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Properties of Roman Lime Mortars in Ancient Lycia Region

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Abstract. In this study, the characteristics of mortars used in some ancient Roman buildings located Xanthos (Antalya), Patara (Antalya) and Tlos (Muğla) in Southern Turkey were determined in order to define the properties of the new mortars to be used in the conservation works of the buildings. For this purpose, their basic physical properties, raw material compositions, mineralogical and microstructural properties were determined by X-Ray Diffraction, Fourier Transform Infrared Spectroscopy, Scanning Electron Microscopy coupled with X-Ray Energy Dispersive Spectroscopy. Analysis results indicated that the binder of the mortars composed of lime and fine aggregates have compact and uniform structure. The mortars are of low density and high porosity were produced from high calcium lime containing magnesium and natural aggregates. The lime and aggregate ratios were between 3/4 – 5/2 by weight and the aggregates with particle sizes greater than 1mm. composed the largest fraction of the aggregates. Lime composed of small size of micritic crystals due to use of aged lime putty. Aggregates were natural and mainly composed of quartz, albite, diopside and amorphous silica that may derived from the use of volcanic ash as pozzolans.

INTRODUCTION

The earliest civilizations such as Incas, Mayas, Chinese, Egyptians, ancient Greeks and Romans used lime mortars in the construction of brick or stone masonry and for rendering the masonry surfaces [1, 2]. Lime mortars are produced by mixing lime as binder and aggregates as filling material. The raw material of lime is calcareous stones which are primarily consisted of calcium carbonate ($CaCO_3$) minerals.

Lime is produced by heating calcareous stones that mainly contain calcium carbonate, forming calcium oxide (quicklime) and then slaking the quicklime with water [1, 3]. After the slaking process, the aging of lime putty is carried out under excess water for extended periods of time. Aging improves its plasticity and water retention capacity [4-7]. The use of the aged lime has been known since the Roman and succeeding periods. In Roman period, it was advised to keep lime at least for three years before using it [8].

Aggregates used as filling material can be classified as inert and pozzolanic aggregates [9]. Inert aggregates do not react with lime. However, the pozzolanic aggregates composed of amorphous silicates and aluminates react with lime in the presence of water [10]. The artificial pozzolans are heat treated clay materials such as bricks, roof tiles, etc.

Mortars produced by mixing lime putty with inert aggregates hardened by the carbonation of lime due to carbon dioxide in the air. Hydraulic mortars produced by mixing lime with the pozzolanic aggregates are hardened by both the carbonation of lime and the reaction between lime and aggregates in the presence of water. This reaction produces calcium silicate hydrates and calcium aluminate hydrates which give high strength to the lime mortars [9]. Romans successfully produced hydraulic lime by combining lime and natural or artificial pozzolans.

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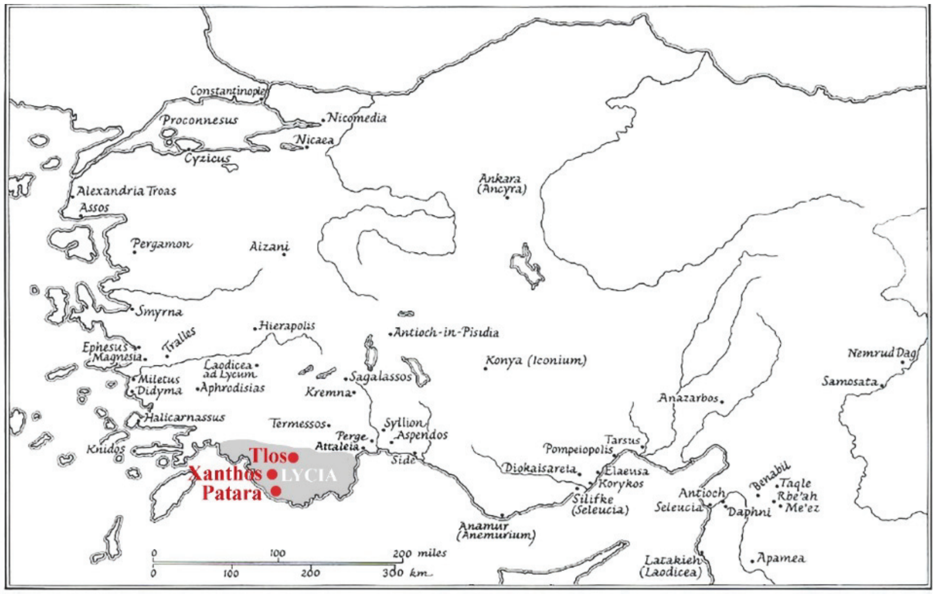


