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PHYSICO-CHEMICAL AND PETROGRAPHICAL STUDIES OF OLD MORTARS AND PLASTERS OF ANATOLIA

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ABSTRACT

The problems which arise from the use of today mortars and plasters because of their easy applicability for restoration work do irreparable damage to the historical monument. Therefore the detailed scientific data are necessary in order to choose the right materials for restoration and also for the instructions which will be followed during the conservation of monuments. In this study, the results of visual, sieve, porosity, thin section and X-ray diffraction analyses of mortars and plasters from Roman, Byzantine and Ottoman period of Anatolia are given. © *1997 Elsevier Science Ltd*

Introduction

It is the fact that a right characterization of old mortars and plasters from different monuments and periods can be succeeded by and interdisciplinary studies which should include chemical, physical, petrographical, minerological and biological examinations of materials and also numereous of samples should be analysed for the statistical data. Many of the research work has been carried out to characterize the original mortars and plasters by using traditional wet chemistry [1-4] and sophisticated instrumental methods [5-7] though, physico-chemical analysis of mortars and plasters is incomplete due to the diffuculties which arise from the inadequate sample size. Physico-chemical characteristics such as microporosity, pore structure, density, aggregate size, etc. of old mortars and plasters are very important with respect to the mechanical strength of a monument in restoration and conservation procedure [8,9]. Physical test methods are very few so that either the characteristic parameters or their applicability limit are not completely identified [10].

The present study is dealing with the minerological, petrographical and physico-chemical indentification lime based mortars and plasters of Roman bath (RB), Tahtakale bath (TB) and Esekapi Madrasah (EK). The RB is from the Roman ages of Anatolia and is located in Ankara (capital of Turkey). The RB had restoration during the Byzantine period and then a restoration was done by Turkish restorers in 1952. The TB and EK are from Ottoman period

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of Anatolia and are located in Istanbul. The EK was and old Byzantine church but, during Süleyman the first (15th century) it was converted to a mosque and madrasah which is a school by the world famous architect, Sinan. The classroom and cells of madrasah are domed while the roof of mosque is damaged at the present state. The TB was built in 1453 at Byzantine harbour, Nerion, after Istanbul was conquered by Mehmet the second. Frigidarium of men's division and caldarium of ladies division have been damaged by the fire in 1726. The original plan of building was altered due to the new functions though, a division with dome remained well conserved and restored properly.

Materials and Methods

The mortar (M) and plaster (P) samples were obtained from different points of monuments in order to show analogous or different characteristics of materials. 79 TB samples were available during the restoration of the monument but, in the case of the RB and EK the number and the size of the samples were limited. 19 samples for RB and 51 samples for EK were taken from the damaged part of the monument. The abbreviations given by the sample number indicate. RB: Roman Bath, TB: Tahtakale Bath, EK: Esekapi Madrasah, M: Mortar and P: Plaster.

Visual Examination. All mortar and plaster samples were examined with respect to the type, size and amount of aggregates, colours and organic matter, etc. For the sake of brevity, the results of chosen samples are shown in Table 1.

TABLE 1

Visual and Porosity Characteristics of Mortars and Plasters.

