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Archaeometric characterization and restoration proposal for filling mortars of Oshki (Öşvank) church

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Abstract

In order to ensure the sustainability of cultural, historical and architectural heritage, preserving and restoring historical buildings and transferring them to future generations is one of the important issues of today. Restoration decisions regarding the repairs of historical buildings are of great importance in this context. Developing correct intervention decisions requires the thorough analysis of the structure, the determination of the structural and material properties meticulously, and the use the most appropriate materials for repairing it. However, only physical and chemical analyses are not sufficient to determine material properties of historical buildings; therefore, archaeometric analyses are needed to determine them more appropriately. In this study, it is aimed to determine the components and characteristics of the filling mortars of Oshki (Öşvank) Church, one of the most magnificent structures of the Eastern Anatolia region, in order to preserve the masonry structure and guide the restoration studies in the light of archaeometric analyses. The raw material properties of the samples taken from the filling mortars of the church were determined by the physical, chemical and petrographic analyses. As a result of the analyses, the aggregate-binding properties of Oshki Church filler mortars were determined. It was revealed that the aggregate content was determined as petrographic, lime was used as a binder and all samples contained pozzolan whereas no cement was used in any of the samples, and the samples did not contain marble rice/powder and organic additives like oil, protein etc. It has also been observed that the aggregate in the mortar content is compatible with the geological formations of near surrounding of the church. In this context, this study emphasizes the importance of archaeometric analysis in restoration of historical buildings. Moreover, differing from other studies in the literature, this study proposes mortar recipe to be used in the restoration of the church, which can pioneer to future studies in the field of architectural restoration.

Keywords Archaeometric characterization, Mortar analysis, Material characterization, Petrography, Restoration, Oshki church

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Introduction

The most important inference adopted jointly in the internationally accepted conventions like Carta Del Restauro [1], the Venice Charter [2] and ICOMOS Charter on the Built Vernacular Heritage [3] can be summarized as such: the restoration is a work requiring expertise, the purpose of the restoration should be to preserve and reveal the aesthetic and historical value of the monument, the restoration is based on the original material taken as a basis and respectfully connected to reliable documents, where traditional techniques are insufficient, the monuments can be consolidated using contemporary techniques validated with scientific data and experiments for protection and construction, and an archaeological and historical examination of the monument should be carried out before and after starting and finishing any restoration work [4].

Masonry structures are structures that carry loads horizontally and vertically with walls built using binding mortar between artificial or natural blocks. The material that connects the blocks such as stone, brick, etc. used during the construction of the load-bearing walls of the masonry structures in order to work with each other as a whole is called mortar [5, 6]. The mixture ratios of the mortar used, the amount of moisture, the mortar thicknesses between the joints and the deformation properties are among the factors affecting the compressive strength, tensile strength, and shear stresses of the masonry structures. In this context, since it is an important factor that directly affects the structural strength and seismic behavior of masonry structures, revealing the true properties of mortars used are of great importance [7–9]. This subject has become more crucial today in the restoration of historical buildings.

There are many cultural assets built by different civilizations in different periods in Türkiye. Materials and mortars used in these buildings, which reflect the cultural, architectural, and aesthetic characteristics of the period in which they were built, show differences according to where and when they were built [10]. In this context, the most variable material among the building materials that make up the historical buildings is the mortar. The correct material selection and/or production in the repair of historical buildings depends on the accurate determination of the material properties of the building [11, 12]. Therefore, before finalizing the intervention decisions, original mortar properties should be accurately determined by experimental methods and intensive care should be taken to ensure that the materials to be used in the restoration of the building are compatible with these properties [13–15].

Characterization of materials used in an object and/or a building with a historical value is an important issue and the first step for the preservation and sustainability of cultural heritage. In the literature, there are several studies focusing on the archaeometric analysis and characterization of materials, building components and objects like stone [16], brick [17], tile, metal [18], wood [19], plaster [20], ceramic [21] and mosaic [22] used in historical buildings, as well as weaving [23], fresco, hand-carved decorations, painting [24], paper [25] and pigment [26] etc. mostly using non-destructive methods in parallel with the developments in the analysis technology. However, there are very limited number of studies on mortars [27, 28] and pozzolanic materials [29], which are abundant in historical archaeological sites.

The characterization of building materials used in historical buildings is examined within the scope of a multidisciplinary science called archaeometry. Thanks to the archaeometric assessment that sheds light on the history by determining the material properties of archaeological findings as well as architectural elements, the raw material contents of the samples, binders and additives etc. can be determined [30, 31]. Characterization studies of both archaeological findings and building components and materials are guiding both in terms of material content and in terms of the construction technique and technology of the period [32, 33]. When evaluated within the framework of protection and restoration, it is possible to repair, complete, strengthen and reproduce the building elements and materials identified by archaeometry with materials that are compatible with the original and/or very close to the original. Thus, historical buildings can be transferred to future generations by preserving their cultural values and original characteristics [34, 35].

The Bagrat Kingdom is a civilization that built monumental buildings in the Tao-Klardjethie region in the seventh-twelfth centuries. Among the religious structures built in the Tao-Klardjetie region during the Bagrat Kingdom, Oshki Church was selected to be examined in this study since it is considered to be the most monumental and magnificent monastery of the northeast Anatolia region [36]. Standard analytical experiments applied for mortars cover only several physical and chemical analyses like unit weight, specific mass, porosity, water absorption by weight, water absorption by volume, compactness and sieve. However, these are not sufficient to determine material properties appropriately [37, 38]. Within the scope of archaeometric analyses, it is aimed to determine the content and physical, chemical and microstructure (petrographic) characteristics of the mortar samples taken from the structural walls of the church by applying the necessary tests and experiments, together with the standard analytical experiments. In this context, this study emphasizes the importance of archaeometric analysis in restoration of historical buildings. Moreover, differing from other studies in the literature, this study proposes mortar recipe to be used in the restoration of the church, which can pioneer to future studies in the field of architectural restoration.

Oshki (Öşvank) church

In the historical resources, in the region covering the borders of Artvin, Ardahan, Erzurum, Kars provinces of Türkiye, in the Çoruh Valley, starting from the first century BCE, the existence of independent Georgian principalities is known. Since Tao and Klardjetie were the most powerful principalities among the Klardjetie, Chavchetie, Artahanı, Tchıldırı, Kola and Tao principalities known to live in the region, the whole region was known as "Tao-Klarjetie" (Fig. 1) [39]. In Tao-Klardjethie region, Bagratids were the builders of the most of the monumental churches between 7-twelfth centuries [4, 36], among which Oshki Monastery was selected to be studied in the scope of this study due to its distinctive architectural features and historical value.

Oshki Monastery is located in Çamlıyamaç Village of Uzundere district of Erzurum province in Türkiye. The monastery takes its name from the old name of the village, Öşvank (Oshki) (Fig. 2). In addition to the church, Oshki Monastery consists of dining hall (refektorium), manuscript room (skriptorium) and chapel structures. The church of the monastery is today largely intact and standing. On the other hand, only the main walls of the dining hall, manuscript room and chapels in the north of the monastery have survived to the present day [36].

The Oshki monastery, which was founded during the reign of Georgian King III. Kuropolat Adarnese's sons David (961–1001) and Bagrat, was first studied scientifically by Takaichvili [43], and he noted that the construction of the church was initiated by Adarnese in 958. Djobadze [44], on the other hand, states that the construction of the church started on March 25, 963 and was completed in 973, based on the inscriptions of the building. Mesipaschvili and Zinzadze [45] stated that it was recorded in the inscriptions on the church that the Oshki Monastery was built by an architect named Oshki Grigol who grew up in Öşvank Village. The name of this architect can be found in three different places: on the south door pediment of the church,