FIGURE 1. Location of Patara, Tlos and Xanthos [18]

The mortars used in many Roman ancient settlements such as Pompeii, Sagalassos, Herculaneum and in many Roman period structures such as the Pantheon, Colosseum and Tournai Cathedral were produced by using natural pozzolans [11-15].

Artificial pozzolans such as bricks and roof tiles were also used in the mortars and plasters of many Roman structures. Those mortars known as 'Cocciopesto' were used for walls as plasters exposed to severely humid environments such as baths or foundations [16-20].

Roman structures are witnesses of the ancient roman time in archaeological sites of Turkey. In this study, properties of Roman lime mortars from different type of buildings in three ancient settlements, Xanthos (Antalya), Patara (Antalya) and Tlos (Muğla), located in the Mediterranean region, southern Turkey were determined to provide historical information of the mortars and to choose the appropriate mortars to be used in the conservation works of Roman structures (Figure 1).

Three of ancient settlements (Xanthos, Patara, Tlos) represent the most unique architectural example of the ancient Lycian Civilization. Xanthos, located in Antalya Province, was the capital of ancient Lycia. The site may have existed during the Bronze Age or during the first centuries of the Iron Age. It illustrates the continuity and combination of the Anatolian, Greek, Roman, and Byzantine civilizations. In 1998, Xanthos was added to the UNESCO List of World Heritage Site with ancient city of Letoon. The tombs, the theatre, the bath, the agora and the basilica are the most important architectural examples of the city [21].

Patara, located in Antalya, was the principal port of Lycia. It was one of the main ancient maritime and trade centers of the eastern Mediterranean. The city gained further significance under Roman rule due to its natural secure harbour. Some significant finds at Patara are acropolis, baths, theatre, bouleterion and light house [22].

Tlos, located in Muğla Province, is another important city in Lycia region. Recent archaeological investigations show that this area was settled long before the Late Bronze Age. Tlos had metropolis status in the Roman period. On 2009, Tlos was added to the UNESCO Temporary List of World Heritage. The rock-cut tombs, the stadium, the agora, the baths, the city basilica and the theater are one of the outstanding examples of the city [23].

METHOD

This study began with the collection of mortar samples from different type of buildings in three ancient cities (Table 1). At least three lime mortar samples were collected from each ancient city when considering European standard (UNI EN 16085) for sampling [24]. Samples were labeled as first letter showing the ancient city they were taken from (X: Xanthos, P: Patara, T: Tlos) and second letter showing the name of function of structure (A: Agora, B: Bath, G: Gate, S: Stadium, T: Theatre).

Experimental study covers a series of laboratory studies in order to determine basic physical properties of mortars (density-porosity), raw material compositions (lime/aggregate ratios and particle size distribution of aggregates), mineralogical and chemical compositions of fine mortar matrices (less than 53 µm), fine aggregates and lime lumps as binder.

Density and porosity of the samples were determined by standard test methods [25]. Density is the ratio of the mass to its bulk volume and is expressed in grams per cubic centimeters (g/cm^3). Porosity is the ratio of the pore volume to the bulk volume of the sample and is usually expressed in percent (% volume).