Sample	Colour	Aggregates Type	Organic Matter	Micro Porosity $cm^3.g^{-1}$	%	Specific Pore Volume, $cm^3.g^{-1}$	Total Volume area $m^2.g^{-1}$	True Density $g.(cm^3)^{-1}$	Apparent Density $g.(cm^3)^{-1}$
TBP 13a	Pink	Broken brick	+	0.269	62.7	--	--	--	--
TBP13b	White	Sand	-	0.338	70.9	0.477	--	--	--
TBP 23	Pink	Broken brick+Sand	+	0.212	94.0	0.228	32.57	1.57	2.44
TBM 64	Pink-White	Sand	+	0.364	92.6	0.393	21.92	1.25	2.47
TBM 66	Pink	Sand	-	0.210	92.4	0.228	--	--	--
EKP 13b	White	Broken brick+Sand	-	0.302	86.9	0.347	4.95	1.36	2.57
EKP 31a	Red	Broken brick+Sand	+	0.410	88.4	0.464	--	--	--
EKP 32	White	Broken brick+Sand	-	0.283	81.9	0.345	--	--	--
EKM 8	Yellow -White	Sand	-	0.169	71.8	0.235	--	--	--
EKM 34	Pink	Broken brick	-	0.270	92.7	0.292	5.28	1.45	2.52
EKM 50	Red	Broken brick	-	0.239	88.8	0.269	--	--	--
RBP 7	Pink	Broken brick+Sand	-	0.065	78.0	0.083	--	--	--
RBM 9	Grey	Sand	-	0.054	74.0	0.073	4.44	2.01	2.35
RBM 14	Red-Pink	Broken brick+Sand	-	0.310	88.6	0.350	--	--	--

a: Inner, b: Outer layer of plaster

TABLE 2

The Results of XRD Analysis.

Mineral	Formula	TBM1	TBM2	TBP2	EKM1	EKM2	EKP1	RBM1	RBP1	RBP2
Quartz	SiO_2	+	+	+	+	+	+	+	+	+
Calcite	$CaCO_3$	+	+	+	+	+	+	+	+	+
Anorthite	$CaAl_2Si_2O_8$	-	-	+	-	+	-	-	-	-
Microline	$KAlSi_3O_8$	-	+	-	+	+	-	-	-	-
Albite	$NaAlSi_3O_8$	-	-	-	-	-	-	+	+	-
Andesine	$(NaCa)(SiAl)_4O_8$	+	+	-	-	+	+	-	-	+
Orthoclase	$KAlSi_3O_8$	-	-	-	+	-	-	-	-	-
Muscovite	$(KNa)Al_2(SiAl)_4O_{10}(OH)_2$	+	+	+	+	-	+	+	+	-
Gypsum	$CaSO_4.2H_2O$	+	-	-	+	+	-	+	+	-
Vermiculite	$(MgFe)_6(SiAl)_4O_{10}(OH)_8$	-	-	-	-	-	-	+	+	+

Sieve Analysis. The weighted amount of the samples was introduced to react, with 10% of HCl solution for 20 hours then filtered, washed with distilled water and dried at 105°C for 24 hours. The insoluble residue was sieved through 1000μ, 500μ, 250μ and 125μ mesh in the given order. The granulometric aggregate size distribution of insoluble part of samples are discussed in the Result and Discussion section.

X-Ray Diffraction Analysis. Mineralogical compositions of mortars and plasters were determined by XRD powder technique using CuK_α radiation. Results were given elsewhere [11] (Table 2). Phillip XL 30, X-Ray Diffractometer was used for the measurement.

Petrographic Analysis. The textural structure of mortars and plasters were determined by Thin Section Analysis (TSA) as well as the mineral nature. For the TSA procedure, the samples were blocked in an epoxy resin under vacuum and were made thinner to 30μ with low speed cutter, (Buehler, Isomet). Then the sample slices were adhered on a microscope slide and polished with 3μ. 1μ and 0.25μ silicon carbide dust respectively. Finally, all samples were examined under Polarizan microscope (James Swift) and thin section photographs were taken. Thin Section photographs are shown in Figures 1-14.

Porosity Measurement. The deterioration reactions of old monuments due to the environmental conditions closely depend on the surface area hence, porosity of construction materials. Therefore, porosity determinations give valuable data for structural characteristics of old monument. Porosity measurements of mortars and plasters were carried out with Mercury Pressure Porosimeter (Micromeritics Porsizer 9320) on samples which have sized as test

FIG. 1.
TSA.TBM No:64, Broken Brick.