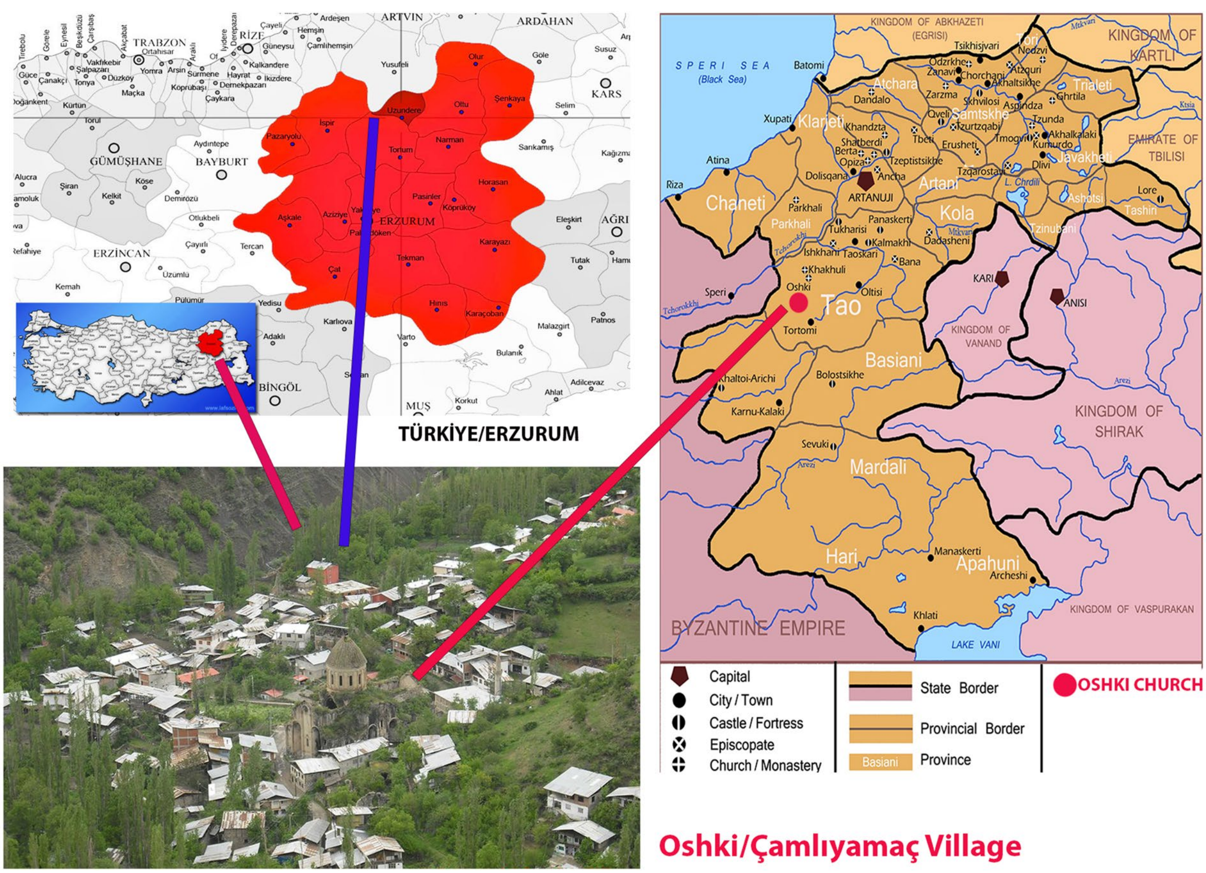

Fig. 1 Location of Oshki Church on the map of Erzurum, Türkiye (left) [40] and the Tao-Klardjetie region (right) [41], and general view of Çamlıyamaç Village [42]

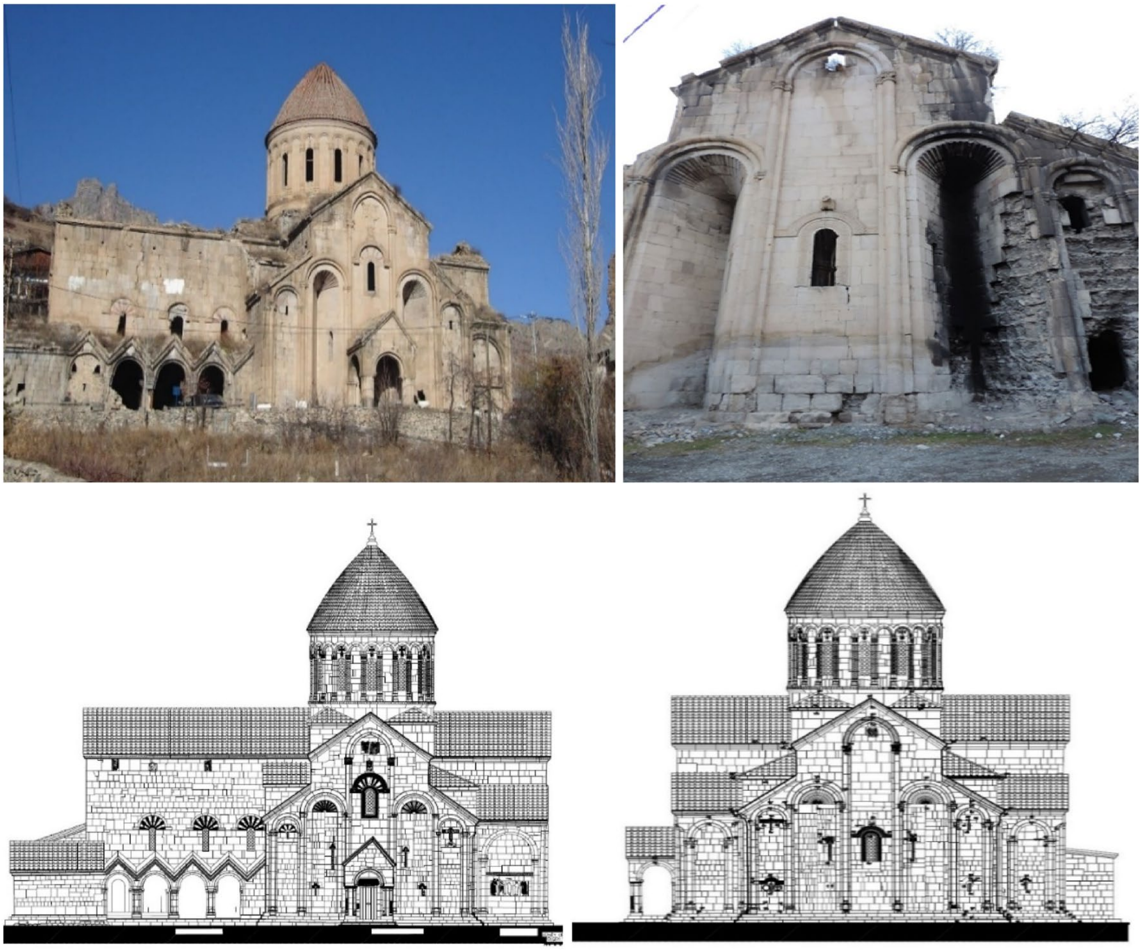

Fig. 2 Oshki Church South (left) and east (right) façade [48]

on the column to the south of the western cross arm, and on the eastern facade [36].

The church is dedicated to John Prodromos, who is called John the Baptist, and depictions of John the Baptist are included in various parts of the church. From the inscriptions in the church, it is understood that the dome was damaged and repaired between 1022 and 1025 during the reigns of the Byzantine Emperor Basil II and Constantine VIII. In the eleventh-twelfth centuries, manuscripts of sacred texts and the Bible were reproduced in the church, and the monastery gained fame in this respect [46]. In the same century, the narthex was added to the west of the church and portico was added to the southwest. Until the fourteenth century, the church was used as an episcopal office. This village is referred to as "Vank-ı Öşk" in the sixteenth century Ottoman records [47]. The church was converted into a mosque in the nineteenth century and was used as a mosque until 1980. The church, which was abandoned and not actively used by the construction of a new mosque to the west of the church, was deformed after the abandonment [36]. The church was registered as an immovable cultural asset with the decision of the High Council of Immovable Real Estate Antiquities and Monuments dated 9.9.1978 and numbered 1297; and the Library and Chapel were registered as an immovable cultural asset with the decision of the Erzurum Regional Council for the Protection of Cultural and Natural Assets dated 25.12.2008 and numbered 1156.

According to the information obtained from the literature, the church, built by an architect named Oshki Grigol in the name of Yahya the Baptist, has a very impressive appearance with both its architecture and its figurative reliefs. Oshki Church, 49.76 m × 29.80 m, is built on a rectangular area and has a mixed plan layout in which the Greek cross, trikonkhos and basilical plan scheme are used together [36] (Fig. 2).

Experimental method

Materials and preparation techniques

During the field studies, the samples were taken by assessing the building on site. A total of 5 mortar samples were taken from the original rubble fillings on the interior walls of Oshki Church to be examined within the scope of material characterization studies (Fig. 3). The samples were photographed and documented before the analysis, and coded for laboratory studies as H1, H2 etc. (H.2; H: code name and 2: sampling location) (Fig. 4). Since the recent restoration history of the building is known, a special attention was paid to sampling from the areas of the building that have not been intervened, that is, which were considered to be original mortars. Special measures were taken not to damage the structure while sampling. In this context, the samples were taken from the inner part of the collapsed interior walls while not giving any deterioration to the masonry structure of the walls. The details of mortar samples are described in Table 1.

