EXAMINATIONS ON TECHNOLOGICAL CHARACTERISTICS OF MUD-BASED CONSTRUCTION MATERIALS IN ULUCAK HOYUK NEOLITHIC SETTLEMENT

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ABSTRACT

EXAMINATIONS ON TECHNOLOGICAL CHARACTERISTICS OF MUD-BASED CONSTRUCTION MATERIALS IN ULUCAK HOYUK NEOLITHIC SETTLEMENT

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The main concern of the study is the definition of mud-based materials technologies belonging to Ulucak Höyük (İzmir) Neolithic settlement (Vth level:6400-6000 BC and IVth level: 5990-5660 BC). Representative samples were collected from the eight different mud masonry houses that were unburnt, partially and fully burnt mudbricks, interior/exterior mud plasters, and floor covering mud mortars. Their laboratory tests were based on raw materials analyses composed of compositional properties of mud mixtures and mineralogical composition of clay content together with basic physical and physicomechanical properties analyses. Mineralogical composition of raw materials was identified by the cross-section and thin section analyses using an optical microscope, X-ray diffraction, FTIR analyses.

The presence of a mica-illite group of clay with or without kaolinite, clay-sized $CaCO_3$ particles in clay mixture and coarse gravel in a certain amount are the main characteristics that highlight the qualified composition of mud-based construction materials belonging to Ulucak Höyük Neolithic settlement and the advanced mud-materials technology achieved in that period. The mud-based brick, floor covering mortar and interior plaster have different bulk densities in coherence with their particular grain size distribution. The clay type, percentage of clay content, percentage of $CaCO_3$ in clay content, presence of kaolinite in clay content are the indicators introduced in the study in order to examine the neighbouring clay and adobe soil resources and to discuss the archaeological questions on social life in relation to settlement period and building construction. The data achieved are guiding for planning conservation approaches on mud materials of the settlement.

Keywords: Mudbrick, Mud Plaster, Raw Material and Compositional Properties, Ulucak Höyük, Neolithic Settlement

ÖZ

ULUCAK HÖYÜK NEOLİTİK YERLEŞİMİNDEKİ KERPİÇ YAPI MALZEMELERİNİN TEKNOLOJİK ÖZELLİKLERİNİN İNCELENMESİ

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Çalışmanın amacı, Ulucak Höyük (İzmir) Neolitik yerleşiminin V. (MÖ 6400-6000) ve IV. (MÖ 6000-5700) tabakalarına ait kerpiç yapı malzemelerinin teknolojilerinin tanımlanmasıdır. Yanmamış, kısmen yanmış ve tamamen yanmış halde olan kerpiç tuğla, taban sıvası ve iç/dış duvar sıvası örnekler bu tabakaları temsil eden sekiz farklı konuttan toplanmıştır. Laboratuvar testleri, kerpiç karışımlarının bileşim özellikleri ve killerin mineralojik bileşimini içeren hammadde analizleri ve kerpiç malzemelerin temel fiziksel-fizikomekanik özelliklerinin belirlenmesi amacıyla yapılmıştır. Hammaddelerin mineralojik bileşimi kalın kesit ve ince kesit optik mikroskop görüntü analizleri, X ışını kırınımı ve FTIR analizleri ile belirlenmiştir.

Ulucak Höyük Neolitik yerleşiminin kerpiç yapı malzemelerinin ana karakteristikleri; kaolin içeren ya da içermeyen mika-illit grubu killerin varlığı, kil boyutunda $CaCO_3$ parçacıklarının bulunması ve malzemelerde belirli miktarda iri tanelerin bulunmasıdır. Bu özellikler, kerpiç malzemelerin nitelikli bir bileşime sahip olduğunu ve o dönemde gelişkin bir kerpiç malzeme teknolojisinin varlığını göstermektedir. Kerpiç tuğla, taban sıvası ve iç sıva malzemeler, tane boyu dağılımları ile uyumlu farklı birim hacim ağırlıklarına sahiptirler. Bölgedeki kil ve kerpiç toprağı kaynaklarını incelemek ve dönemin sosyal yaşamı ile ilgili arkeolojik soruları zaman ve mekân farklılıkları çerçevesinde tartışmak amacıyla çalışmada ortaya çıkarılan göstergeler kil cinsi, kil yüzdesi, kil bileşimi içerisindeki $CaCO_3$ oranı ve kaolin killerinin varlığıdır. Elde edilen veriler, yerleşimin kerpiç malzemelerinin koruma yaklaşımlarının planlanmasında rehberlik etmektedir.

Anahtar Kelimeler: Kerpiç Tuğla, Kerpiç Sıva, Ham Madde ve Bileşim Özellikleri Ulucak Höyük, Neolitik Yerleşim

To My Family

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CHAPTER 1

INTRODUCTION

The archaeological settlement is the most inclusive of the material remains for the archaeology discipline, which tries to understand the human and its past from the remnants of the present day. The settlement is the whole of dwellings and open fields as well as the component of a larger structure with the surrounding environment and other contemporary settlements. Visual architectural characteristics of the settlement such as open spaces, the distribution of the dwellings, the function of the dwellings and their relationship with each other give a lot of clues about the Neolithic social life which need to be supported and enriched by the characterization of those cultural materials that make them up (Karul, 2017).

Neolithic period is characterized by sedentism, and in that period, the architectural changes on the structure, shape, and type of dwellings were observed through thousands of years (Love, 2013). Different types of materials such as stone, clayey soils, or wood were used as a raw material of dwellings. The usage type of those raw materials makes an important contribution to the character, mood, and state of the dwellings; therefore, they are the essential topics in architectural expression. In addition to this, the dwellings' materials and the choices of their independent production techniques provide a lot of knowledge on the Neolithic social life in terms of material culture, shared resources, organization of labor, and variations in construction practices (Love, 2012).

Archaeological and ethnographic examples point to the fact that in Anatolia, the choice of building materials did not only depend on the availability of raw materials, but also cultural factors played important roles (Karul, 2017; Love, 2012). In addition, the examples in the first usage of different raw materials show that the natural material sources were not randomly selected. Raw materials with specific properties might have been preferred, and some processes could be performed for the preparation of building materials (Karul, 2017).

The usage of mud materials such as mudbricks, mud plasters, mud mortars etc.in dwellings of settlements in Anatolia and Levant dates back to 9500 BC (Cauvin, Hodder, Rollefson, Bar-Yosef, & Watkins, 2001). Mudbrick had been the primary building material used on the central Anatolia since the first part of the Neolithic period (Karul, 2017). The tradition of mudbrick structures in the settlement of Aşıklı Höyük, Canhasan I and III, Çatalhoyuk (both east and west part) had been continued from the second half of the 9th B.C. to the first half of 6th B.C (Özbaşaran, 2012; French et al., 1972; French 1998; Hodder 2012; Biehl and Rosenstock, 2007). Those mudbrick dwellings had some common properties such as that they were built again at the same place and side by side while they are differentiated from each other in terms of the dimension, structure plan, raw materials, and usage during the Neolithic period (Karul, 2017).