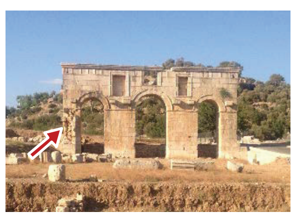
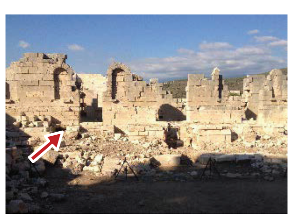
Measurement of density and porosity was carried out on two specimens of each sample. At first, samples were dried in an oven at low temperatures (40ºC) for at least 24 hours. Then they were weighed by a precision balance (AND HF-3000G) to determine their dry weights (M_{dry}). Subsequently, they were entirely saturated with distilled water in a vacuum oven (Lab-Line 3608-6CE Vacuum Oven). The bulk density of the sample was calculated from dry, saturated, and suspended weight in water by using the precision balance.

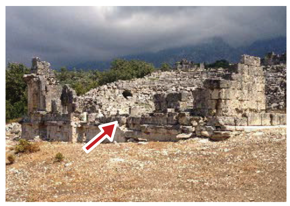
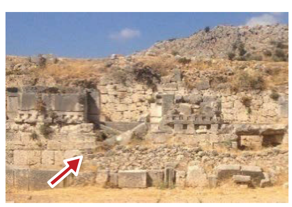
Raw material composition analyses were carried out in order to determine lime/aggregate ratios and the particle size distributions of the aggregates. Ratio of lime and aggregate used in the preparation of the mortars were determined by dissolving of carbonated lime ($CaCO_3$) from acid insoluble aggregates in dilute hydrochloric acid (5%) solution [26]. Determination of particle size distributions of aggregates was carried out by sieving them through a series of sieves having the sieve sizes of 53 µm, 500 µm, 1180 µm by using an analytical sieve shaker. Particles remained on each sieve surface were weighed by a precision balance and their percentages were calculated.

Mineralogical compositions of fine mortar matrices (less than 53 µm) composed of small grain sized silica and carbonated lime called as 'binder', fine aggregates (less than 53 µm) and white lumps in the mortar matrices were determined with X-Ray Diffractometer (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) analysis by Philips X-Pert Pro and by Spectrum BX II FTIR spectrometer (Perkin Elmer). The XRD spectra were collected at 45 kV and 40mA from 5° to 60° with 2θ and processed by using Philips X-Pert Pro software.

Scanning electron microscope coupled with X-Ray Energy Dispersive Spectrometry (SEM-EDS) were used to determine the elemental compositions of binders and white lumps. SEM-EDS analyses were performed by a Quanta 250FEG on pellets of binders prepared by pressing powder samples under 10 $tons/cm^2$ pressures. SEM-EDS analyses of white lumps were performed on broken surfaces of mortar samples. Results were taken from three different areas and average values were calculated. The elemental compositions were given in the form of oxides and normalized to 100%.

TABLE 1. Roman lime mortar samples from Patara, Tlos and Xanthos

Name of Sample	Location of Sample	Sample	Definition
PG (Patara-Gate)			Mortar from the rubble core of wall faced with ashlars
PCB (Patara-Central Bath)			Mortar from the rubble core of wall faced with ashlars
PT (Patara-Theatre)			Mortar from the wall comprised of ashlars

TB (Tlos-Bath)			Mortar from the wall comprised of ashlars
TS (Tlos-Stadium)			Mortar from the wall comprised of ashlars
TT (Tlos-Theatre)			Mortar from the wall comprised of ashlars
XA (Xanthos-Agora)			Mortar from the wall comprised of mortared rubble throughout
XB (Xanthos-Bath)			Mortar from the rubble core of wall faced with ashlars
XT (Xanthos-Theatre)			Mortar from the wall comprised of mortared rubble throughout

X: Xanthos P: Patara T: Tlos A: Agora B: Bath G: Gate S: Stadium T: Theatre

RESULTS AND DISCUSSION

Physical properties of mortars: Lime mortars collected from three ancient Roman settlements are white and grayish, depending on the type of the aggregates they contain. All mortars are of low density and high porosity. Density and porosity values of mortars are between 1.20 and 1.97 g/cm^3 and 20 and 53 %, by volume, respectively. These values were almost in the same ranges with other mortars of some Roman buildings [19-20, 27-29].