Characterization of materials

The raw material properties of the samples taken from the filling mortars of the church were determined by physical, chemical, and petrographic analyses. In this

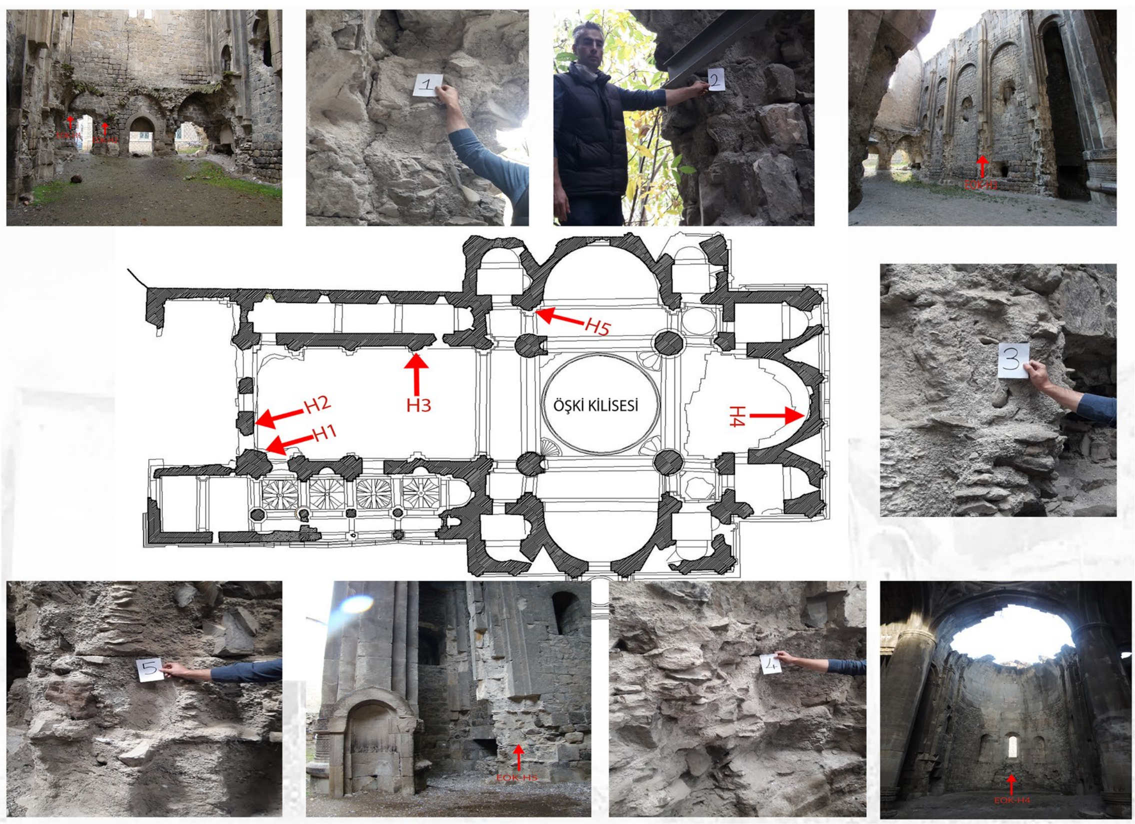

Fig. 3 Oshki Church sampling studies

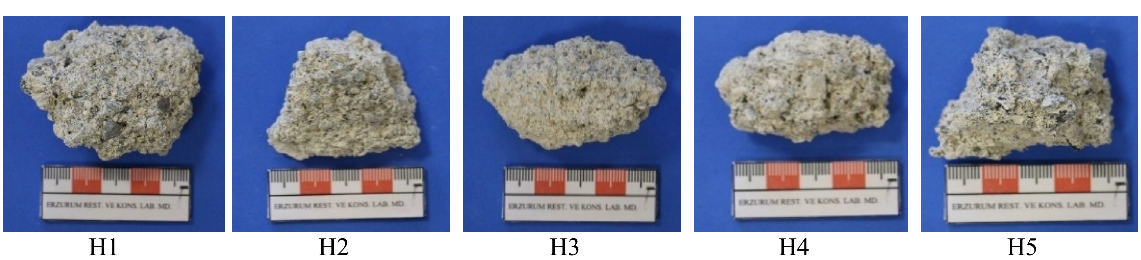

Fig. 4 Macro photos of the samples

Table 1 Mortar samples

Sample name	Description	Function
H1	From the collapsed section of the western wall of the western crucifix	Rubble Filler
H2	From the part of the window that collapsed on the western wall of the western crucifix	Rubble Filler
H3	From the collapsed column of the north wall of the western crucifix	Rubble Filler
H4	From the collapsed east wall of the apse section	Rubble Filler
H5	From the collapsed western wall of the northern arm of the cross	Rubble Filler

context, the physical tests applied to the samples of the filling mortars: water absorption rate by weight and volume, unit volume weight and specific gravity determination and determination of porosity and compatibility. In the context of chemical tests, conductivity analyses, water-soluble salt analyses, silicate aggregate/binder analysis, granulometric sieve analysis- particle size distribution in the aggregate, gravimetric analysis, X-Ray Fluorescence analysis, PED, XRF analyses were performed. The petrographic thin section optical microscope examinations were performed in the context of petrographic analysis. Some of the chemical analyses and petrographic analyses were carried out in Ankara University Earth Sciences Application and Research Center (YEBİM), some of the chemical analyses were carried out in Kastamonu University Central Laboratory (MERLAB); other analyses were carried out by the authors in Erzurum Restoration Conservation Regional Laboratory Directorate. The detailed information about the applied experimental procedures and methods can be obtained from the authors' previous study [4]. The findings of the archaeometric analyses are presented in the following sections.

Findings and results

As a result of physical analyses; porosity and compactness values, water absorption percentages by weight, specific gravity and unit volume weights were determined (Table 2). When the physical analysis results of the mortar samples taken from the structure are examined, it is seen that the porosity percentages vary between 10.27 and 23.01; the compactness percentages vary between 76.99 and 85.79, the porosity average is 16.50%; and the compatibility average is 83.50. It was determined that the water absorption percentages by weight varied between 8.67 and 15.91 and the average was 10.92, the specific weights (density) varied between 1.72 and 1.92 g/cm^3, and the unit volume weights varied between 1.41 and 1.72 g/cm^3. The average of the specific weights (density) was calculated as 1.87 g/cm^3; the average of the unit volume weights was calculated as 1.54 g/cm^3. While the lowest porosity value is seen in H5 sample, it is seen that H3 sample has the highest porosity value. Similarly, the lowest water absorption value of the samples is seen in H5 sample, while the highest one is H3.

In order to determine the chemical properties of the samples; conductivity analyses, acid treatment and granulometric sieve analyses, spot salt tests (water-soluble salt, chloride, phosphate, nitrate detection), X-Ray Fluorescence (PED-XRF) and FTIR analysis were performed. In the context of chemical analyses, acid loss experiment was first performed on mortar samples, and the remaining material after acid treatment was evaluated as aggregate and the particle distribution ratios were determined by sieve analysis (Table 3). In addition to the binder material, aggregates containing calcite such as limestone and marble and lost in acid were also included in the total loss rate (Fig. 5).

Considering the loss/remaining data of the samples: it was determined that the loss rate varied between 29.61% and 41.50%, and accordingly, the remaining rate varied

Table 2 Physical analysis results of mortar samples

Sample	Porosity (%)	Compatibility (%)	Percentage of water absorption by weight (Sa) (%)	Specific gravity (Density) (δ) (g/cm^3)	Unit volume weight (Δ) (g/cm^3)
H1	17.61	82.39	11.20	1.91	1.57
H2	13.48	86.52	8.67	1.80	1.55
H3	23.01	76.99	15.91	1.88	1.45
H4	18.13	81.87	12.86	1.72	1.41
H5	10.27	89.73	5.98	1.92	1.72
Average	16.50	83.50	10.92	1.87	1.54

Table 3 Aggregate/binder ratio of mortars and sieve analysis results

Sample	Acid loss (%)		< 63 µm	63–125 µm	125–250 µm	250–500 µm	500–1000 µm	1000–2500 µm	2500–5000 µm	> 5000 µm
	Loss	Remaining								
H1	33.85	66.15	0.51	0.36	1.43	6.09	13.67	32.60	24.87	20.47
H2	29.61	70.39	0.39	0.39	1.47	5.33	10.40	28.77	15.99	37.26
H3	41.50	58.50	1.55	0.72	1.27	5.70	20.38	55.15	12.46	2.77
H4	37.67	62.33	1.23	0.51	1.46	5.66	20.15	53.40	15.76	1.83
H5	40.57	59.43	1.40	0.96	1.69	4.29	12.38	24.13	18.98	36.17
Average	36.64	63.36	1.02	0.59	1.46	5.41	15.40	38.81	17.61	19.70