The differences of the mudbrick dwellings explained not only the source availability but also specific and cultural choices (Love, 2012). Human choices guide the varieties of techniques. The differences between the materials are the indicator of the multiple technical choices during the production process (Stark, 1998; Dobres, 1999). The production of mudbricks involves the selection and combination of raw materials, type of temper, technology, and design (Love, 2012). Those selection and combinations provide us the interpretation of ancient architecture in terms of material culture, material properties, and, materiality. Like the other material assemblages, architecture is a dynamic component of material culture (Love, 2013). Mudbrick architecture gives an idea about the relationship between the people and the materials. The different choice of raw materials and additives in the mudbrick dwellings indicate that some mudbrick manufacture options emerged. Mudbricks are the results of a complex series of socially informed choices, and the analyses of compositional and technological properties of the mudbricks have the potential to identify the range of social practices during manufacture (Love, 2012).

In this research, Ulucak Höyük Neolithic settlements remain of mud materials such as the mudbricks, mud plaster of the interior/exterior walls and the floor covering mortar are investigated in terms of their physical and physicomechanical properties, raw materials characteristics and compositional properties. Those studies are expected to help the better understanding the ancient mudbrick building technology and the varieties of mud materials produced for the different building components. The results are evaluated in terms of raw material sources, preferences of additive and, differences among production technology. This evaluation is also associated with spatial and temporal variations. Throughout the study, the indicators of social behavior and practices of ancient people were discussed with the help of the experimental results.

In this chapter, the research question of this thesis, the aim, and scope of the study and the disposition of the thesis are presented in the following subheadings.

1.1. The Research Question of the Thesis

The knowledge about the Neolithic period in Aegean Turkey has been known for twenty years. The excavations on the Western coast of Turkey, which are Ulucak, Çukuriçi, Dedecik - Heybelitepe, Yeşilova, and Ege Gübre provide information about the characteristics and development of the Neolithization process of the region. Ulucak Höyük is the important site of the region with eight meters of Neolithic deposits represents a thousand years (Çevik & Abay, 2016).

Ulucak Höyük has well-preserved mudbrick remains (Çevik & Abay, 2016; Derin, 2005) that provide comprehensive investigation on the architectural structure of each level, the building material, and the properties of these materials, etc. In this research, the mud materials' compositions and properties of the Ulucak Höyük Neolithic settlement remain were investigated in terms of their usage and, spatial and temporal changes. The collected samples from eight different houses represent the Vth, and IVth levels of the settlement and the maximum time difference between the samples is 700 years.

The main question of this study is how the mudbrick technology had been shaped by the ancient people in Ulucak Höyük settlement during the long years of the Neolithic period. What were the roles of the spatial differences, raw materials preferences, and the usage purpose of materials on the technological changes of mudbricks? Were there any indicators of social and cultural effects on the mudbrick technology? What is the relationship of the architectural material culture of Ulucak Höyük Neolithic settlement between the technological and social knowledge of the ancient people?

The laboratory analyses were performed to answer the questions of the research. Physical/physicomechanical properties, raw material characteristics, and compositional properties of the samples were determined. The results were discussed in terms of the research questions.

1.2. Aim and Scope of the Study

The aim of this research is

- to produce knowledge on the performance, technological and compositional properties of the Ulucak Höyük Neolithic settlement mudbrick materials to determine the dwelling materials technologies that period,
- to determine the effect of raw materials preferences, and the usage purpose of materials on the technological properties of mudbricks,
- to compare the differences of the mudbrick samples technological and compositional properties in terms of temporal and spatial,
- to determine the indicators of the social and cultural effects on the mudbrick technology,

- to assess the relationship of the architectural material culture of Ulucak Höyük Neolithic settlement between the technological and social knowledge of the ancient people.
- to contribute to the establishment of most suitable analytical methods to be performed in the laboratory to produce quantitative data on the source analyses of raw materials and the description of the technologies related to various mud materials serving their purpose such as mudbricks used on the walls, internal and external plasters, floor mortars.

  Finally, the study also targets to discuss the experimental results for their probable contribution to building up conservation strategies and development of conservation methods for those mud materials at the site and in the museum.

1.3. Disposition

This study is presented in 6 chapters; the first one is the introduction part. The research question of the thesis aim and objectives of the study are presented in that chapter. Also, the structure of the thesis is described in the disposition part.

The literature review is the second part of the study. In this section earth building materials, their compositional and technological properties are given. After that, the Ulucak Höyük excavation site is defined in detail in terms of its geological structure, architecture, Neolithic Period levels and building definitions of the site.

Material and method is the third chapter of the study. In this chapter, the experimental procedures of physical, physicomechanical and raw materials compositional analyses, salt analyses and microstructural analyses of the mudbricks, mud plasters, and floor covering mortars are described.

Experimental result is the fourth chapter, where all results are given. The properties of mudbrick, mud plaster, and floor covering mortar samples are explained with relevant figures, graphics, and tables.

In the fifth chapter, the results are discussed by the comparative evaluations. The similarities and differences of the usage of raw material sources are discussed in terms of XRD analyses, calcium carbonate, and organic matter content. The compositional and performance properties of the sample are discussed in terms of particle size distribution, organic matter and calcium carbonate content, pozzolanic activity properties, and cross-section image analyses. The definition of mineralogical composition is evaluated by thin section, XRD, FTIR, and thin section image analyses. The performance properties of the samples were discussed in terms of basic physical - physicomechanical properties.

In the last chapter, the summary of the study, recommendations, and some further studies are presented as a conclusion part.

CHAPTER 2

LITERATURE REVIEW

2.1. Earth Building Materials and Their Technological Properties

Earth is an essential natural building material used all over the world for thousands of years. Earth has been used in buildings as a load-bearing material, filling material inside another bearing structure, mortar, and plaster (Oliver, M 1993). When used earth materials as a bearing material, there are different methods such as:

a. “cob” or “chalk mud” construction (without formwork)

b. Rammed earth (pisé in French) technique (with formwork)

c. Earth blocks (hand-molded clay lumps)

e. Mudbricks (formed in a mold, air-dried, or sun-dried before use) (Davey, N. 1961; Olivier, M. 1993).

Cob is possibly the most primitive method that direct shaping of the walls. The walls are directly molded on-site without using any mold or formwork. In this method, the clay and straw are mixed with sufficient water to give a suitable consistency to the mixture for easy compaction. The mixture is first stacked on top of each other, then shaved. This type of architecture is soft and round due to hand modelling. A stone foundation is used to protect the wall from rising damp and rain splashing. The typical examples of this architecture are found in Devonshire (England), Northern Yemen, and northern Togo. The “cob” technique is called “bauge” in French, “zabour” in Yemeni (Davey, N. 1961; Olivier, M. 1993).

