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Araştırma/Research

Characterization of Mortars from the Byzantine Basilicas in Side Archaeological Site, Türkiye

[SİDE ARKEOLOJİK ALANINDA BULUNAN BİZANS DÖNEMİ BAZİLİKALARINA AİT HARÇLARIN KARAKTERİZASYONU]

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Anahtar Kelimeler
kireç harçları; puzolan; koruma; Bizans; bazilika; Side

ABSTRACT

This study focuses on the characterization of mortar samples from a large Byzantine Basilica dating back to 5th/6th century CE and a smaller Basilica from late Byzantine period in Side, Türkiye. Samples were collected from various parts of the remains and investigated for their basic physical, physico-mechanical and compositional properties to gather essential information to be used in conservation/restoration interventions of the basilicas. For the determination of the basic physical and physico-mechanical properties density and porosity tests, ultrasonic velocity measurements were conducted. The characterization of the binder and aggregates were performed by petrographic analyses of thin sections and XRPD analyses. Mortars were treated with dilute HCl acid solution for the estimation of the binder/aggregate ratios. Particle size distribution of acid insoluble aggregates was evaluated by sieve analyses. Pozzolanic activity tests were carried out on the finer aggregates. Mortar samples were found to have relatively low density and high porosity. Binder is observed to be pure lime in all samples with high percentages of acid soluble parts. Aggregates of the samples were observed to be composed of mainly limestone and siliceous rock fragments. Use of crushed brick was observed in some of the mortar samples. Fine aggregates of mortars were found to have comparatively good pozzolanicity.

ÖZET

Bu çalışmada, Side, Türkiye'de bulunan MS 5./6. yüzyıla tarihlenen Bizans Bazilikası ve bu bazilikanın içinde bulunan ve geç Bizans dönemine tarihlenen küçük bazilikaya ait harç örneklerinin karakterizasyonu yapılmıştır. Bazilikalara ait kalıntıların farklı bölümlerinden alınan örneklerin; koruma ve onarım müdahalelerinde yardımcı olabilecek temel malzeme özelliklerinin belirlenmesi amacıyla fiziksel, fiziko-mekanik ve bileşimsel özellikleri araştırılmıştır. Fiziksel ve fiziko-mekanik özellikler, birim hacim ağırlığı ve gözeneklilik değerlerinin belirlenmesi, ultrasonik hız ölçümleri ile incelenmiştir. Bağlayıcı ve agregaların karakterizasyonu, ince kesitlerin petrografik analizleri ve XPRD analizleri ile gerçekleştirilmiştir. Harçların, bağlayıcı/agrega oranlarının yaklaşık olarak tespit edilmesi için seyreltik HCl asit çözeltisi kullanılmıştır. Asitte çözünmeyen agregaların parçacık büyüklüğü dağılımı elek analizleri ile değerlendirilmiştir. İnce agregalar üzerinde, puzolanik aktivite deneyleri yapılmıştır. Harç örneklerinin nispeten düşük yoğunluğa ve yüksek gözenekliliğe sahip olduğu bulunmuştur. Bağlayıcının, tüm numunelerde kireç olduğu ve örneklerin asitte çözünen kısımlarının yüksek oranda olduğu gözlenmiştir. Agregalarının ağırlıklı olarak kireçtaşı ve silisli kaya parçalarından oluştuğu belirlenmiştir. Harç örneklerinin bir kısmında terracotta kırıklarının kullanımına rastlanmıştır. İnce harç agregalarının görece olarak yüksek puzolanikliğe sahip olduğu görülmüştür.

1. Introduction

To propose proper repair materials (i.e., mortars/plasters) for the conservation works of historical structures, it is important to have detailed information about the physical/mechanical properties, raw material and compositional characteristics of the original materials used for the construction. The data gathered from the characterization studies should then serve as a basis to define the compatibility features between the original and the repair materials in terms of measurable parameters. Material investigation studies would also help to understand the construction techniques and technologies for a given structure of a certain period. In the case of historical buildings where lime mortars and plasters were extensively used, raw material characteristics and pozzolanic additives are of special importance, thus investigation of mortars and plasters samples from still standing structures are sources of valuable information for the preparation of durable and compatible repair mortars to ensure the success of the interventions.[1]

In this study, mortar samples belonging to a large basilica dating back to early Byzantine period and a smaller basilica build inside the large basilica during late Byzantine period in Side antique city was investigated in terms of basic physical/physico-mechanical and raw material characteristics to gain insight about their composition and unique features.