The values in the table indicate the proportions of aggregates passing the sieve

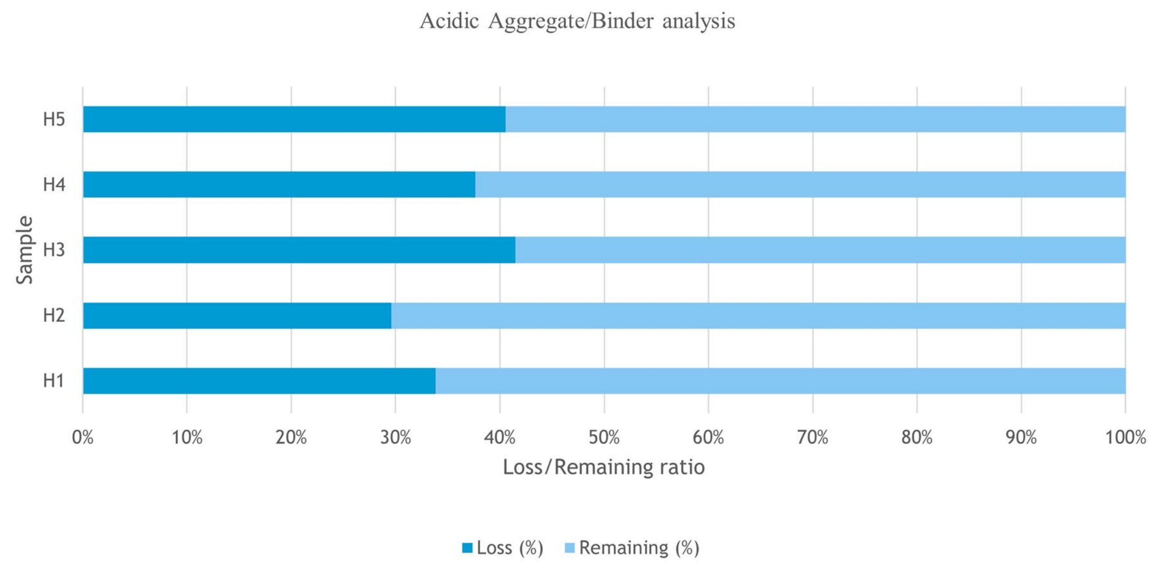

Fig. 5 Acidic aggregate/binder analysis results of mortars

between 70.39% and 58.50%. The average loss was calculated as 36.64%; 63.36% of the total remaining average.

In the Turkish Standard titled as TS 1500 Classification of Soils in Civil Engineering [49], materials smaller than 75 µm are classified as clay/silt, materials between 75 µm and 0.2 mm as fine sand, materials between 0.2 and 0.6 mm as medium sand, materials between 0.6 and 2 mm as coarse sand, materials between 2 and 6 mm as fine gravel. The aggregates of the mortar samples taken at the end of the sieving using different sieves were classified according to TS 1500 (Fig. 6) and their distribution was made and shown in Fig. 7.

Considering the granulometry data of the samples; in H1; it was determined that the most material was collected in a 1mm sieve (approximately 33%), most of the aggregate (approximately 45%) consisted of fine gravel, the amount of sand in its content was approximately 54%, the amount of gravel was approximately 45%, and the remaining material was clay/silt. In H2, it was determined that the highest amount of material was collected in a 5 mm sieve (approximately 37%), most of the aggregate was composed of fine gravel (approximately 53%), the amount of sand in its content was approximately 46%, the amount of gravel was approximately 53%, and the remaining material was clay/silt. In H3, it was observed that the highest amount of material was collected in a 1 mm sieve (approximately 55%), the majority of the aggregate consisted of coarse sand (approximately 76%), the amount of sand in its content was approximately 83%, the amount of gravel was approximately 15%, and the remaining material was clay/silt. In H4, it was observed that the highest amount of material was collected in a 1 mm sieve (approximately 53%), the majority of the aggregate consisted of coarse sand (approximately 73%), the amount of

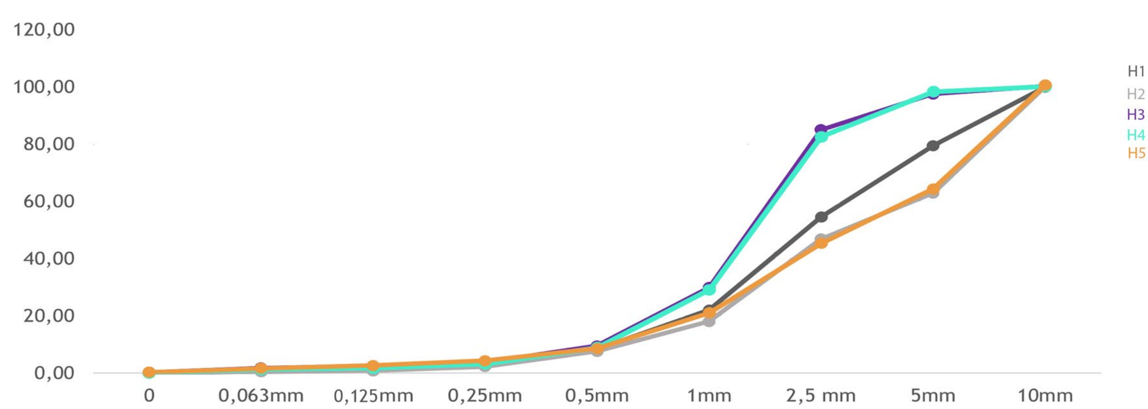

Fig. 6 Diagram showing the results of aggregate sieve analysis

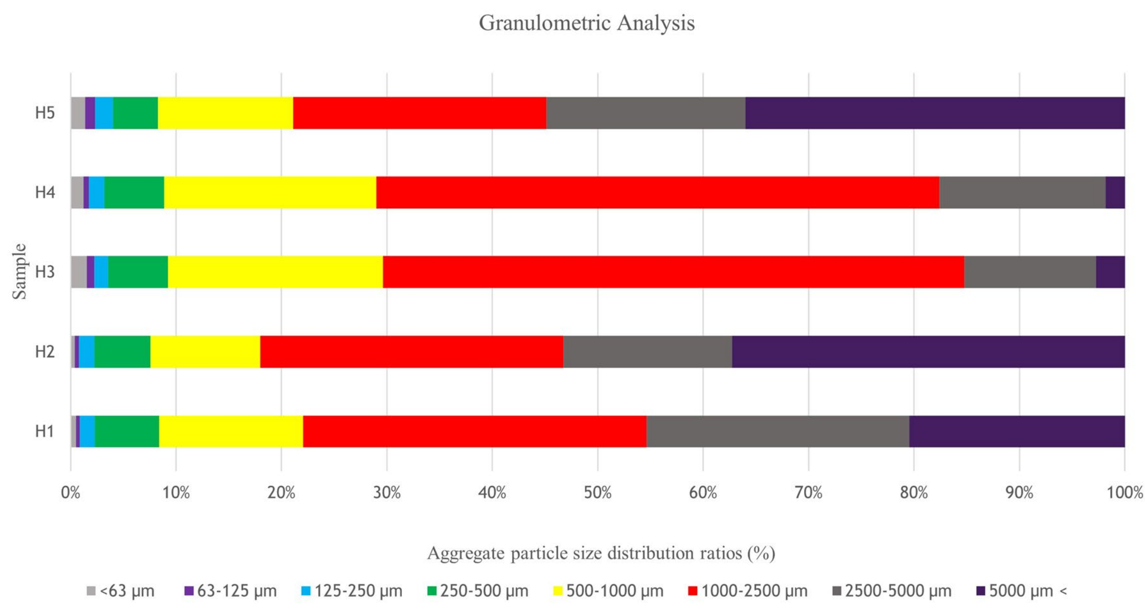

Fig. 7 Acidic aggregate granulometric analysis results

sand in its content was approximately 81%, the amount of gravel was approximately 18%, and the remaining material was clay/silt. In H5, it was determined that the maximum amount of material was collected in a 5 mm sieve (approximately 36%), most of the aggregate was composed of fine gravel (approximately 55%), the amount of sand in its content was approximately 44%, the amount of gravel was approximately 55%, and the remaining material was clay/silt.

The moisture, molecular water, organic matter, carbon dioxide loss and calcium carbonate percentages in the material are determined by the calcination analysis made by pulverizing the binder part of the mortar samples and presented in Table 4.

Considering the gravimetric (calcination) data of the samples, it was determined that the humidity ranged between 0.99% and 2.13%; the organic loss ranged between 1.98% and 6.38%; the carbon dioxide loss ranged between 12.77% and 16.33%; and the calcium carbonate ratio ranged between 29.01% and 37.11%. The average moisture loss was calculated as 1.65%, the average organic loss as 3.31%, the average carbon dioxide loss as 14.33% and the average calcium carbonate as 32.57%.

