made using the Leica Qwin Digital Imaging Program. The rocks and minerals that make up the aggregate are defined by Point Counting Program (Kerr 1977; Rapp, 2002).

Element contents of stone / rock, ceramic, mortar, plaster and soil samples were determined by X-Ray Fluorescence Analysis Method (PED-XRF). The pellet is prepared from approximately 3 grams of powdered sample and analyzed by placing it in the sample cup. SPECTRO X-Lab 2000 PEDX brand spectrometer used in X-Ray Fluorescence analysis works in Polarized Energy Dispersive (PED-XRF) system. The device has a nitrogen-cooled Si (Li) detector. The resolution values are <150 eV Mn Ka, 5000 cps. The spectrometer is capable of analyzing sodium (Na) with an atomic number of 11 to uranium (U) of 92. The sensitivity limit of the device is 0.5 ppm for heavy elements and 10 ppm for light elements (La Tour 1989: 3-9; Johnson et. Al 1999: 843-867; Shackley 2011).

4.0. RESULTS OF ARCHEOMETRIC EXAMINATIONS

Unit Volume Weight Test

Samples	BHA-Doygun (g/cm^3)	BHA-Kuru (g/cm^3)	SEK (%)	P (%)
KKY-H1c	2.34	1.66	17.59	29.19
KKY-H2a	2.39	1.60	20.72	33.12
KKY-H4	2.22	1.44	24.36	35.11
KKY-H5	2.06	1.25	31.13	39.02
KKY-H6	2.25	0.91	65.41	59.55
KKY-H7	2.19	1.33	29.58	39.30
KKY-H8	2.39	1.20	41.24	49.66
KKY-H9	2.29	1.58	19.68	31.04
KKY-H10	2.12	1.20	36.21	43.43
KKY-H11	2.21	1.38	26.93	37.28
KKY-H12	2.12	1.30	29.45	38.42
KKY-H13	2.35	1.37	30.58	41.78
KKY-H14	2.26	1.45	24.66	35.78
KKY-H15	2.26	1.53	20.98	32.14
KKY-H17	2.16	1.16	39.97	46.32
KKY-H18	2.32	1.55	21.64	33.43
KKY-H19	2.45	1.68	18.86	31.63
KKY-H20	2.40	1.49	25.35	37.82
Avg.	**2.27**	**1.39**	**29.13**	**38.56**

Table 2. Unit Volume Weight, Water absorption and porosity data

Saturated / dry unit volume weights were determined from the physical properties of the adobe mortar samples of Yassıhöyük settlement (Table 2). According to the structural properties, low density and high porous samples constitute the sample group with low strength. Accordingly, saturated unit volume weights of mortars were 2.06-2.45 g / cm^3 (average 2.27 g / cm^3), dry unit volume their weight ranges between 0.91-1.68 g / cm^3 (average 1.39 g / cm^3). Water absorption capacity of adobe mortar samples ranged between 17.59% and 65.41% (mean 29.13%) (Table 2).

Conductivity Test

According to the conductivity test results (Table 3) of the Yassıhöyük mudbrick mortar samples, the total salt amount is between 0.35-2.88% (average 1.49%) and the most intense amount of salt is seen in the sample with KKY-H15 code.

Samples	**Total Salt (%)**
KKY-H1a	1.01
KKY-H1b	0.75
KKY-H1c	1.38
KKY-H2a	0.60
KKY-H2b	0.86
KKY-H3a	0.35
KKY-H3b	0.65
KKY-H4	1.83
KKY-H5	1.43
KKY-H6	1.42
KKY-H7	0.59
KKY-H8	1.70
KKY-H9	2.52
KKY-H10	1.15
KKY-H11	0.75
KKY-H12	1.64
KKY-H13	2.50
KKY-H14	2.23
KKY-H15	2.88
KKY-H16	2.33
KKY-H17	1.97
KKY-H18	2.44
KKY-H19	2.07
KKY-H20	0.84
Avg.	**1.49**

Table 3. Salt test of adobe mortar samples

Salt Type Analysis

Nitrite, nitrate and sulphate were found to be negligible throughout the samples (Table 4). Chlorine (Cl-) was determined extensively in samples numbered KKY-H3b, KKY-H7 and KKY-H11, dense carbonate (CO32-) in 8 mortar samples and high carbonate in only 2 mortars. When the pH levels were examined, it was found that the pH levels of the mortars where the carbonate was detected at a high rate were also basic.