Another earth walling technique is rammed earth (known as “pisé” in French) which performed with formwork or basketwork. In this method, the soil mixture is compacted in between two wooden forms with the help of specialized hand tools. For the rammed earth method, the best earth mixture containing no more than 30% of clay, %70 of sand, with only sufficient water. The mixture should be moist, not wet. 3 to 6 cm gravels are often found in rammed earth, which is straighten the elements such as corners, foundations, windows, and doors (Davey, N. 1961; Olivier, M. 1993).

Earth blocks are made by shaping the material with hand into loaf-shaped pieces after preliminary air drying is laid in mud mortar in horizontal courses. The mud mixture is the same as the “cob” constructions (Davey, N. 1961).

Mudbricks are the most extensively used method of earth walls, and their production is performed by using square or rectangular forms molds. Wet mud mixture is poured into these molds, small blocks which are immediately unmolded and then sun-dried or air-dried. Fine and clayey soil is mixed with 25 to 30% of water and fibers such as straw, grass, cisal, or palm may be added to obtain mud (Davey, N. 1961; Olivier, M. 1993).

2.2. Composition and Durability of Mud Brick Structures

The performance of mud-based materials is mainly depending on its particle size distribution, clay mineral composition and the additives. Certain types of distribution of the mud-based materials have more durable properties.

The particle size classification established by the International Society of Soil Science is as follows;

pebbles: 200 mm – 20 mm,
gravel: 20 mm – 2 mm,
coarse sand: 2 mm – 0.2 mm,
fine sand: 0.2 mm – 0.06 mm,
silts: 0.06 mm – 0.02 mm,
fine silts: 0.02 mm – 0.002 mm
clays: < 0.002 mm (Brown and Clifton, 1978).

The silt-clay portions of a soil act as a binder in the mud-based materials and the soil should contain sufficient quantities of silt-clay mixture to form a matrix. In the matrix, the sand particles are firmly embedded. For the durability of the mud-based construction materials the suitable mixture should have contain approximately 70 – 80 %sand and 10 – 15% silt and clay. The rammed earthwork need 12 – 15% water and the mud bricks formed in molds need 25 – 30% water, these percentages varies due to the clay and silt contents. The presence of high amount gravel or clay could be adversely affect the durability of a mud-based structures. Dimensionally stable adobe, in general, has high sand to silt-clay ratio with a minimum amount of gravel (Brown and Clifton, 1978).

The performance of an adobe soil is mainly depending on its particle size distribution. The amount of the gravel aggregates and clay portion is important for the durability properties. The clay type and portion of it are important for both the expansion on the absorption of moisture and its shrinkage on drying. So, the portion of clay and its mineralogical properties affects the durability (Brown and Clifton, 1978). Clay minerals having non-swelling molecular units such as kaolinite and illite are thought to produce more durable mudbricks than the swelling ones (Brown and Clifton, 1978).

Clay minerals, consist of sheets of silicon dioxide attached to sheets of aluminum hydroxide. These sheets can be combined with other elements like calcium, sodium, iron, potassium, and magnesium into the crystal structure. Various types of clay minerals in the soil can be classified into three main groups as illites, kaolinites, and smectites. Illites group of clay consist of two silicon dioxide sheets to a single aluminum hydroxide one, but the structure is closer. The main interlayer cation is potassium. Illite has not swelling properties in their molecular units. Kaolinites group clays consist of one silicon dioxide sheet and one aluminum hydroxide sheet in their molecular units. Kaolinite molecular unit has not swelling properties. Smectite group clays cover a range of compositions involving a structure of two silicon dioxide sheets to one aluminum hydroxide sheet, magnesium or iron can substitute for the aluminum and sodium, and calcium can be found in interlayer spaces. Smectites clays have swelling properties by taking of water to their interlayer spaces. Most of the soil resources contain various proportions of all groups of clays.

Clay is actually a complex collection of minerals and organic matter. The most important minerals involved in the clay are the quartz sand grains, the iron oxides and hydroxides, and the actual clay minerals.

Organic additives such as egg whites, blood, fig juice, hog’s lard, casein, fats, curdled milk, oils, plant, and animal fibers were used to improve workability, to extend or retard the setting time to increase cohesion and the strength of mud-based materials such as mudbricks, mud-plasters and mud-mortars (Sickels, 1981). Animal fibers such as fur and hair from livestock and synthetic fibers such as cellophane, and glass wool are known to be used in adobe mixtures.

Fiber additives, very often straw, are widely used for stabilization. Straw is regarded as a structural reinforcement agent, similar to gravel. Earth materials are reinforced with fibers to prevent cracking in step with the increase in the stress. In addition, fiber reinforcement provides good compressive strength depends on the quantity of the fiber and the initial compressive strength of the soil (Houben & Guillaud, 1994). Fiber additives is used for all methods of mud material production to accelerate drying. Fibers improve the drainage of moisture towards the outer surface through the channels afforded by the fibers, but they increase absorption in the presence of water. As the fibers light material, they are reducing the bulk density and improving mud-based materials’ insulating properties. Fibers may hinder cracking upon drying by distributing the tension arising from the shrinkage of the clay throughout the bulk of the material and by increasing tensile strength (Houben & Guillaud, 1994).

The good compressive strength for the mud-based materials can also be achieved with fiber reinforcement because the degree of shear strength depends mainly on the tensile strength of the fibers. Some research suggests that the preliminary rotting of the straw in the soil for a period of several weeks produces lactic acid, which has a secondary effect on the efficiency of the stabilizing action (Houben and Guilloud, 1994).

Mud-based construction materials exhibit excellent stability in dry climates. The exposure of a mud-based structure to moisture, such as rain, rising groundwater or high humidity, causes deterioration called weathering. In the ancient Mesopotamia, mud-brick structures were usually protected from environmental conditions by covering them with burnt brick set in bitumen (Davey, 1961).

Mud-based construction material should be protected against moisture, when moisture entered into the mud materials it should be removed from the materials as quickly as possible before causing further problems. This is a significant property, called as ‘breathing property’. Other major weathering processes include erosion or leaching of the silt clay matrix, soluble salt-action, and dimensional instability associated with cyclic wetting and drying, and freeze-thaw damage (Brown and Clifton, 1979).

The weathering rate of the mud-based materials is affected by its mineralogical composition such as particle size distribution, porosity distribution, and soluble salt content as well as its moisture content (Brown and Clifton, 1979).

The durability of mud bricks -similar to stone, mortar, and brick- is expressed with their physical and mechanical properties such as bulk density, porosity, water vapor permeability, modulus of elasticity, wet to dry strength ratio, etc. (Tunçoku, 2001).