Considering the amount, conductivity value and salt percentages of water-soluble chloride (Cl^-), nitrate (NO_3^-), phosphate (PO_4^{3-}) salts of the samples (Table 5): it was determined that chlorine varied between "too many" and "yes–no", nitrate varied between "too much" and "yes", and phosphate varied between "yes" and "no". The conductivity value was measured between 129 µS and 580 µS, and accordingly, the salt amounts were calculated as 0.72% and 3.22% (Table 5).

X-Ray Fluorescence (PED-XRF) analysis was applied to determine the chemical compositions of mortar samples, and the results were evaluated. The basic oxide compound values determined as a result of the PED-XRF analysis of the mortar samples are as presented in Table 6.

Table 4 Gravimetric (calcination) analysis results

Sample	Humidity (105 °C, %)	Organic loss (H_2O) (550 °C, %)	Carbon dioxide loss (CO_2) (1050 °C, %)	Calcium carbonate ($CaCO_3$, %)
H1	1.01	3.03	13.13	29.84
H2	2.08	3.13	14.58	33.14
H3	0.99	1.98	14.85	33.75
H4	2.04	2.04	16.33	37.11
H5	2.13	6.38	12.77	29.01
Average	1.65	3.31	14.33	32.57

Table 5 Qualitative and semi-quantitative analysis of water-soluble salts of samples and protein and fat analysis results

Sample	Cl^- (Chloride)	NO_3^- (Nitrate)	PO_4^{3-} (Phosphate)	Conductivity (µS)	Amount of salt (%)
H1	++++	++	+	507	2.82
H2	++	+++	+	456	2.54
H3	±	+	±	129	0.72
H4	++	+++	+	471	2.62
H5	+++	++++	±	580	3.22

(−: None; ±: Yes–No; +: There are few; ++: Yes; +++: There are more; ++++: There are too many)

Table 6 Basic oxide compound values obtained as a result of PED-XRF analysis of the samples

Basic oxide compounds (%)	H1	H2	H3	H4	H5
Na_2O	0.600	0.800	0.083	0.270	0.650
MgO	1.166	1.456	1.077	1.274	1.431
Al_2O_3	5.343	6.362	4.942	5.587	5.691
SiO_2	37.440	43.780	35.920	40.210	40.690
P_2O_5	0.0573	0.0622	0.0508	0.057	0.079
SO_3	0.085	0.114	0.056	0.084	0.066
Cl	0.531	0.208	0.022	0.152	0.355
K_2O	0.679	0.817	0.675	0.795	1.084
CaO	23.230	21.770	27.560	24.030	20.600
TiO_2	0.363	0.396	0.301	0.345	0.380
V_2O_5	0.014	0.014	0.009	0.016	0.015
Cr_2O_3	0.003	0.002	0.022	0.003	0.002
MnO	0.079	0.083	0.101	0.092	0.096
Fe_2O_3	4.188	4.050	3.886	4.201	4.390
Loss of ignition	26.49	20.85	25.74	22.56	24.76
Total	100.27	100.77	100.42	99.68	100.29

Table 7 Calculated Cementation Index (CI) values of the samples

Sample	CI (Cementation Index)	Binding
H1	4.57	NC/C
H2	5.56	NC/C
H3	3.74	NC/C
H4	4.71	NC/C
H5	5.45	NC/C
Average	4.81	NC/C

< 0.30 Oily Lime; 0.30–0.50 Weak Hydraulic Lime; 0.51–0.70 Average Hydraulic Lime; 0.71–1.10 Hydraulic Lime; 1.11–1.70 Natural Cement; > 1.70 Natural Cement/Cement; NC: Natural Cement; C: Cement

Considering the oxide compound data obtained from the PED-XRF results of the samples, it was determined that the magnesium oxide (MgO) ratio varied between 1.077 and 1.456%, the aluminum oxide (Al_2O_3) ratio between 4.942 and 6.362%, the silicon dioxide (SiO_2) ratio between 35.920 and 43.780%, the calcium oxide (CaO) ratio between 20.600 and 27.560%, and the iron oxide (Fe_2O_3) ratio between 3.886 and 4.390%. In addition, the mean magnesium oxide (MgO) was calculated as 1.281%, the mean aluminum oxide (Al_2O_3) as 5.585%, the mean silicon dioxide (silica, SiO_2) as 39.608%, the mean calcium oxide (CaO) as 23.438%, and the mean iron oxide (Fe_2O_3) as 4.145%.

Cementation Index (CI) is described as the ratio of the acid-soluble part to the base-soluble part. Lime mortars are distinguished as oily mortar or hydraulic mortar depending on the binder content and aggregate type. Mortars with a total aggregate content of less than 5% are called oily mortars, and the lime (CaO) ratio of these mortars is quite high. Mortars with a total aggregate ratio above 5% are called hydraulic mortars, and the CaO rate of these mortars is low. Silicon dioxide (SiO_2), aluminum oxide (Al_2O_3) and iron oxide (Fe_2O_3) ratios are high in the composition of such mortars [50, 51]. Based on the chemical compositions of the mortar samples, Cementation Index (CI) values were calculated, and binder types were determined (Table 7). The Cementation Index (CI) data of the samples ranged from 3.74 to 5.56, and the average was calculated as 4.81. All samples were found to be in the natural cement/cement (NC/C) range.

The mortar samples analyzed were grouped by showing their positions in the Keily diagram according to their chemical contents and oxide compound ratios (Fig. 8). It was determined that the samples were located near the region of the blast furnace slag in the Keily diagram.

As a result of the FTIR analysis applied to the H1 and H2 samples; 1405cm^{-1} peak and 872cm^{-1} band indicating the presence of lime in the H1 sample; 3450cm^{-1} band and 1028cm^{-1} peak indicating the presence of clay; 872cm^{-1} peak and 464cm^{-1} band indicating the presence of silicate; 1405cm^{-1} peak and 711cm^{-1} band indicating the presence of nitrate were observed in the H1 sample (Fig. 9). In the H2 sample, 1409cm^{-1} peak and 873cm^{-1} band indicating the presence of lime; 3452cm^{-1} band and 1035cm^{-1} peak indicating the presence of clay; 873cm^{-1} peak and 457cm^{-1} band indicating the presence of