Samples	**Nitrit (NO_2^-)**	**Nitrat (NO_3^-)**	**Sülfat (SO_4^{2-})**	**Klor (Cl^-)**	**Karbonat (CO_3^{2-})**	**pH**
KKY-H1a	0.050	-	-	3	112	8.34
KKY-H1b	0.025	25	20	3	112	8.50
KKY-H1c	0.075	10	20	3	80	8.58
KKY-H2a	0.050	25	-	3	80	8.52
KKY-H2b	0.025	10	-	3	80	8.52
KKY-H3a	0.050	50	-	3	192	8.46
KKY-H3b	-	10	-	30	192	8.47
KKY-H4	-	-	-	3	80	8.54

KKY-H5	-	-	-	3	80	8.54
KKY-H6	0.025	-	-	3	80	8.59
KKY-H7	0.025	10	-	100	80	8.52
KKY-H8	0.025	-	-	6	80	8.57
KKY-H9	0.025	-	-	3	80	8.58
KKY-H10	0.025	-	20	3	80	8.58
KKY-H11	-	-	20	69	112	8.51
KKY-H12	-	-	-	3	80	8.58
KKY-H13	-	-	-	3	112	8.58
KKY-H14	0.025	10	-	3	80	8.49
KKY-H15	0.025	-	-	3	80	8.50
KKY-H16	0.025	-	-	3	112	8.46
KKY-H17	0.025	-	-	3	80	8.45
KKY-H18	0.025	-	-	3	80	8.47
KKY-H19	0.025	-	-	3	112	8.43
KKY-H20	0.025	-	-	6	80	8.39

Table 4. Salt type and pH results of adobe mortars

Aggregate-Binder Analysis

The aggregates obtained from Yassıhöyük were subjected to acidic treatment and the aggregate / total binder (% TA /% TB) ratios of the samples were obtained (Table 5). According to the data obtained from acid treatment in adobe mortar samples, total binder (% TB) was between 26.56-98.97 and 47.22% on average, total aggregate ratio (% TA) was between 1.03-73.44 and average value was determined as 52.78%.

Samples	TB (%)	TA (%)
KKY-H1a	53.77	46.23
KKY-H1b	38.35	61.65
KKY-H1c	44.27	55.73
KKY-H2a	95.17	4.83
KKY-H2b	42.64	57.36
KKY-H3a	33.82	66.18
KKY-H3b	37.04	62.96
KKY-H4	32.95	67.05
KKY-H5	43.93	56.07
KKY-H6	46.26	53.74
KKY-H7	56.74	43.26
KKY-H8	36.79	63.21
KKY-H9	45.21	54.79
KKY-H10	40.48	59.52
KKY-H11	56.67	43.33
KKY-H12	50.41	49.59
KKY-H13	44.12	55.88
KKY-H14	35.91	64.09
KKY-H15	98.97	1.03
KKY-H16	45.02	54.98
KKY-H17	45.10	54.90
KKY-H18	34.19	65.81
KKY-H19	48.84	51.16
KKY-H20	26.56	73.44
Ort.	**47.22**	**52.78**

Table 5. Aggregate-Binder Ratios

Granulometric Analysis

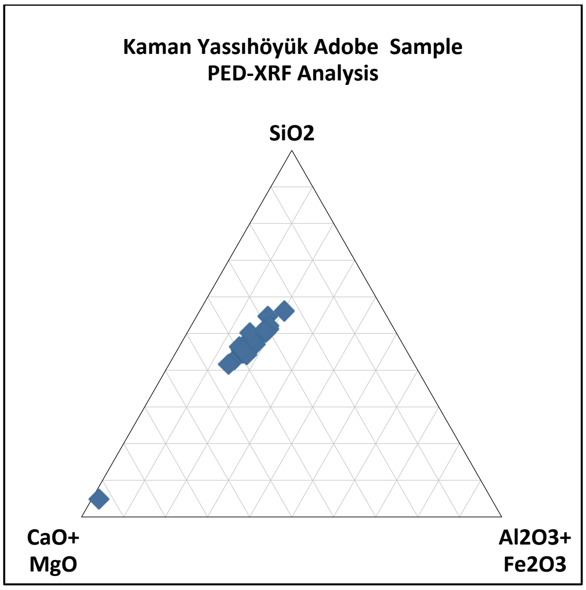
After the acidic aggregate / binder analysis, the aggregates obtained were passed through a systematic sieve (the sieves used were between 63-1000 μm) and the ratio of aggregate particle distribution was reached (Table 6).