The larger basilica was built in the 5th/6th century, during which the city was the metropolis of Pamphylia, over the foundations of the Apollo and Athena temples. Some of the stones used for the construction of the basilica were observed to be gathered from the temple. While there are no definitive data to indicate the exact construction date of the smaller basilica, the architectural features such as cross vaults and the closed cross plan in the Grecian style suggest that it was likely built after the eighth century.[2] The aerial view of both the basilicas can be seen in Figure 1.

2. Materials and Methods

Mortar samples were taken from the apse of the larger basilica, one of the northern niches, the west and north walls of possible martyrion belonging to the larger basilica, and the north wall of the smaller basilica. Sample locations and nomenclature are given in Table 1.

Thin sections of mortar samples were prepared and examined under a microscope (Leica Z16) for petrographic analysis. Cohesion of the samples was assessed as per the recommendation Normal 12/83 issued by Italian CNR and ICR.[3] Bulk density and effective porosity tests were performed to investigate the basic physical characteristics of the samples using the standard gravimetric test method.[4] Bulk density values and the results of ultrasonic pulse velocity measurements (PUNDIT Plus Ultrasonic test equipment using 220 kHz probes) were used to estimate the modulus of elasticity (E) of the samples indirectly[5]

Percentages of acid soluble and insoluble parts were determined by treating the samples with dilute hydrochloric acid solution (5 %) and dissolving the acid soluble fractions. The granulometric analysis were performed on the acid insoluble parts using sieves with mesh sizes of 1000, 500, 250, 125 µm.

Mineralogical phases of the samples were determined by X-Ray powder diffraction spectrometry (XRPD) (Bruker D8 Advance Diffractometer with Sol-X detector) using Cu Kα radiation with the instrument operating at 40 KeV and 40 mA. The XRPD traces were recorded in the 3° - 70° 2θ range.

Finer aggregates smaller than 125 µm were examined for pozzolanic activity by comparing the difference in electrical conductivity (ΔEC, mS/cm) of the saturated Ca(OH)2 solution before and after immersion of the aggregates into the solution[6] using a conductometer (Metrohm Konduktometer E 382).

3. Results and Discussions

The color of the general matrix of the samples were observed to be white with only sample SB1 from the smaller basilica having a beige tint. Samples were resistant to crumbling against moderate force exerted by hand. Bulk density

1 Klisińska-Kopacz et al. 2010; Papayianni and Stefanidou 2003; Leslie and Gibbons 2000; Faria et al. 2008.

2 Kaderli 2017.

3 Normal 1983.

4 TC 25-PEM 1980.

5 ASTM D2845 2000.

6 Luxán et al. 1989.

values of mortars were found to vary between 1.39 and 1.77 g/cm3 and effective porosity values were determined to be in the range of 31-45% (Table 2). The relatively high effective porosity and low bulk density values observed were in agreement with the research on historical lime mortars in literature.[7] The modulus of elasticity results varied between 2.3 – 4.9 GPa for LB samples and 1.7 GPa for the SB1.

Petrographic analysis of the samples revealed that binder of the samples consists of calcite. Aggregates were determined to be composed of mainly limestone and siliceous rock fragments in all samples. Aggregates with rounded edges which might have been obtained from a source such as a river bed were detected in the samples. In addition, in the samples LB2 and SB1 quartz fragments with angular edges were identified indicating the possible use of crushed rocks in the mortar mixtures. In the matrix of the samples LB3 and SB1 some terra cotta fragments were also identified (Fig. 2). Thin section examinations of the samples LB3 and LB4 taken from the martyrion revealed the presence of carbonated lime lumps throughout the binder matrix. In addition, recrystallized CaCO3 crystals were also encountered in the fine cracks (Fig. 2).

Calcite and quartz were determined as the main mineral phases in all mortar samples by XRPD analysis. Dolomite was also observed in the SB1 sample (Table 4) as a differentiating mineral phase in the mortars of the two basilicas. Because of the high intensity peaks of calcite and quartz in the XRPD spectra, the possible pozzolanic reaction product peaks could not be observed.

The percentages of acid soluble and insoluble parts of mortars were calculated after treating the samples with a dilute solution of hydrochloric acid. The results are given in graphical form in Fig. 3. Acid soluble parts were observed to vary between 74 – 84 % for mortar samples. The high percentage of acid soluble parts for mortar samples indicated the use of calcareous aggregates at high percentages in the mortar mixes.