Raw material compositions of mortars: Compositions of mortars were defined by lime/aggregate ratios and particle size distribution of aggregates by weight. Lime/aggregate ratios were in the range of 3/4 - 5/2. Aggregates which had particle sizes greater than 1180 µm constituted the major fraction of total aggregates in all mortar samples. Raw material compositions of investigated mortars are similar to Roman lime mortars found in different buildings in Turkey [19, 30], Italy [27, 31] and Spain [32]. These results show the use of similar raw material compositions in production of mortars in some Roman structures in Italy, Spain and Turkey.

Mineralogical and chemical compositions of binders: Binder of the mortars composed of carbonated lime and small size of aggregates less than 63 µm were hard and compact due to strong adherence between aggregates and lime [33-34]. The cohesion between fine aggregates and lime was strong in all mortar binders.

In this study, mineralogical compositions of binders were determined by XRD analysis. In the XRD patterns of binder strong peaks of calcite and quartz and weaker peaks of hematite, dolomite, albite, anorthite minerals were indicated (Table 2 and Figure 2). Calcite was originated from carbonated lime, while quartz and other minerals were aggregates.

The oxide compositions analysis of binder of mortar matrices by EDS analysis indicated that nearly all binders were composed of high amount of CaO and SiO_2 and moderate amount of Al_2O_3 and MgO (Table 2). These results showed that binders composed of high amount of carbonated lime containing magnesium and aggregates composed of mainly silica, alumina and iron.

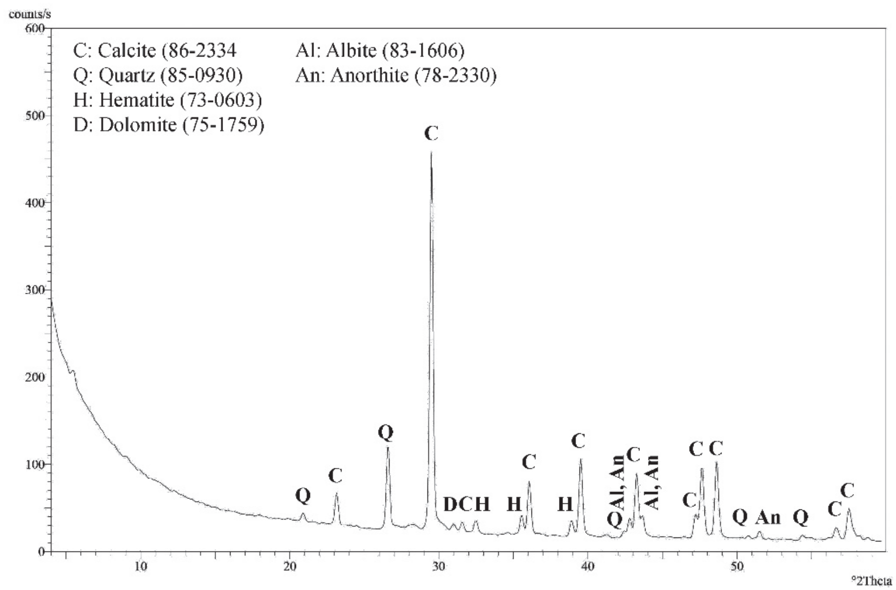


FIGURE 2. XRD patterns of binder (TT)