There are many kinds of clayey material suitable for making bricks. Every mudbrick type requires different materials and manufacturing techniques to produce brick types that vary in terms of strength, durability, weight, texture, and color. (Plumridge and Meulenkamp, 1993)

2.3. Burnt Mud Materials

Fire changes the physical, mechanical and raw material properties of mud-based materials depending on the temperature and conditions of firing. The color of the mud-based materials is changed by the fire, as the higher the temperature, the more intense the color therefore the color change may be a rough indication of the firing temperature. Due to variations in the oxidizing/reducing atmosphere during firing, the color can vary from red to black, depending on the amount of calcium, the color of brick can develop from red to yellow, as the calcium silicates incorporate Fe_2O_3 in their lattices (Heimann 1978; Franke and Schumann, 1998).

The fire makes a change in the porosity characteristics of the mud-based materials. Besides the fire, production techniques as molding or extrusion, the grain size distribution of the components, water content, additional fluxes are the other factors that influence porosity characteristics (Franke and Shuman, 1998). Grain size distribution of the sand also plays a significant role to determine the pore size distribution of the fired mudbrick.

Different mineral phases occur during the fire depending on the composition of clay minerals in the mud mixture. The most important clay mineral thermal decomposition is that of kaolinite to mullite and illite to gehlenite. Kaolinite structure is totally disturbed at around 500ºC and at about 800ºC, it changes into metakaolin. Illite peaks start to disappear at 850ºC, and gehlenite is formed at 900ºC (Franke and Shuman, 1998).

2.4. The Geological and Geomorphological Properties of Ulucak Region

Kemalpaşa region has three different places in terms of land usage. These are plain base, boom, and high, defective mountainous-hilly areas. Here, the most critical factor determining the land usage potential is geographical formations. The geographical formations have developed under the control of the geological structure, processed by the climatic characteristics over time, covered with appropriate soil and vegetation cover (Kayan, 1999).

Figure 2.1. Large geographical formation units of the wide perimeter of the Kemalpaşa plain (Kayan, 1999).

The rift zone where the Kemalpaşa plain is located shaped as a stipe of the Gediz basin between Manisa (Spil) mountain in the north and Kemalpaşa (Nif) mountain in the south. The Kemalpaşa and Manisa mountains are located in a different zone in terms of geological structure. The rocks forming both mountain masses were formed by the change of muds accumulated in a sea in this area in the 2nd Geological Age (Mesozoic) with various effects. The structure of these mountains generally consists of sandy-clayey rocks at the bottom and limestone in large blocks at the top (Kayan, 1999).

The properties, which are connected to the geological structure, are essential in terms of water resources around the Kemalpaşa plain. Since limestone are very cracked rocks, rainwater leaks from these cracks rather than flowing over them. The clayey flysch formation at the bottom prevents the water from seeping deeper, and the water comes out of the places where two different rock units are opened to the earth and form springs. On the other hand, on the slopes of the Manisa Mountain, the limestone covers large areas and this area is drier since there are no suitable features for the leakage of rainwater. Undoubtedly, this factor has a significant role in the fact that Kemalpaşa, the settlement center of the plain, is located in the south (Kayan, 1999).

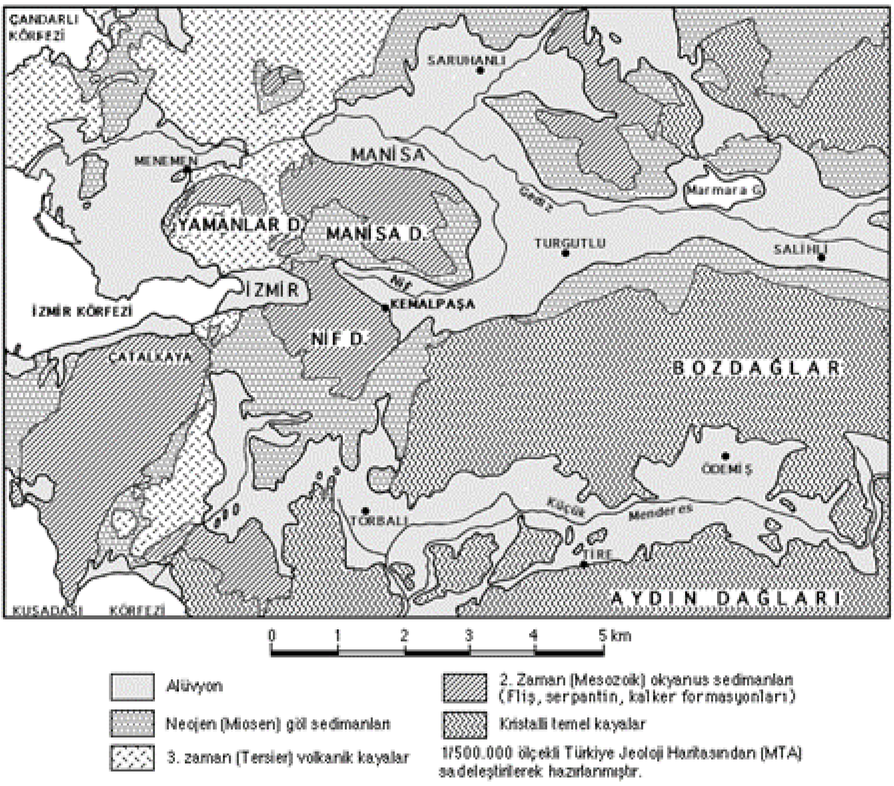


Figure 2.2. Large structural-geological units of the wide perimeter of the Kemalpaşa plain (Kayan, 1999).

Rock fragments erode from high places, transport to graben area and deposit in there. This formation continues by the physical environment characteristics, especially the climate. The characteristics of sediments that fill the İzmir-Kemalpaşa graben indicate that lakes existed under humid climatic conditions and volcanic ashes are occasionally involved in the lake sediments due to occasional volcanic eruptions (Figures 2.1. and 2.2.) (Kayan, 1999).

More recently in geological terms (such as 5-3 million years), the lakes in the graben dried up and were covered with flood deposits. These reddish-colored, stony-sandy fillings are common in the north of Kemalpaşa plain, especially around Ulucak-Damlacık-Kuyucak and Sancaklı circles in the north of Kemalpaşa plain, especially in the west. These areas are higher than today's plain floor (Kayan, 1999).

The Kemalpaşa plain is formed in a graben width of 3-5 km on average between the mountains rising to 1500m. The stony deposits brought by the ephemeral coming from the mountains formed a permeable-porous filling where groundwater can be stored (Figure 2.3.). Since the slopes of Manisa Mountain in the north are limestone, the rainwater falling on them leaks to the ground to a great extent and feeds the groundwater. Although the Kemalpaşa plain is not very wide, it can be said that it is a rich area in terms of the presence of water.