The particle size distribution of the acid insoluble parts of the samples showed that finer aggregates smaller than 125 µm constitute 4 – 26 % of the total acid insoluble part (Fig 4).

Pozzolanic activity tests were performed on the finer aggregates of the mortar samples having sizes smaller than 125 µm. According to the classification of Luxan et. al.,[8] if the variation in electrical conductivity is greater than 1.2 mS/cm, the material can be classified as a good pozzolan. The results of the pozzolanic activity tests for the finer aggregates of the examined samples showed that the change in electrical conductivity is at least 2.2 mS/cm and up to 6.5 mS/cm for LB1 to LB4 and 0.7 mS/cm for SB1. For samples with higher ΔEC values than 1.2 mS/cm (LB1-LB4), the finer aggregates can be designated as good pozzolans whereas for SB1 the finer aggregates can be categorized as variable pozzolans according to the classification of Luxan et. al.[9] These results also suggested the conscious use of the finer aggregates as pozzolanic additives to enhance the performance of the mortars. The pozzolanic characteristics of the aggregates of the larger and smaller basilica also highlights the difference in the selection of raw materials for the preparation of mortars in different periods.

4. Conclusions

The bulk density (1.39 – 1.77 g/cm^3) and porosity values (31 – 45 %) of the mortar samples investigated were observed to be in alignment with the values of historic lime mortars in the literature. Lime was assessed to be the binder in the examined mortars. The use of calcareous aggregates resulted in high percentages of acid soluble parts for the mortar samples meaning that the values do not indicate the absolute percentages of the binder. Aggregates were composed of mainly limestone and siliceous rock fragments in all samples with some samples having terra cotta fragments. Finer aggregates (<125 µm) from all mortars except the one from the small basilica showed relatively high pozzolanic activity suggesting their intentional use as additives. The difference in modulus of elasticity values between LB and SB samples also showed the effect of pozzolanic materials for enhancing the performance of lime mortars. Since ensuring the compatibility between the original and repair materials is crucial for the success of the restoration and conservation interventions, parameters such as

7 Belfiore et al. 2023; Bakolas et al. 1995; Caner and Güney 2018; Tenconi et al. 2018; Borges et al. 2014; Tunçoku and Caner-Saltık 2006; Franzini et al. 2000.

8 Luxán et al. 1989.

9 Luxán et al. 1989

the basic physical/physico-mechanical and raw material characteristics found in this study can be used as a basis for the design of repair mortars. To verify the final characteristics of repair mortar mixtures, the mortars should be evaluated in comparison with the original mortars.

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Fig. 1. Aerial view of the basilicas (Archive of Side Municipality, 2006).