Samples	**<63 μm**	**>63 μm**	**>125 μm**	**>250 μm**	**>500 μm**	**>1000 μm**
KKY-H1a	6.14	22.69	27.47	17.68	22.08	3.93
KKY-H1b	6.97	32.54	33.74	12.00	8.25	6.50
KKY-H1c	2.76	9.53	17.31	18.12	23.52	28.75
KKY-H2a	5.35	11.10	19.48	21.14	22.95	19.98
KKY-H2b	6.79	16.76	14.50	15.03	16.83	30.09
KKY-H3a	13.76	34.23	27.44	13.75	6.94	3.88
KKY-H3b	9.17	18.80	32.73	24.77	9.62	4.90
KKY-H4	3.08	6.17	14.49	18.22	22.47	35.56
KKY-H5	5.60	7.80	12.22	21.51	28.35	24.51
KKY-H6	4.36	9.10	11.08	14.80	22.05	38.61
KKY-H7	5.75	10.17	11.66	16.63	22.96	32.83
KKY-H8	37.23	15.67	4.68	6.57	11.10	24.74
KKY-H9	5.27	10.09	13.02	16.04	20.86	34.73
KKY-H10	5.02	11.46	13.11	16.24	20.99	33.18
KKY-H11	7.29	9.96	10.98	14.26	18.35	39.16
KKY-H12	5.15	12.74	13.28	17.03	20.88	30.92
KKY-H13	5.10	12.71	12.03	15.37	20.09	34.70
KKY-H14	4.88	10.32	11.88	14.41	18.25	40.27
KKY-H15	20.69	10.68	15.14	16.90	18.08	18.50
KKY-H16	3.33	10.28	21.96	22.52	25.70	16.20
KKY-H17	5.90	17.45	21.09	19.25	19.82	16.48
KKY-H18	4.30	10.76	12.21	14.97	18.30	39.47
KKY-H19	6.61	12.10	15.05	15.43	18.09	32.73
KKY-H20	4.84	6.52	11.29	13.67	18.33	45.35

Table 6. Results of gravimetric analysis of adobe mortar samples

PED-XRF Analysis

The chemical content of the mortars was determined by X-ray Fluorescence analysis (PED-XRF) and the properties of those with different or similar chemical contents were tried to be understood (Figure 2). PED-XRF analysis and thin section optical microscope results support each other. The main element contents (> 1%) of the mortar samples were examined and their contents (SiO_2- CaO + LOI- MgO + Al_2O_3 + K_2O + Fe_2O_3) were evaluated. SiO2 content in mortar samples ranged from 29.02% to 42.69% (average 32.92%).



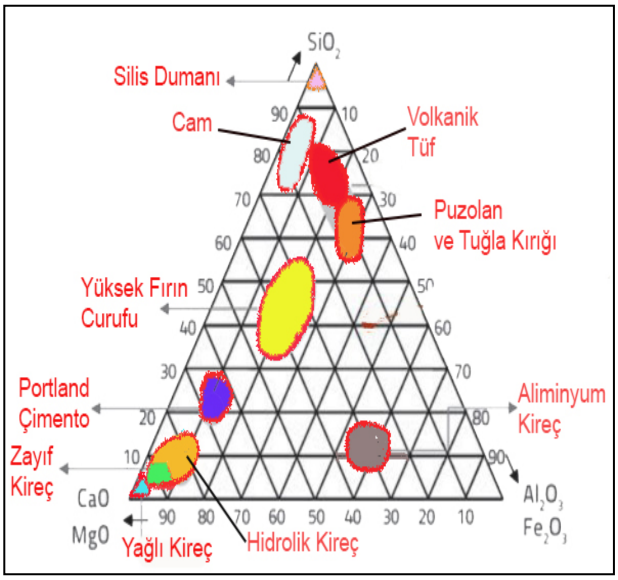


Figure 2. Grouping of adobe samples of Yassıhöyük settlement structures by main element contents via PED-XRF analysis data (Triangle Plotting)

Petrographic Analysis

According to the results of petrographic thin section optical microscope analysis, properties of mineral structure are determined (Table 7-8). According to this analysis, the collected samples are divided into 6 groups. It was found out that the mudbrick samples were exposed to a temperature of 300-400°C.