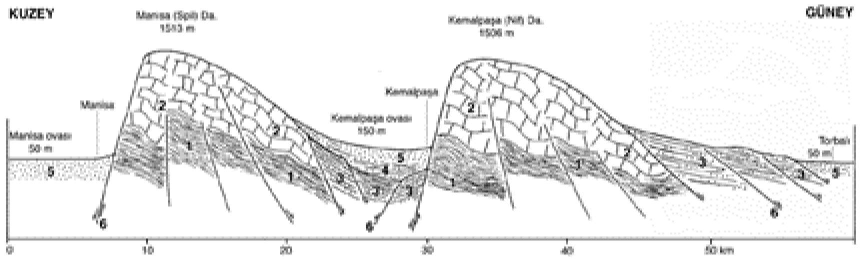


Figure 2.3. Structural section of Kemalpaşa plain between Manisa and Torbalı (Schematic model). 1. Mesozoic ocean sediments, generally composed of sandy-clayey flysch formation. 2. Mesozoic ocean sediments, usually composed of carbonated rock. 3. Generally carbonate-clayey-sandy Miocene lake sediments. 4. Generally sandy-gravelly Pliocene flood sediments 5. Alluvium 6. Faults (Kayan, 1999)

2.5. The Neolithic Architecture of Ulucak Höyük

The Neolithic architecture of the Ulucak Höyük settlement is mentioned in terms of Vth and IVth layers. Layer V is expressed as an "Early Neolithic Period" and has well-preserved buildings in the latest two sub-phases of Ulucak Va-b (6200-6000 BC). The wall of these buildings was constructed with both wattle and daub technique and pise technique (rammed earth) without stone foundations. "Pisé" walls show a thickness of 15-20 cm. The structures are characterized by one-roomed rectangular dwelling and their walls and their measures about 20 m^2. The interior corners of the rectangular buildings were rounded (Çevik & Abay, 2016; Derin 2005).

Layer IV is expressed as “Late Neolithic Period” and the architecture of this period differs from the earlier levels by their construction technique. The dwellings of layer IV were built of sun-dried mudbrick walls on stone foundations and had flat roofs (Çilingiroğlu et al. 2004; Çilingiroğlu et al. 2012). Level IV has ten sub-phases. The dwellings of the IVb sub-phases were arranged along the narrow streets, have generally been constructed in a rectangular plan and the size of them ranging between 30 and 40 square meters. Mudbricks above stone foundations have a uniform, and well-planned structure and two sizes of mudbricks can be distinguished: a bigger one (55x35x8 cm - 50/48x34x8 cm) and a smaller one (50x18x8 cm) (Çevik & Abay, 2016; Derin 2005).

2.5.1. Level V: Early Neolithic Period

Vd_Building 54: The preserved dimensions of the building are 4.50x5.20m. Evidence for the walls of the building, which was made of pise technique, was found in the northeast corner. The outer part of the wall could not be detected in this area and only the plaster was preserved in the inner part of the wall. The floor of the structure is made of rammed earth. There are two holes in the structure of the wooden poles supporting the roof. The space was consciously closed after the end of its life (Figure 2.4.) (Çevik, Ö. et al 2017).

Figure 2.4. Building 54: Vd sub-layer of Ulucak Höyük Neolithic settlement (Çevik, Ö. et al 2017).

Vb_Building 51: The determined section of building measures 2.80x3.70m. The wall of the building was made with pise technique and the thickness of that is ranged between 0.15 to 0.20 m. The entrance of the space is 0.70 m. width. The floor of the structure is made of rammed earth. 8 wooden poles holes were found on the floor of the room which did not show a certain order (Figure 2.5.) (Çevik, Ö. et al 2015).

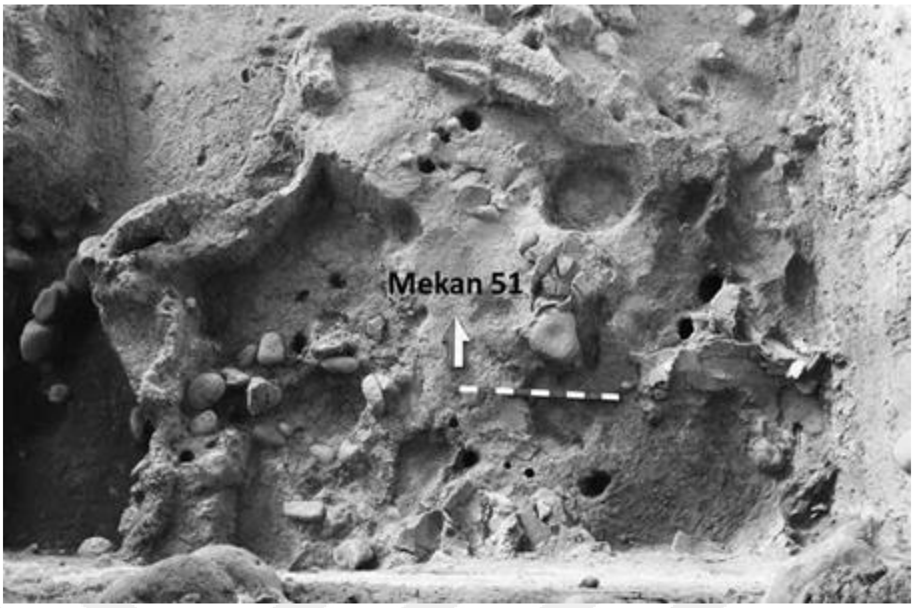


Figure 2.5. Building 51: Vb sub-layer of Ulucak Höyük Neolithic settlement (Çevik, Ö. et al 2015).

2.5.2. Level IV: Late Neolithic Period

IVb Building 13: This is the well-preserved building which is a slightly trapezoidal shape and has two rooms measuring 7.00x5.50 m. An inner wall divides a smaller room in the southern part. A door opening with a width of 1.53 m was found on the western wall. The outer walls were built of mud brick on a stone foundation measuring 55 cm in width and the thickness of the interior wall measures 30 cm. Paint decoration was found on the wall which was not well protected due to fire, was painted with red brown paint (Figure 2.6.) (Derin, 2005; Derin et al, 2003).

Figure 2.6. Building 13: IVb sub-layer of Ulucak Höyük Neolithic settlement.

IVb_Building 48: It has been determined that the building has a usage area of 50 m2. Apart from the western wall of the building, all walls are preserved approximately 2.00 m. The walls have an interior/exterior plaster and were built on 3 rows of stone foundations as mudbrick bricks and their thickness was 0.50 m. The entrance to the building is provided by a 1.60 m door opening in the centre of the western wall. The floor of the structure is made of rammed earth. It was determined that the floor of the Building 12, which appears to have been exposed to heavily fire, was plastered at least 3 times (Figure 2.7.) (Çevik, Ö. et al 2014).