TABLE 2. Mineralogical and chemical compositions of binders

Sample Code	Mineralogical Composition: Minerals	Chemical Composition: CaO [%]	MgO [%]	SiO_2 [%]	Al_2O_3 [%]	FeO [%]	Other [%]
PG	C, Q, Al, An, H, D	66,16±0,82	3,25±0,47	22,55±1,15	5,98±0,74	2,06±0,36	-
PCB	C, Q, Al, An, H, D	85,15±1,41	1,59±0,14	10,62±1,08	2,63±0,46	-	-
PT	C, Q, Al, An, H, D	68,43±2,35	2,56±0,63	20,27±2,36	6,29±0,23	2,46±0,32	-
TB	C, Q, Al, An, H, D	77,94±1,12	10,11±0,68	7,11±0,76	10,11±0,45	-	2,80±0,46
TS	C, Q, Al, An, H, D	77,23±4,45	6,86±0,83	11,86±1,91	2,47±0,21	1,83	1,46±0,46
TT	C, Q, Al, An, H, D	79,73±1,18	9,05±0,38	8,85±0,42	1,93±0,10	1,29	-
XA	C, Q, Al, An, H, D	61,06±3,52	4,69±0,79	22,95±1,54	6,37±0,64	3,91±0,48	1,03±0,36
XB	C, Q, Al, An, H	68,70±3,15	3,01±0,71	22,03±1,50	4,87±0,13	1,68±0,54	0,84
XT	C, Q, Al, An, H, D	68,34±1,41	3,39±0,28	19,80±0,64	4,96±0,75	3,50±0,26	-

C: Calcite D: Dolomite Q: Quartz Al: Albite An: Anorthite H: Hematite

Mineralogical and chemical compositions of lime: White lumps found as round and soft fragments in the mortars were accepted as being the carbonated lime particles used in the production of mortars. Hence, their mineralogical, microstructural and chemical properties give information about the lime used in the preparation of the mortars. Mineralogical compositions of white lumps determined by FTIR analysis instead of XRD due to the small amount of samples. These analyses show that they were composed of calcite crystals and showed the characteristics of $CaCO_3$ bands at 1,438 cm^{-1} (C–O stretching), 873 and 713 cm^{-1} (C–O bending) (Figure 3).

Their chemical and micro structural analysis carried out SEM-EDS analyses indicated that they were composed of small-sized micritic calcite crystals containing high amount calcium and low amounts of magnesium and silicon (Table 3 and Figure 4). These results show that the lime used in the production of mortars was high calcium lime obtained from almost pure calcareous stones. Similar results have also been found from the mortars used in some Roman structures [20, 27, 35].

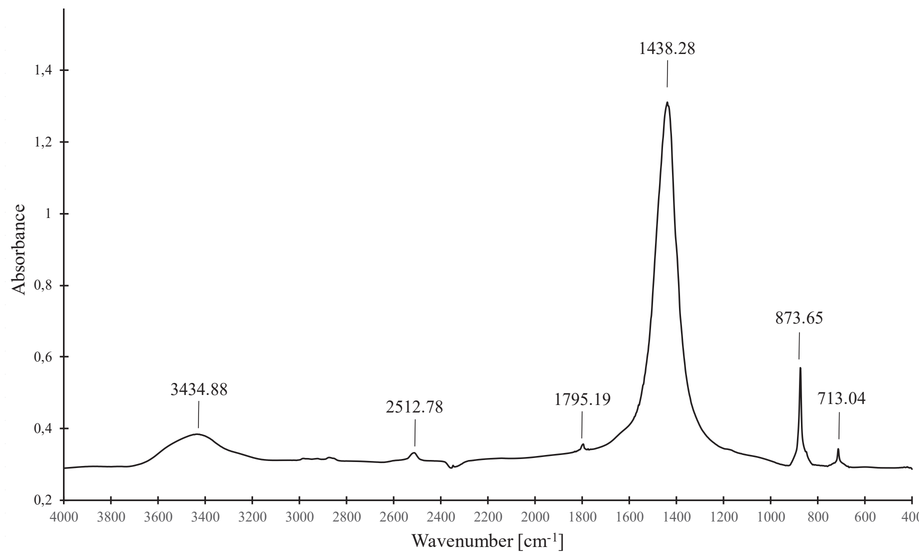
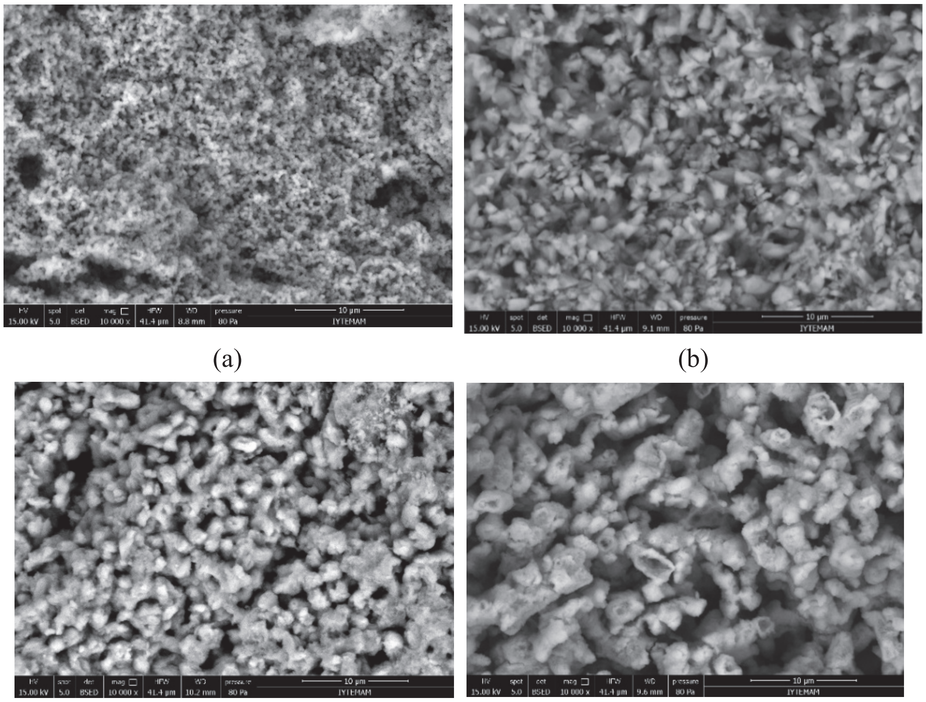