Harç Örnek Grupları	MTB (%)	Matriks Bağlayıcı İçeriği (%100)			
		Kireç	Kil	Çm	Alçı
Harç Gr1	62	80	20	-	-
Harç Gr2	58	60	40	-	-
Harç Gr3	62	80	20	-	-
Harç Gr4	75	75	25	-	-
Harç Gr5	84	90	10	-	-
Harç Gr6	55	60	40	-	-

Table 7. Petrographic properties - binding structure of Kaman Yassıhöyük mortar samples

Harç Örnek Grupları	MTA (%)	Matriks Agrega İçeriği (%100)		
		Kayaç & Mineraller*	TK	Org
Harç Gr1	38	97 (Q,Ç,Pl,Or,By,Py,Qs,Sy,Op)	2	1
Harç Gr2	42	96 (Q,K,C,Ç,Pl,By,Py,Am,Or,Sy,Op)	2	2
Harç Gr3	38	97 (Q,K,C,Pl,Or,By,Mk,Sy,Op)	2	1
Harç Gr4	25	99 (Q,C,Ç,Pl,Or,By,Py,Qs,Sy,Op)	1	-
Harç Gr5	16	100 (Q,K,Ç,Pl,Py,By,Op)	-	-
Harç Gr6	45	98 (Q,K,Ç,Pl,By,Or,Py,Op)	2	-

Table 8. Petrographic properties of Kaman Yassıhöyük mortar samples - aggregate structure

(*) A: Andezit, Am: Amfibol, B: Bazalt, By:Biyotit C: Kalsit, Ç: Çört, Çm: Çimento, G: Granit, K: Kireçtaşı, Kt: Kumtaşı, Mk: Mikroklin, Op: Opak Mineraller, Or: Ortoklas, Pl: Plajiyoklas, Py: Piroksen, Q: Kuvars, Qs: Kuvarsit , Sy: Siyenit, TK: Tuğla Kırığı Parçaları

Plaster Gr1: KKY-H1a, KKY-H1b, KKY-H1c, KKY-H2a,
KKY-H2b, KKY-H3a, KKY-H3b, KKY-H9, KKY-H17
Plaster Gr2: KKY-H4, KKY-H11, KKY-H12, KKY-H13, KKY-H19
Plaster Gr3: KKY-H5, KKY-H10, KKY-H14, KKY-H18, KKY-H20
Plaster Gr4: KKY-H6, KKY-H8
Plaster Gr5: KKY-H7, KKY-H15
Plaster Gr6: KKY-H16

5.0. DETERIORATIONS

In general, the porosity of the mortars and therefore their water absorption capacity is below 40%. Depending on the total salt content is less than 2%. Due to the low porosity and low water absorption capacity, no deterioration was detected due to the salt content.

Since the binder ratios of the mortars are above 50% and the majority of this binder is composed of lime, the remaining part of the binder ratio is made up of clay, the general purpose of the mortars is to match the cementation index ratios with the natural cement. As a result of the archaeometric analysis, it is seen that the adobe mortars do not deteriorate according to their chemical content and properties. Since the stable state of subsoil conditions cannot be sustained after discovery and the ambient conditions change completely, it is thought that the deterioration that occurs occurs physically affects the adobe. Considering this situation, it is thought that the physical deterioration is caused by atmospheric, climate, day and night difference, seasonal temperature and relative humidity difference, although their durability is high.

Deteriorations detected in mudbrick mortars:

• Deformations caused by rain, caused by direct penetration of rain water directly into the adobe structure, and the deterioration caused by the mud again flowing into muddy and dispersed,

• Caused by wind; deterioration of the raindrops of the wind blowing at high speed during the rain up to the rupture of pieces by hitting the surface of the adobe,

• Direct rainwater contact to some parts of the adobe structure caused by constant humidity, deterioration in the form of deep splits and fractures as a result of continuous exposure of these areas to moisture due to the evaporation of water accumulated in the lower parts of the building walls,

• High and low temperatures caused by seasonal cycles are common in the region. The high temperature of the adobe suddenly evaporated, causing erosion, erosion and consequently loss of resistance of the adobe. However, the sudden drop in temperature also causes sudden freezing of water or moisture in the adobe, and the deterioration which is thought to cause erosion, abrasion and lower adobe resistance again during re-thawing.

• The fact that Yassıhöyük mudbrick has undergone a fire brings about the deterioration caused by fire. The most obvious distortion is caused by color change and visible distortion.

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