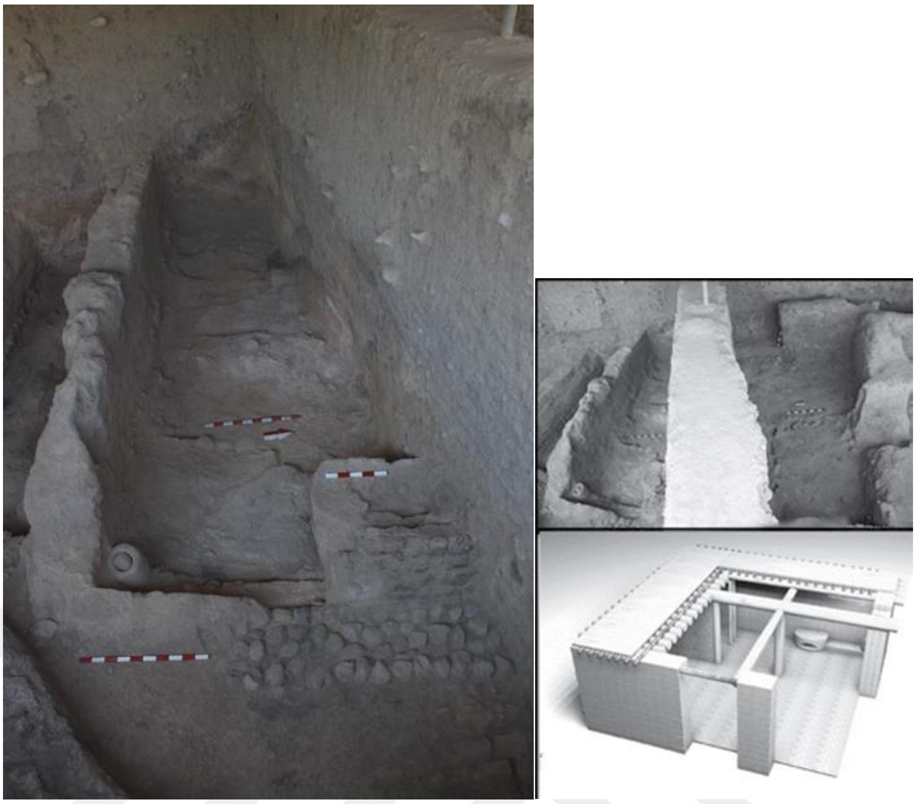


Figure 2.7. a.Building 48: IVb sub-layer b.The reconstruction of the building (Çevik, Ö. et al 2014).

IVb_Building 52: The size of the building is 3.70x4.50 m and its walls were built on 3 rows of stone foundations with sun-dried mudbricks and their thickness was 0.60 m. Based on the preserved plaster traces on the walls, it is noteworthy that the interior and exterior surfaces were plastered with mud and the northern wall was plastered with lime. The entrance to the building was provided by a door opening of 1.00 m on the eastern wall. The floor of the structure is made of rammed earth. (Figure 2.8.) (Çevik, Ö. et al 2015).

Figure 2.8. Building 52: IVb sub-layer_ late Neolithic period of Ulucak Höyük settlement (Çevik, Ö. et al 2015).

Figure 2.9. Building 55: IVc sub-layer_ late Neolithic period of Ulucak Höyük settlement (Çevik, Ö. et al 2017).

IVc_Building 55: The size of the building is 3.00x5.50 m and the walls have a thickness of 0.40 m. The entrance to the building is through a door opening of 0.60 m on the eastern wall. The floor of the structure is made of rammed earth (Figure 2.9.) (Çevik, Ö. et al 2017).

IVc_Building 62: The size of the building is determined 3.20x5.00 m. The walls of the building were preserved to a height of approximately 0.80 m and their width ranged from 0.30 to 0.50 m. No evidence of entrance was found on the preserved walls of the building. The floor of the structure is made of rammed earth (Figure 2.10.) (Çevik, Ö. et al 2017).

The buildings which were built in the combined order belonging to the IVc phase of the Late Neolithic Period are the workshops used in ceramic production. The presence of grinding devices, grinding stones, lumps made from clay and hematite indicates the production of ceramics (Figure 2.11.) (Çevik, Ö. et al 2017).

Figure 2.10. Building 62: IVc sub-layer_ late Neolithic period of Ulucak Höyük settlement (Çevik, Ö. et al 2017).

Figure 2.11. Building 55 and Building 62: IVc sub-layer_ late Neolithic period of Ulucak Höyük settlement (Çevik, Ö. et al 2017).

CHAPTER 3

MATERIAL AND METHOD

In this chapter, description of the mudbrick, mud plaster and mud floor mortar samples, their nomenclatures and the laboratory analyses are done. Laboratory analyses included the determination of the basic physical and physicomechanical characteristics, raw material properties and mineralogical composition of the samples. Basic physical and physicomechanical properties were expressed by their color, bulk density, effective porosity, water vapor permeability, ultrasonic pulse velocity and modulus of elasticity values. Binder-aggregate ratios of mudbricks and mud plasters, their particle size distributions, pozzolanic activity of finest aggregates, losses on ignition at 600°C and 900°C and soluble salt content were determined as their raw material properties. Mineralogical composition of raw materials and their relative proportions were identified by combined interpretation of several types of analyses such as the cross section and thin section analyses using an optical microscope, X-Ray diffraction and FTIR analyses.

3.1. Sampling and Description of Samples

In the study, mudbrick, mud plaster and floor mortar samples were collected from two different Neolithic Period levels of the Ulucak Höyük settlements' remains. These are Level V (6500-6000 BC) which was named as“Early Neolithic Period” and Level IV (6000-5700 BC) named “Late Neolithic Period”. These two levels had several sub-phases, and the collected samples represented the “IVb, IVc, Vb, and Vd” sub-phases of the settlement (Çevik & Abay, 2016). Twenty samples were collected from eight different buildings that were situated in the four different sub-phases. Two samples are belonging to the Level IV, but their phases have not determined yet by the excavation team (Table 3.1).

Table 3.1. The classification of the samples indicating the time periods, their layers, sub-phases, building numbers and number of samples.

Layer and Sub-phase	Time Period	Number of Samples	Number of Buildings
IV	6000-5700 BC	2	1
IVb	5840-5710 BC	10	3
IVc	6005-5840 BC	6	2
Vb	6390-6080 BC	1	1
Vd	6380-6210 BC	1	1

Four different mudbrick structure materials, namely mudbrick, interior / exterior wall mud-plasters and floor covering mortar samples were collected from 8 different buildings. Since large-scale fires took place in the settlement, some of the samples were damaged to varying degrees by fire while some of them remained intact. The architecture of level IV is shown in Figure 3.1.

Figure 3.12 Architectural remains of the Ulucak Höyük; in Level IV (Derin, 2005).