FIGURE 3. FTIR spectra of white lump (PT)

TABLE 3. Mineralogical and chemical compositions of white lumps

Sample Code	Mineralogical Composition	Chemical Composition CaO [%]	MgO [%]	SiO_2 [%]	Al_2O_3 [%]	FeO [%]	P_2O_5 [%]
PG	Calcite	92,45±3,68	1,06±0,50	3,79±1,89	2,19±0,57	1,52	-
PT	Calcite	99,57±0,74	-	1,29	-	-	-
TB	Calcite	90,63±1,37	4,12±0,75	1,98±0,08	3,27±0,61	-	-
TS	Calcite	92,10±1,85	4,50	2,15±0,37	1,25±0,31	-	-
TT	Calcite	91,31±1,16	4,10±0,77	3,15±0,07	0,85±0,12	-	0,88±0,14
XA	Calcite	98,50±0,45	-	0,91±0,20	0,90±0,30	-	-
XB	Calcite	97,74±0,60	0,63±0,01	1,25±0,28	0,58±0,22	-	-
XT	Calcite	97,74±2,01	-	1,12±0,02	2,28±0,62	-	-



(a) (b)

(c) (d)

FIGURE 4. SEM images of white lumps composed of carbonated lime crystals (10000) of (XA) (a), PT (b), TB (c) and TS (d)

Mineralogical and chemical compositions of aggregates: Mineralogical compositions of fine aggregates in the mortars were determined by XRD analyses. Their XRD results show strong peaks of quartz (SiO_2), albite (Na ($AlSi_3O_8$) and anorthite ($CaAl_2Si_2O_8$) diopside ($MgCaSi_2O_6$) and the diffuse band between 20-30 degrees for the amorphous silica. Amorphous silica that may derived from the use of volcanic ash as pozzolan (Figure 5). Chemical compositions of fine aggregates in the Roman lime mortars were determined by SEM-EDS analyses. The results of the analyses revealed that aggregates were mainly composed of high amount of SiO_2, moderate amounts of Al_2O_3, and low amounts of FeO, MgO, CaO, P_2O_5, K_2O (Table 4).