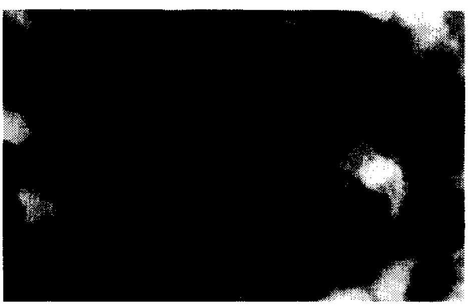

FIG. 2.
TSA.TBM No:45, Pebble like aggregates.

FIG. 3.
TSA.TBP No:10, Fiber Additives.

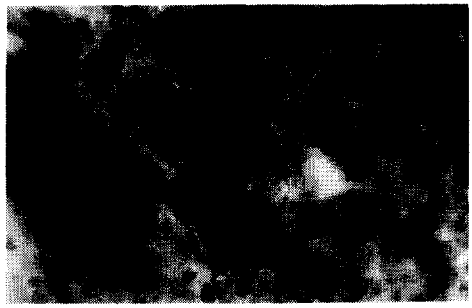

FIG. 4.
TSA.TBP No:27a, Inner Layer, Aggregates.

pieces of (2 cm)^3. The usual technique was to measure the reduction in the level of mercury in a bulb containing the sample as pressure was progressively applied. Porosity Curves (PC) are given in Table 1 and Figures 15-18.

Results and Discussions

Thin Section analysis of mortar and plaster samples of RB showed that they were quite homogenous with most of the mass made up of quartz, calcite and very little plagioclase and feldspar. The quartz content was rather large as it was seen in Fig.13. The aggregate content was over 65% and were very close to each other (Fig.12). Quartz and calcite grain sizes ranged from 0.1 mm to several mm. and their shapes were angular with low spherical property. Just a few samples of RB had crushed and ground brick pieces (Fig.14). Mortars and plasters samples of TB and EK had very different characteristics from those of RB samples. Mortar samples mostly consisted of little amount of quartz, carbonates, calcites (Fig.7), plagioclase and feldspar (Fig.2). The apperance of carbonate and broken shells indicated that these aggregates were prepared by crushing the "Küfeki" stone, which is a kind of calcareous sedimentary local stone. The mortar samples of domes (outside) were different from the wall samples. They did not contain any crushed bricks and stone pieces, but large amounts of (over 65%) pebble like aggregates where their size ranged from 0.3 to 3 mm. Although the tows included mostly in the inner plaster layer (Figures 3, 9), some of mortar samples had also these kind of additives.

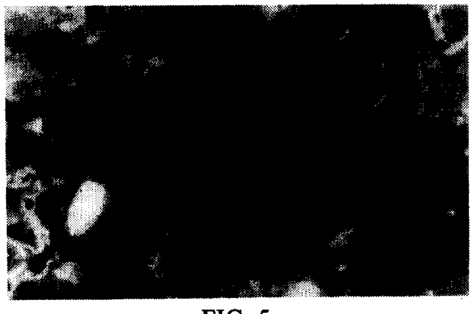

FIG. 5.
TSA.TBP No:27, Inner and Outer Layer.

FIG. 6.
TSA.TBP No:27b, Deterioration as Flaks and Crust.

FIG. 7.
TSA.EKM No:19, Quartz, Carbonate and Calcite Minerals.

FIG. 8.
TSA.EKM No:8, From Dome, Pebble like Aggregates.

The inner layers of plaster samples were very similar to the mortar samples. The only difference were the size of aggregates, which changed from very fine to 2 mm (Fig.4). The outer white layer was soft and very homogeneous. The amount of aggregates were not more than 15% and the sizes were less than 0.1 mm (Fig.6). The lines between inner (pink) and outer (white) plaster layers of TB and EK samples showed different characteristics. The lines of samples were straight (Fig.5) while the lines of EK were uneven (Fig.10). This difference should have been caused by different application technique of plaster layers. The outer layer of TB should have been applied after setting the inner layer while before setting in EK case. The white, outer layer plasters were highly affected from air pollution. The flakes and crusts composed gypsum, soot and dust, were originated by environmental pollution. These affects were observed at both, TB and EK white plaster samples (Figures 6,11).