3.1.1. Nomenclature

Each sample was given a code describing its “level, sub-phases of the settlement”, “building name”, “material type”, “serial number of the sample” and its “firing state”. Nomenclature explanation of the samples are described below, respectively.

1. Level of the settlements: “V & IV”

2. Sub-phases of the level: “a, b, c, d”

3. Building name: “B65, B13, B48, B52, B55, B62, B51, B54”

4. Material type:

MB: Mudbrick

IWP: Interior Wall Plaster

EWP: Exterior Wall Plaster

FCM: Floor Covering Mortar

5. Serial number given during sampling: “1, 2, 3, 13, 14, 16, 17, 18, 19, 21A, 24, 26, 27, 28, 29, 30, 34, 38, 39, 43”

6. Firing State:

CB: Completely Burnt

PB: Partially Burnt

NB: Not Burnt

As an example, two samples coded by the nomenclature described above are shown in Table 3.2.

Table 3.2. Description of the nomenclature for the samples IVc.B55.FCM.2.NB and Vb.B51.IWP.43.PB

IV	**c**	**B55**	**FCM**	**2**	**NB**
Level of the settlements	Sub-level	Building name (**B**uilding 55)	Material Type (**F**loor **C**overing **M**ortar)	Serial number of the sample	Firing State (**N**ot **Burnt**)
V	**b**	**B51**	**IWP**	**43**	**PB**
Level of the settlements	Sub-level	Building name (**B**uilding 51)	Material Type (**I**nterior **W**all **P**laster)	Serial number of the sample	Firing State (**P**artially **Burnt**)

Definition of the samples are shown in Table 3.3.

Table 3.3. Definition of the samples examined in the study.

Sample Code	Level& Sub-phase	Building Name	Description	State of Burning	Presence of fibre	Presence of aggregates
IV.B65.MB.38.PB	IV	B65	**M**ud **B**rick	**P**artially **Burnt**	-	+
IV.B65.FCM.34.CB	IV	B65	**F**loor **C**overing **M**ortar	**C**ompletely **Burnt**	+	+
IVb.B13.MB.19.CB	IVb	B13	**M**ud **B**rick	**C**ompletely **Burnt**	+	+
IVb.B13.FCM.21A.NB	IVb	B13	**F**loor **C**overing **M**ortar	**N**ot **Burnt**	-	+
IVb.B13.IWP.18.CB	IVb	B13	**I**nterior **W**all **P**laster	**C**ompletely **Burnt**	+	+
IVb.B48.MB.17.CB	IVb	B48	**M**ud **B**rick	**C**ompletely **Burnt**	+	+
IVb.B48.FCM.24.CB	IVb	B48	**F**loor **C**overing **M**ortar	**C**ompletely **Burnt**	+	+
IVb.B48.EWP.26.CB	IVb	B48	**E**xterior **W**all **P**laster	**C**ompletely **Burnt**	+	+
IVb.B52.MB.27.PB	IVb	B52	**M**ud **B**rick	**P**artially **Burnt**	+	+
IVb.B52.FCM.29.CB	IVb	B52	**F**loor **C**overing **M**ortar	**C**ompletely **Burnt**	+	+
IVb.B52.IWP.30.CB	IVb	B52	**I**nterior **W**all **P**laster	**C**ompletely **Burnt**	+	+
IVb.B52.EWP.28.CB	IVb	B52	**E**xterior **W**all **P**laster	**C**ompletely **Burnt**	+	+
IVc.B55.FCM.2.NB	IVc	B55	**F**loor **C**overing **M**ortar	**N**ot **Burnt**	-	+
IVc.B55.IWP.3.PB	IVc	B55	**I**nterior **W**all **P**laster	**P**artially **Burnt**		+
IVc.B55.EWP.1.CB	IVc	B55	**E**xterior **W**all **P**laster	**C**ompletely **Burnt**	+	
IVc.B62.MB.16.CB	IVc	B62	**M**ud **B**rick	**C**ompletely **Burnt**	+	+
IVc.B62.FCM.14.PB	IVc	B62	**F**loor **C**overing **M**ortar	**P**artially **Burnt**	+	+
IVc.B62.IWP.13.CB	IVc	B62	**I**nterior **W**all **P**laster	**C**ompletely **Burnt**	+	+
Vb.B51.MB.43.CB	Vb	B51	**M**ud **B**rick	**C**ompletely **Burnt**	+	+
Vd.B54.IWP.39.NB	Vd	B54	**I**nterior **W**all **P**laster	**N**ot **Burnt**	+	+

3.2. Determination of Basic Physical Properties

Basic physical properties of the mudbrick, mud-plaster, floor mortar and compacted earth lining samples were determined by the laboratory analyses in terms of their bulk density (ρ), porosity (φ) and water absorption capacity (θ). Color analyses were determined by the Munsell Soil Chart as a physical property.

3.2.1. Bulk Density, Porosity, Water Absorption Capacity

Determinations of bulk density, porosity and water absorption capacity properties were done for two parallel samples of each sample. The samples were dried in the drying oven at 60°C to constant weight and the dry weight of the samples (M_{DRY}) were recorded. Samples were identified under two groups and analysed by two different experimental methods depending on their state of burning. Completely burnt and some of the partially burnt samples were placed in a beaker and left under distilled water for 24 hours. Afterwards, samples were left under vacuum by using a HEREUS vacuum chamber at 0,211atm (160 torr) for about 30 minutes until the water completely penetrates to the fine pores. The weights of the water-saturated samples were recorded (M_{SAT}). Samples submerged into distilled water and their Archimedes weights were recorded (M_{ARCH}).

Bulk density (ρ) is the ratio of the mass to the volume of the sample, it is calculated using Equation 3.1 (Teutonico, 1988; RILEM, 1980).

$$\rho = \frac{MDRY}{MSAT - MARCH}, \text{g/cm}^3 \qquad \textbf{(3.1)}$$

Porosity (φ) is the fraction of the total volume of a solid that is occupied by pores, expressed as percent volume of the solid mass. It is calculated using Equation (3.2) (Teutonico, 1988).

$$\varphi = \frac{MSAT-MDRY}{MSAT-MARCH} \text{ x } 100, \% \qquad \textbf{(3.2)}$$

Water absorption coefficient (θ) is the maximum quantity of water absorbed by a material, it is calculated by Equation (3.3) (Teutonico, 1988; RILEM, 1980).

$$\theta = \frac{MSAT-MDRY}{MDRY} \text{ x } 100, \% \qquad \textbf{(3.3)}$$

Some of the partially burnt and not burnt samples dispersed in water, therefore their bulk density values determined by a different way. For the bulk density determination of these samples, the parallel samples dried in the drying oven at 60°C to constant weight and the dry weight of the samples (M_{DRY}) were recorded. Afterwards, the samples were covered with three layers of stretch film to prevent contact with water. These samples were dipped in a water and the difference of the water displacement volume (V_{WATER}) was recorded. The density of these samples was calculated by Equation (3.4) (ASTM:D7263-09, 2009).