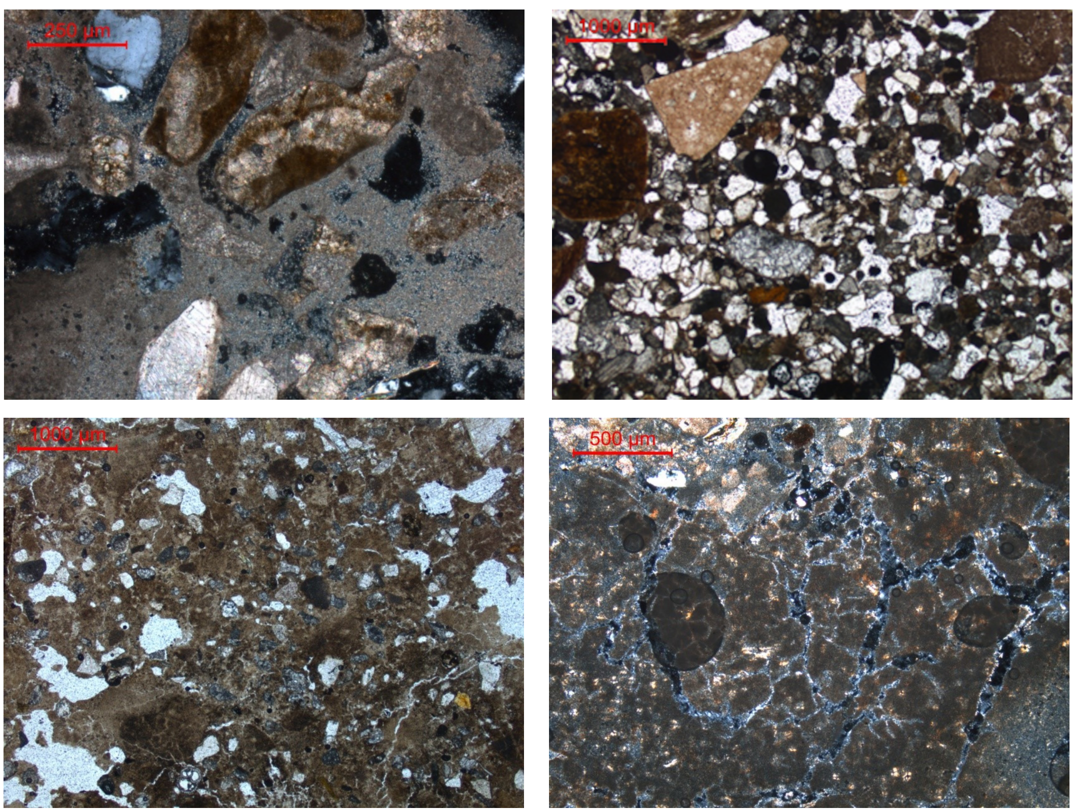

Fig. 2. Thin section images of limestone aggregates (LB2, top left) and terra cotta fragments (SB1, top right), lime lumps and drying cracks/recrystallization zones (LB3, bottom left, LB4 bottom right) in the samples.

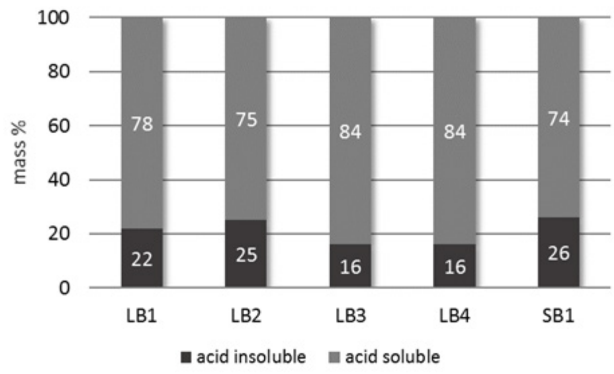

Fig. 3. Mass percentages of acid soluble/insoluble parts of mortar samples.

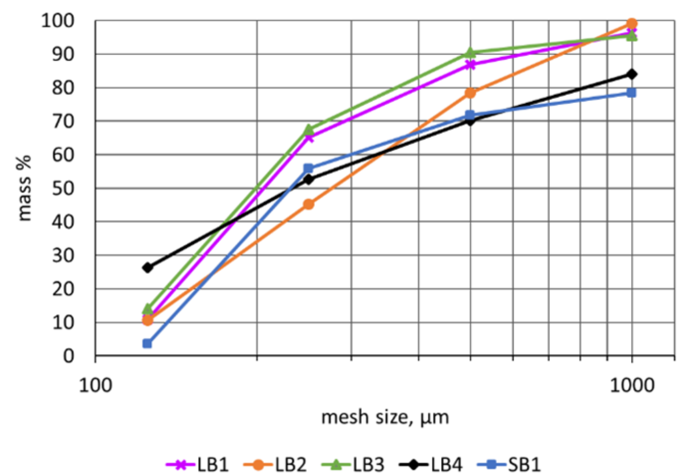

Fig. 4. Particle size distribution (cumulative) of acid insoluble parts of samples.

Table 1. Sample nomenclature and descriptions.

Sample	Description
LB1	Mortar from the apse (large basilica)
LB2	Mortar from one of the north niches (large basilica)
LB3	Mortar of the west wall of martyrion (large basilica)
LB4	Mortar from the north wall of martyrion (large basilica)
SB1	Mortar from the north wall (small basilica)

Table 2. Basic physical and physico-mechanical properties of the samples.

Sample	Bulk density (g/cm3)	Effective porosity (%)	Modulus of elasticity (Gpa)
LB1	1.77	33	2.3
LB2	1.39	45	2.5
LB3	1.53	41	4.9
LB4	1.73	35	2.6
SB1	1.69	31	1.7

Table 3. Pozzolanic activity values of acid insoluble parts (<125 µm).

Sample	Pozzolanic activity ΔEC, mS/cm
LB1	2.5
LB2	5.2
LB3	6.5
LB4	2.2
SB1	0.7

Table 4. Mineral phases in mortar samples determined by XRPD analysis.

Sample	Mineral phases
LB1	calcite, quartz
LB2	calcite, quartz
LB3	calcite, quartz
LB4	calcite, quartz
SB1	calcite, quartz, dolomite