In general the distrubution of aggregates in mortar sample were mainly silicates and carbonates. The binders contained broken pieces of bricks and brick dust as pouzzolanic filler. The diameter of aggregates were smaller and the amount of aggregates were less in plaster samples.

The XRD data showed that the TB and EK samples consisted of mainly calcite, quartz, lesser amount of anorthite and muscovite. The major content of RB samples were quartz, calcite, albite and small amount of muscovite and vermiculite. Gypsum found in some RB and EK samples should have come from environmental pollution.

The porosity data shown in Table 1 exhibited that, in general, the RB samples were quite different than TB and EK samples. The RB samples from the deeper part of the monument

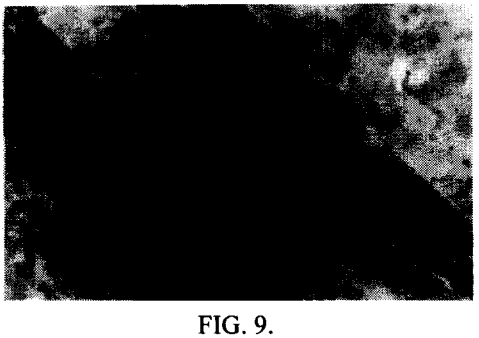

FIG. 9.
TSA.EKP No:42, Fiber Additives.

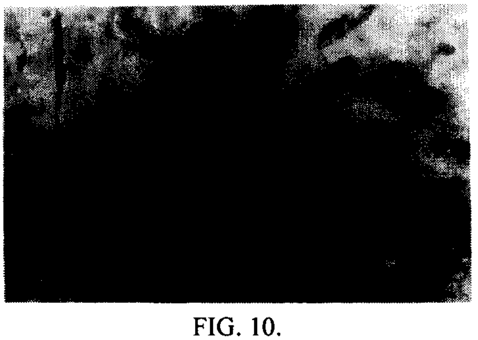

FIG. 10.
TSA.EKM No:31, Inner and Outer Layer.

FIG. 11.
TSA.EKP No:6, Flakes and Crust in Outer Layer.

FIG. 12.
TSA.RBM No:9, Close Aggregates.

had similar specific pore volume (SPV) with TB and EK samples (Fig.18). The low SPV value of RB samples (0.08-0.09 cm^2-g^{-1}) did not permit to the high concentration of water soluble salts which were outer origin thus, the RB monument had more efflorencence than the TB and EK monuments. The TB and EK samples showed nearly the same SPV and pore size distribution (Fig.15, 17). The inner (pink) layer of the plaster samples exhibited different characteristics due to being from interior and exterior part of the monuments. The pink layer of plaster samples, from inside (Fig.16) showed rather homogeneous size distribution while those from outside exhibited cumulation. The SPV of white plaster layers of TB and EK were analog to each other and had rather homogeneous size distribution. The mortar samples of domes were different from all other samples so that pore size distribution of dome samples were homogeneous but, SPV of the samples were lower (0.16-0.26 cm^3g^{-1}) than the other mortars and the pink plaster samples. The high SPV value of mortar and plaster samples of TB and EK tolerated the volume expansions of water soluble salts during the conversion reactions of freezing-melting or crysallization-solubilization. There were not significant difference in the densities of all samples.

The results of sieve analysis showed that the aggregate size distribution of TB and EK were quite analog, but the amount of aggregate is less than the standard amount given within the same sieve pores (9). Materials which have smaller diameter than 125μ are higher in TB and EK samples than RB samples. Although the aggregate size of RB were smaller than the TB and EK aggregates, they show homogeneous distribution in the studied sieve ranges.