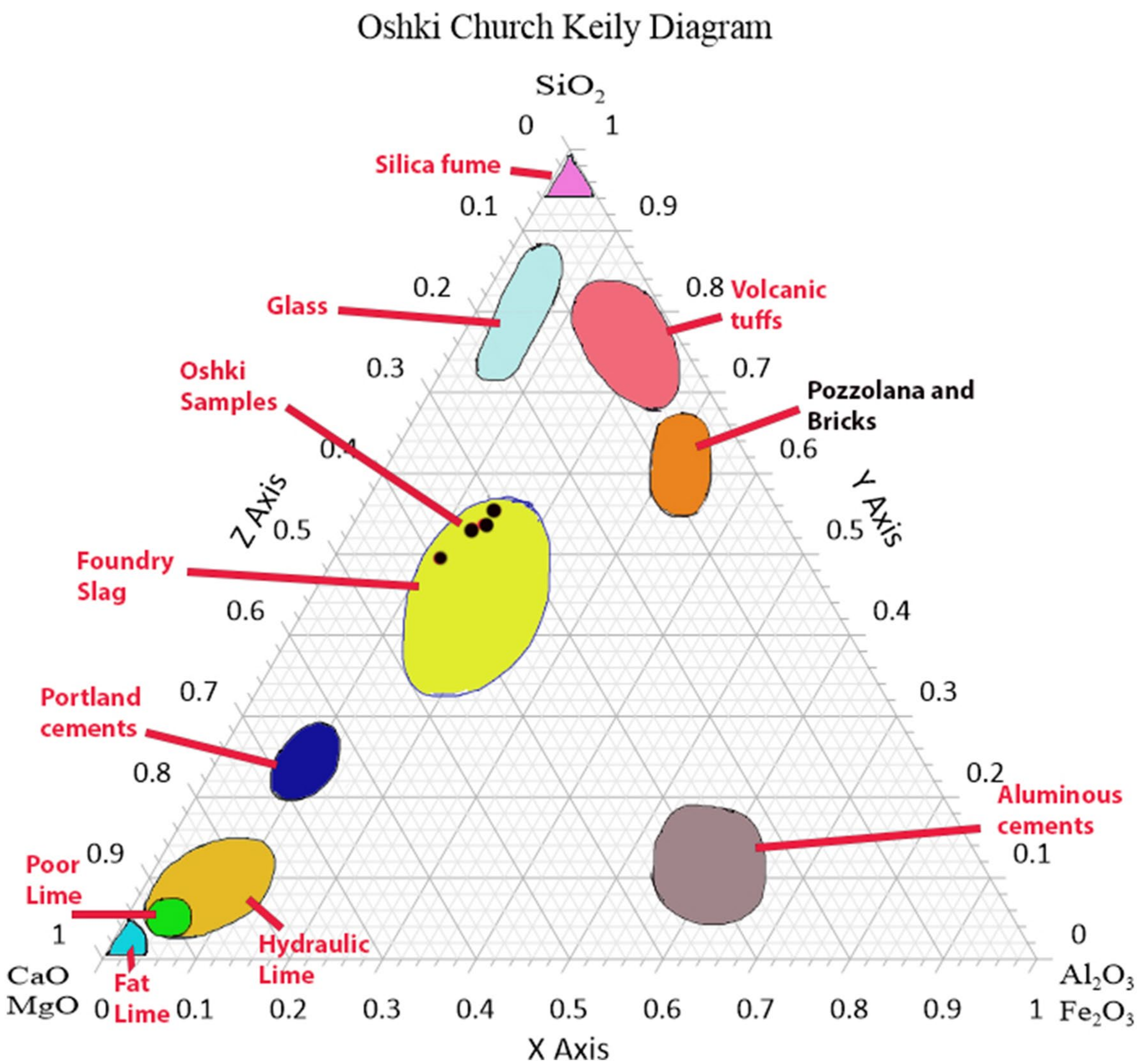

Fig. 8 The locations of the samples in the Keily diagram ($CaO+MgO-SiO_2-Al_2O_3-Fe_2O_3$) (adapted from [52])

silicate; 1409cm^{-1} peak and 844cm^{-1} band indicating the presence of nitrate were observed (Fig. 10) [53].

Petrographic examinations were carried out under a polarizing microscope by preparing thin sections and the mineralogical properties of the samples were determined (Fig. 11). When the petrographic properties of the samples were evaluated, it was determined that all samples were collected in a single group, 25% of their contents were binder and 75% were aggregate (Table 8). It was understood that 80% of the binding part consisted of lime and 20% of clay, and cement or plaster was not added to the mixture. It has been determined that the aggregate part contains some rock fragments and minerals (sandstone, basalt, biosparitic limestone, biomicritic limestone, granite porphyry rock fragments, chert, quartz and plagioclase minerals). In addition, based on the aggregate forms, it was observed that the material used was stream sand. In the thin-section (micro) photographs of the samples, it was concluded that the aggregates in the mortar were river sand due to their rounded edges (Fig. 11). On the other hand, the results showed that the samples did not contain marble rice/powder and organic additives like oil, protein etc.

Discussions

With this study, it is aimed to define the characteristics of the filling mortars of Oshki (Öşvank) Church through archaeometric analyses. In this context, physical, chemical, and petrographic analyses were applied to the mortar samples taken from the structure and the evaluations made in the light of the data obtained are discussed in this section.

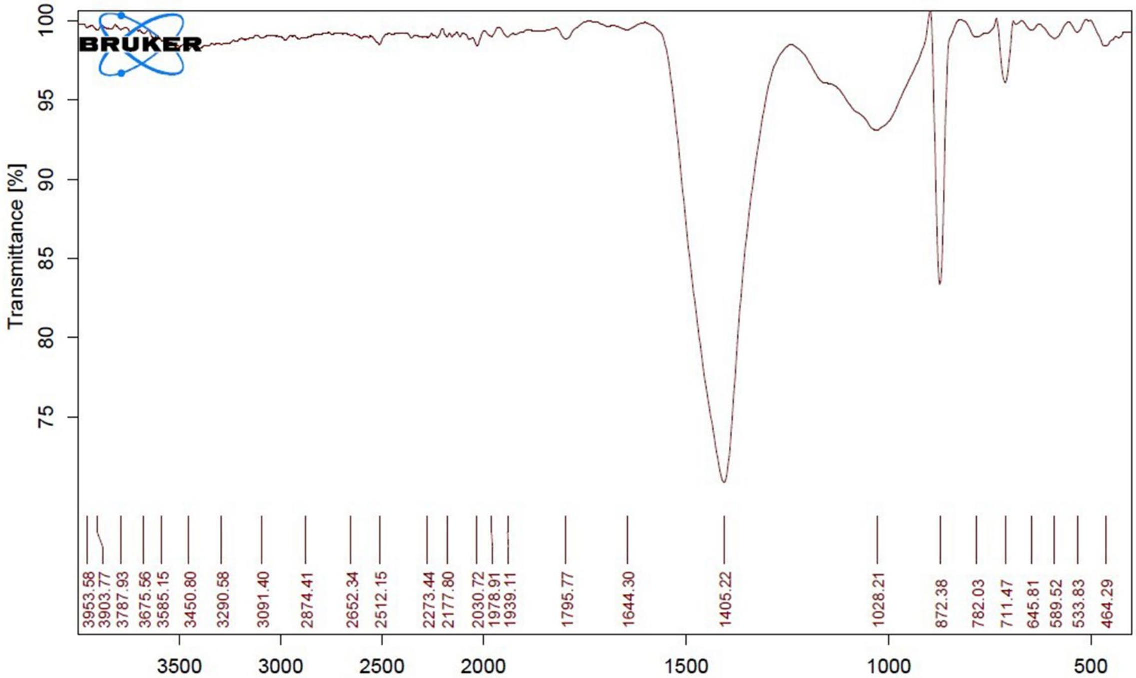

Fig. 9 FTIR spectrum graph of H1

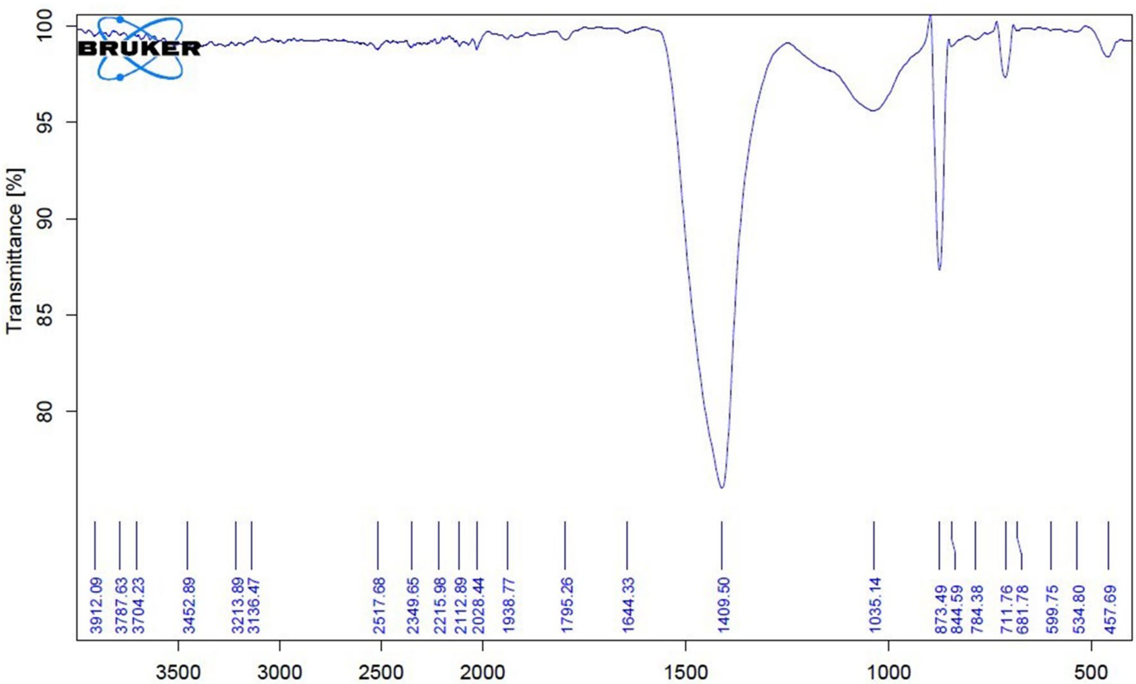

Fig. 10 FTIR spectrum graph of H2

As a result of physical analyses, based on the porosity and density values of the samples, it was concluded that the filled volume of the samples was high density although there observed voids in sample. The filled volume outside the void volume was high density. The mortar samples show different physical properties according