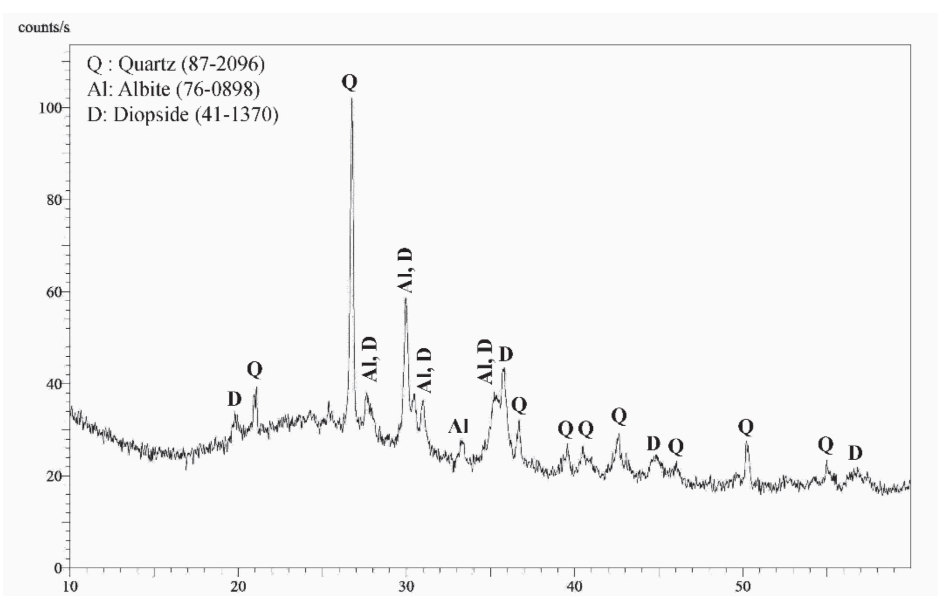


FIGURE 5. XRD patterns of aggregate (PCB)

TABLE 4. Chemical compositions of aggregates determined by SEM-EDS

Sample Code	SiO_2 [%]	Al2O3 [%]	FeO [%]	MgO [%]	CaO [%]	P_2O_5 [%]	K_2O [%]	Other
PT	87,80±1,89	6,32±1,18	2,33±0,08	1,03±0,14	1,02±0,24	-	1,31±0,45	0,54
TB	83,42±2,53	8,13±1,51	3,86±0,40	2,07±0,29	1,55±0,42	-	1,49±0,25	-
TS	69,41±1,38	14,66±0,71	3,91±0,68	3,48±0,44	3,42±0,48	3,59±0,56	1,55±0,09	-
TT	66,03±1,78	10,92±0,84	3,69±0,31	6,53±0,74	7,05±0,94	4,10±0,30	1,68±0,35	-
XA	56,05±3,26	13,84±1,13	9,55±1,35	6,84±1,23	10,43±2,26	3,29±0,42	-	-
XB	66,35±0,74	13,52±0,18	6,50±0,76	4,51±0,29	7,51±0,42	1,63	-	2,53±0,09
XT	67,25±1,03	12,40±0,63	8,42±0,48	5,34±0,46	1,59±0,49	2,04±0,20	2,43±0,06	0,79

CONCLUSION

In this study, the characteristics of lime mortars from Roman period buildings in three ancient cities in southern Turkey were determined. Roman mortars have a low density and high porosity. Mortars were mainly produced by using high calcium lime containing magnesium and natural aggregates composed of mainly silica and alumina. The lime and aggregate ratios were between 3/4 – 5/2 by weight and the aggregates with particle sizes greater than 1mm. composed the largest fraction of the aggregates.

Lime used as a binder was obtained from almost pure calcareous stones containing magnesium. Lime composed of small size of micritic crystals due to use of aged lime putty.

Aggregates were natural and mainly composed of quartz, albite, diopside and amorphous silica that may derived from the use of volcanic ash as pozzolan. They were mainly composed of a high amount of SiO_2, Al_2O_3 and moderate amount of Fe_2O_3, and CaO. The binder of the mortars composed of lime and fine aggregates have uniform structure. These results are important to define the properties of the new mortars to be used in the conservation works of Roman period structures.

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