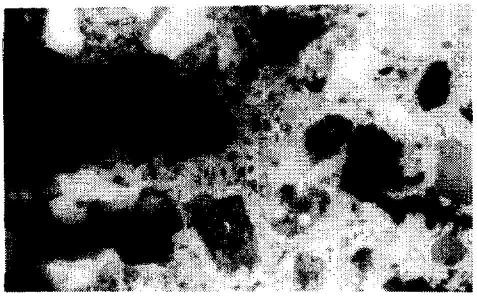

FIG. 13.
TSA.RBM No:6, Quartz in Floor-screed.

FIG. 14.
TSA.RBM No:7, Broken Brick.

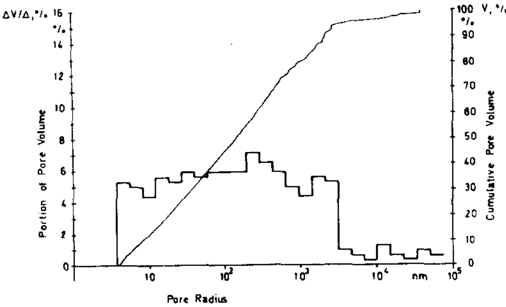

FIG. 15.
PC.TBM No:66.

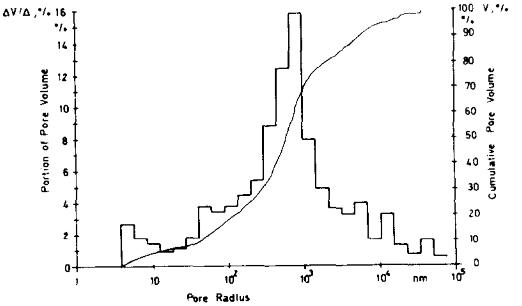

FIG. 16.
PC.EKP No:32.

Conclusion

The mortar samples of TB and EK composed of uniformly distributed binder, fine and coarse aggregates of various colours and textures. Crushed and partly ground bricks (artificial pouzzolanic material) were mostly used as aggregates. Plaster samples of TB and EK contained large amount of tows as organic material. There were two layers of plaster, the inner layer was similar with the mortars in composition, colour and texture. The undercoat layer was rather thin and consisted of very fine aggregates and binders.

The mortar samples of RB were mostly composed of randomly distributed limeless binder, fine and coarse, pebble like aggregates in various texture, which showed modern mortar characteristics. This might be due to the modern cement use in the previous restoration (1952) of monument.

The TB and EK mortars were generally called "Khorosany Mortar" after Roman period. Vitrivius explaned such composition as "Roman mortar" in his book [12]. This type of mixture has been in use since the antiquity, and generally, it has not been damaged by the water soluble salts [13].

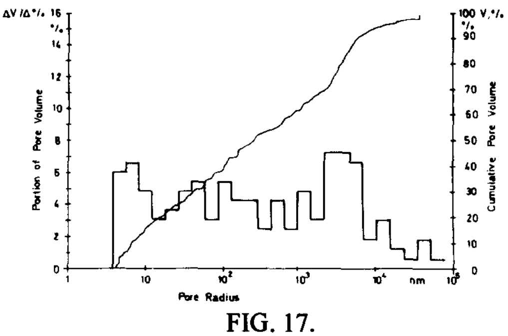

FIG. 17.
PC.EKM No:8.

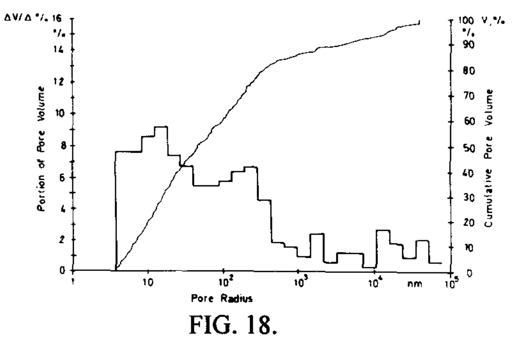

FIG. 18.
PC.RBM No:14.

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