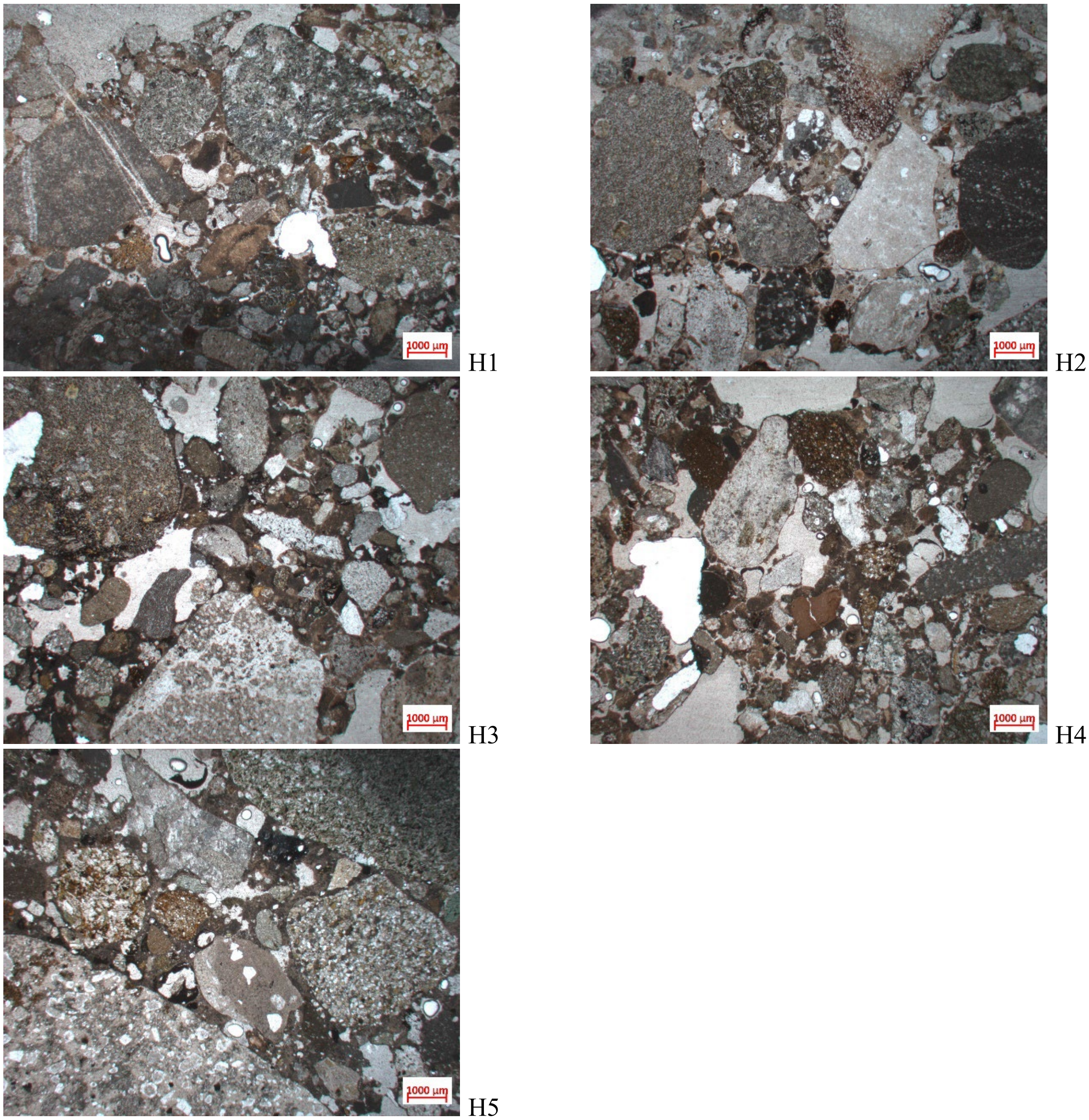

Fig. 11 Micro photographs of the samples taken under the polarisone microscope

to the type and mixture ratios of the materials that make up their contents. As porosity increases, the resistance of mortars against freezing/thawing and water-soluble salts decreases, however, since it is known that it causes discontinuity in load transfer, it is possible to say that the most resistant mortar against physical and chemical factors among the samples is H5.

As a result of the acid treatment analyses, the loss average of the samples was calculated approximately, and it was observed that the loss values of the samples were close to the average. Based on the determination of the amount of binder as 25% in the petrographic analyses, it was concluded that the difference of approximately 12% between the data obtained as a result of acid treatment and petrographic analysis was due to other calcite-containing materials other than the binder in the mixture.

As a result of the granulometry analyses, it was observed that the material ratios collected under the sieve (< 63 µ, clay/silt), 63µ, 125µ and 250µ sieve were close to each other. While the material ratios collected in the 500µ and 2500µ sieves were partially close to each other, it was determined that the differences in granulometry were caused by the 1000µ and 5000µ

Table 8 Petrographic properties of the samples

Sample	MTB (%)	MTA (%)	Matrix Binder Content (%100)				Matrix Aggregate Content (%100)			Descriptions
			Lime	Clay	Cm	Plaster	Rocks & Minerals	BF	Org	
H1-H2-H3-H4-H5	25	75	80	20	–	–	100 (Sandstone, Basalt, Biosparitic Limestone, Biomicritic Limestone, Granitporfir rock fragments, Chert, Quartz and Plagioclase minerals)	–	–	River sand

B: Binder; A: Aggregate; Cm: Cement; BF: Brick fracture; Org: Organic matter sieves. It was determined that the largest grain size was over 5 mm and most of the aggregates consisted of sand.

As a result of the gravimetric (calcination) analyses, it was determined that all values were close to the average, the highest moisture and organic loss was at H5, and the highest calcium carbonate ratio was at H4. However, it was observed that the least moisture and organic loss was at H3 and the least calcium carbonate loss was at H5.

As a result of the amount, conductivity value and salt percentages of chloride (Cl^-), nitrate (NO_3^-), phosphate (PO_4^{3-}) salts; it was determined that the amount of chloride varied between the samples and was found to be at H1 at most, and it was found to be very small in H3 unlike other samples. It was observed that the phosphate was close to each other in all samples and was found in small amounts. It was determined that the presence of nitrate varied among the samples just like the presence of chloride, while it was at a very high level in H5, it was at a very low level in H3. It was determined that the conductivity values were close to each other except for H3, and that H3 was at a very low level in the conductivity value as in the presence of chloride and phosphate. Based on the conductivity value, the amount of salt was calculated at least at H3. Since it is known that the presence of chloride is caused by materials such as cement, soil, sea water or sea sand that cannot be cleaned sufficiently or from the ground, it is assumed that the chloride differences between the samples are caused by the material forming the mortar mixture. However, since nitrate and phosphate salts are generally known to be caused by the wastes of living creatures (birds, microorganisms, etc.) or environmental factors (fertilizer used around the building, waste, etc.), it is thought that the nitrate differences between the samples are caused by the bird and other animal wastes exposed due to the fact that the church is open to external conditions. When the salt tests performed and the chloride (Cl) data obtained as a result of PED-XRF analysis are compared, it is seen that the results are consistent. The amount of salt was calculated very high in all samples. Because > 0.15% in the soil indicates a high amount of salting [54, 55].

As a result of PED-XRF analyses; magnesium oxide (MgO), aluminum oxide (Al_2O_3), iron oxide (Fe_2O_3), silicon dioxide (SiO_2) and calcium oxide (CaO) ratio were found to be close to each other. While the CI value increases as the silicon dioxide (SiO_2), aluminum oxide (Al_2O_3) and iron oxide (Fe_2O_3) values increase, the CI value decreases as the calcium oxide (CaO) and magnesium oxide (MgO) values increase. In this context, the fact that the oxidized compound values of the samples were close to each other and the high percentage of those that increased the CI value caused the CI values to be calculated close to each other and high. The fact that the CI value is high is an indication that the mortar strengths are also high.

When the Keily diagram is evaluated, it is seen that the samples are located near the blast furnace slag. Since there are also contemporary materials (blast furnace slag, silica fume) that cannot be used in the Oshki Church examined within the scope of the study within the scope of the Keily diagram, it would be more accurate to evaluate the regions where the mortars are located in terms of the rates of oxidized compounds (Al_2O_3, Fe_2O_3, MgO, CaO, SiO_2) in the mortar content rather than which material they correspond to. When the subject is evaluated from this point of view, it becomes clear that there is a pozzolan in all samples.

As a result of FTIR analyses; strong 1405–1417cm^{-1} and 844–872cm^{-1} characteristic bands indicating the use of lime are seen [53]. While the sulfate band was prominent in the samples, it overlaid the peaks 1415–1419 on the lime peaks in the other examples. These results are also consistent with Cl tests. No trace of protein or oil use was detected in any of the samples, and although the positive effects of the mortars are known, the fact that no additives are used in the filler mortars brings to mind the idea that economic or other conditions do not allow it.

As a result of the petrographic data, it was determined that the binding content of the samples consisted of lime and clay, and no trace of cement or gypsum was found in any sample. It would be correct to evaluate the clay (20%) detected in the binder content as pozzolan. The high Cementation Index (CI) values already support the fact that mortars contain pozzolan. The rock fragments forming the aggregate were classified as igneous rocks (depth-surface-vessel), metamorphic and sedimentary (carbonate and crumbly) rock fragments. Magmatic rocks are grouped as vein-diabase, depth-granite, surface rock-basalt. Sedimentary rocks were separated from carbonate rocks as limestone, biosparitic and biomicritic, and clastic sedimentary rocks were found to be chert and sandstone. Metomorphic rock fragments were observed to be marble and quartzite. Minerals are divided into quartz, beachoclase, calcite and opaque minerals. It has been determined that there is stream sand in the aggregate content. In the light of all data, it was understood that lime was used as a binder, the samples did not contain cement or plaster, and all samples contained pozzolan. The results of the analysis obtained are summarized in Table 9.

Conclusions

In order for the historical artifacts to be repaired in accordance with the original and transferred to the future; it is of great importance to analyze the damage

Table 9 Archaeometric analysis results of Oshki Church

Sample	Physical Analysis			Acid Loss		Gravimetric Analysis		Salt Values		CI	XRF Values					Petrographic Data				
	P (%)	δ (g/cm^3)	Δ (g/cm^3)	Loss (%)	Remaining (%)	H_2O (%)	CO_2 (%)	Conductivity (μS)	Salt (%)	CI	MgO	Al_2O_3	SiO_2	CaO	Fe_2O_3	MTB (%)	MTA (%)	Lime (%)	Clay (%)	Org. (%)
H1	17.61	1.91	1.57	33.85	66.15	3.03	13.13	507	2.82	4.57	1.16	5.34	37.44	23.23	4.18	25	75	80	20	-
H2	13.48	1.80	1.55	29.61	70.39	3.13	14.58	456	2.54	5.56	1.45	6.36	43.78	21.77	4.05	25	75	80	20	-
H3	23.01	1.88	1.45	41.50	58.50	1.98	14.85	129	0.72	3.74	1.07	4.94	35.92	27.56	3.88	25	75	80	20	-
H4	18.13	1.72	1.41	37.67	62.33	2.04	16.33	471	2.62	4.71	1.27	5.58	40.21	24.03	4.20	25	75	80	20	-
H5	10.27	1.92	1.72	40.57	59.43	6.38	12.77	580	3.22	5.45	1.43	5.69	40.69	20.60	4.39	25	75	80	20	-

P: Porosity; δ: Density; Δ: Unit volume weight; CI: Cementation Index; MTB: Matrix total binder; MTA: Matrix total aggregate; Org: Organic material detection, deterioration causes, material properties and static situation [56]. In order to obtain accurate results, it is important that these studies are carried out jointly by the relevant professional groups. Since one of the most criticized issues in today's restoration studies is the use of materials that are incompatible with the structure, determining the material characterization and choosing a repair material compatible with the original material plays a major role in increasing the quality of restoration [29, 35]. With this study, it is aimed to determine the content and characteristics of the filling mortars of Oshki (Öşvank) Church, one of the most magnificent structures of the Eastern Anatolia region, in order to preserve the masonry structure and guide the restoration studies in the light of archaeometric analyses.

The results showed that the physical properties of the mortar samples taken from the church are not very close to each other. However, based on the density values, it was concluded that the mortar used showed similar physical properties, and the porosity differences were due to labor and compression between the applications. The fact that the mortars have reached the present day without dispersing under external conditions is the most important indicator that the physical properties of the mortars are in good condition.

It was determined that the loss rate of the mortar samples belonging to the church was not high in acid, and lime and pozzolan were used as binders. In addition, it can be said that some calcite-containing aggregate is used in the samples. However, due to the differences caused by the 1000μ and 5000μ sieves, it cannot be said that the granulometry graphics are compatible. Based on the aggregate forms, it has been determined that stream sand is used in mortar samples. Since the region is rich in rivers, it is thought that the sand in question is obtained from the rivers and streams in the immediate vicinity. Based on the macro-physical structures of the mortar samples, it can be said that the rich aggregate type, which is preferred as a result of a certain elimination, has a heterogeneous distribution and is compatible with the local formation, participates in the mortars.

Since no cement or derivative material was detected in the mortar content, it suggests that the presence of chloride in the samples originated from the ground or the material (binder, aggregate, mixing water) that formed the mortar. However, it was determined that the presence of phosphate in the samples was very low, the presence of nitrate varied and was high in some samples. Since it is known that the presence of nitrate and phosphate is usually caused by the wastes of living things in or around the structure, the location of the artifact and environmental factors, it brought to mind the idea that the nitrate differences between the samples are caused by the bird and other animal wastes as the church is exposed to external environment.

Since the amount of salt is more than 0.15%in the soil, which is known to indicate a high amount of salting [54], it is understood that the amount of salt is very high in all samples. Since it is known that the church has not been restored for a long time, this cannot be associated with a new application/intervention to the building. Then, the high salt content in the samples suggests the possibility that the material in the mortar content is due to mixing water or soil.

The small differences between the oxidized compounds (CaO, MgO, Al_2O_3, Fe_2O_3, SiO_2) resulting from the analyses of the samples with XRF are thought to be due to the rock content used in the aggregate, as the binder/aggregate ratios were found to be the same. The high CI values of all samples are an indication that the mortar strengths are also high. However, the fact that these values are high should not bring to mind the idea that cement and similar materials are used in the mortar content. Because the petrographic evaluation of the samples showed that no cement was found in the mortar mix.

Lime/clay mixture was used as binder in all samples. Since the amount of binder was kept low, it was observed that it remained below the mixture ratio of 2:1 (aggregate: binder), which is frequently seen in traditional applications. It was determined that local rocks in the immediate vicinity were preferred as aggregates, and consisted of aggregates with certain grain sizes rather than random mix of aggregate sizes. It was determined that grinded, sieved brick/tile powder and/or volcanic clay/tuff powder were added to the mortar content with pozzolanic properties, and although its positive effects on mortars were known, it was understood that no additives such as oil or protein were added to the church mortars. The lack of additives in the filling mortars might result from the economic situation at the time of the construction.

Despite the church has been located on highly seismic area (East Anatolian Fault Zone) and it has survived several sizable earthquakes throughout its life time, the church is still structurally solid and standing. In general, it was concluded that the building materials in the immediate vicinity were used in the church, and it was built with quality materials and workmanship.

There has been no research on the materials used in other churches in the region, except from a master thesis [4]. When compared with the results of the mortar analysis of Barhal, Haho and İşhan churches, built in the same period in the region by the Bagrat Kindgdom [4], the collective sizes of all churches appear to be similar. In addition, the rock fragments that make up the aggregate have also similar properties. They are classified as igneous rocks (depth-surface-vein),

Table 10 Oshki church filling mortar proposal for restoration

Total binding (1 Unit)		Total aggregate (3 Units)	Water
Lime 80%	Natural or artificial pozzolan (Volcanic clay/ tuff powder) 20%	Local rock fragments 5% Clay/Silt 75% Sand 20% Fine Gravel	Adequately

metamorphic and sedimentary (carbonate and clastic) rock fragments. It has been observed that the mortars used in these churches belonging to the same culture. No brick fractures were seen in the mortars of Barhal İşhan and Oshki churches while the brick fractures were detected in the mortars of Haho church. The first use of brick was seen in the Bagrat Kingdom region in the mid-tenth century. The fact that there are no broken bricks in the mortars in these structures, which are thought to have been built before this date, is also important in terms of reflecting the characteristics of the period [57].

In the light of the data gathered from the archaeometric analyses conducted within the scope of this study, mortar prescriptions were proposed to be used in restoration of the Oshki church as presented in Table 10 below. Such studies can be applied for other historical buildings; therefore, both material properties of the structures can be determined more appropriately and the most appropriate mortar content for restoration works can be developed accordingly.

Consequently, deliberate care should be given to keep the type and amount of the test wide during the material characterization phase to be carried out as a guide to the restoration works. Based on the data to be obtained only from chemical or petrographic analyses, it should be taken into account that determining the original mortar properties may lead to erroneous results. After evaluating and comparing the data obtained from all experimental studies including physical, chemical and petrographic, it was understood that they were consistent, and it was concluded that it was a more accurate way to present the results. In this context, the material mixes to be used in the restoration of historical buildings can also be determined more accurately.

Author contributions
Conception of idea (EK, FZC); introduction (FZC, EK); archaeological overview (EK); material and method, analysis and discussions, conclusions (EK, ME, MC, FZC).

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Data availability
Materials used for the study are available upon reasonable request.

Declarations

Ethics approval consent to participate
The authors declare the integrity of the scientific record. All authors, read, reviewed, edited and approved the manuscript.

Competing interests
The authors declare no competing interests.

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