$$\rho = \frac{\text{MDRY}}{\text{VWATER}} \text{ , g/cm}^3 \qquad \textbf{(3.4)}$$

$$\text{SD} = [\psi\text{L x A x (P1 - P2) / I}] - \text{SL, m} \qquad \textbf{(3.5)}$$

3.2.2. Color Measurements

Color is the most visually distinctive characteristic of mudbricks related with the origins of sediments used in mud materials and their quantity. Besides that, color might be a rough indication of the firing temperature of the mudbrick. (Franke and Schoppe, 1988). The color measurements of the samples were determined by visual color assessment using Munsell Soil Color Charts (Munsell, 1971). The Munsell Soil Color Chart has hue (a specific color), value (lightness and darkness), and chroma (color intensity) components which generate the color notation. The color notation of “5YR 8/4” refer to the 3 attributes of color; 5YR is the Hue (or color), 8 is the Value (or lightness/darkness) and 4 is the Chroma (weak/strong) (Munsell, 1971).

3.3. Determination of Basic Physicomechanical Properties

Ultrasonic pulse velocity (UPV) and modulus of elasticity (MoE) properties were examined for the expression of the physicomechanical properties of mud materials.

3.3.1. Ultrasonic Pulse Velocity

Ultrasonic pulse velocity (UPV) covers the determination of the propagation velocity of longitudinal stress wave pulses through material (ASTM D 2845-08:2017). UPV measurements were conducted on all samples in direct mode by using the pulse generating test equipment, PUNDIT plus with its probes, transmitter and receiver of 54 kHz. The transmitter and receiver were placed in opposing sides of the samples for the direct transmission measurements. The thickness of the samples where the probes measure the pulses and the measured microsecond data by the equipment were recorded.

The ultrasonic velocity of the waves is calculated by Equation 3.8 (ASTM D 2845-08:2017, RILEM, 1980).

$$V : l/t$$ **(3.8)**

where;

V : velocity (m/s)

l : the distance traversed by the wave (mm)

t : travel time (s)

3.3.2. Modulus of Elasticity

Using the direct ultrasonic pulse velocity and bulk density measurements, the modulus of elasticity (Emod) values were determined (ASTM D 2845-08:2017; RILEM 1980). The modulus of elasticity (Emod) is defined as the ratio of stress to strain and shows the deformation ability of a material under the effect of external forces (Timoshenko, 1970).

The modulus of elasticity is obtained by the Equation 3.9 (ASTM D 2845-08:2017; RILEM 1980):

$Emod = D \text{ x } V\ 2 \text{ x } (1 + \nu dyn) \text{ x } (1 - 2\nu dyn) / (1 - \nu dyn)$ (3.9)

Where;

Emod: modulus of elasticity (N/m2)

D: bulk density of the sample (kg/m3)

V: wave velocity (m/s)

ν dyn: Poisson's ratio

Poisson's ratio differs from 0.1 to 0.5. νdyn = 0.18 seemed to be a reasonable value for this case (Topal 1995; Tunçoku 2001).

3.4. Determination of Raw Materials Composition

Raw material compositions were examined in terms of loss on ignition, particle size distribution and pozzolanic activity analyses.

3.4.1. Loss on Ignition

This method has been used for determining the amount of organic materials and $CaCO_3$ in the mud materials. The organic materials are ignited approximately between the temperatures of 200°C - 550°C. CO_2 gas output from $CaCO_3$ begins at about 800°C and is completed when it is reached 850°C. The basic mechanism of this method is based on the weight loss of the samples before and after heating to 600°C and 900°C and the percentage of the weight losses are determined.

Loss on ignition experiments were carried out for twenty samples which were mudbricks and mud plasters. Before the analyses, samples were powdered in an agate mortar. Because of the variations of the samples, this technique was applied on the duplicates of each sample. The empty ceramic crucibles were desiccated for an hour to remove moisture and were weighted. Approximately 5 gr samples were placed in and the samples were dried at 105°C in a furnace for 16 hours. The crucibles were placed to a desiccator for cooling to room temperature, after cooling the crucibles (with samples) were weighed. This was the dry weight of the samples and was the basis used for the calculations. The crucibles were placed in furnace again and heated to 600°C for 6 hours, after cooling to room temperature in a desiccator, they were weighted. The difference between that weight and dry weight is accepted to be the amount of the organic materials in the samples. The crucibles were placed to the furnace one more time and were heated to 900°C for 2 hours, samples were cooled and weighted. The weight loss between 600°C and 900°C represented the amount of CO_2 evolved from carbonate minerals in the samples. The $CaCO_3$ amount was calculated by dividing the CO_2 content by 0.44, which was the fraction of CO_2 in $CaCO_3$. For this calculation it was assumed that the amount of carbonate was consisted of only from the calcium carbonate. (Stein, 1984; Love, 2012; Davies, 74; Gale and Hoare, 1991).

3.4.2. Particle Size Distribution

Sediments used in mudbrick production vary in quality according to the amount of sand, silt and clay parts. Particle size analysis is an essential test for mudbrick analyses and becomes the basis for determining the mudbrick composition. Particles types and their proportions provide the mudbricks a particular character and behavior (Love, 2017; Teutenico 1988).

Particle size distribution test consist of two procedures; first one is sieving procedures for “coarse” particles (gravel and sand) and second one is sedimentation procedures for “fine” particles (silt and clay) (Teutenico, 1988). To determine of the sand / silt-clay proportions not burnt samples and partially burnt samples were prepared. Not burnt samples were selected as two parallel samples but partially burnt samples were prepared as one sample because there were not enough materials. Selected samples were placed in a 600 ml beaker and dried overnight in an oven at 110ºC. After drying to constant weight samples were cooled and weighed. Then samples covered with 500 ml distilled water, stirred 15 minutes for dispersing, and waited 1,5 minutes. Sand part of the samples was settled down the beaker and silt/clay suspension was transferred into another beaker. This procedure was repeated about 4-5 times until the sand part was completely washed. Afterwards, the sand part was placed in an oven and dried overnight at 110ºC, then cooled to room temperature and weighed. Sand part was run through the sieves varying from larger size (4 mm) down to smaller sizes (0,063mm). In this procedure, it is important to be careful not to lose any material. Sieves were shaken by hand for about 10 minutes and each sieve was weighed with the sample (Teutenico, 1988).

Silt-clay part is considered as a “binder” part of the mudbrick. With this analysis, the particle size distribution and the binder/aggregate ratio of the mudbricks were determined.

3.4.3. Pozzolanic Activity

Pozzolanic activity properties of the samples were determined as described in TS EN 196-5, 2002. For the test, burnt and partially burnt samples were powdered and separated by standard sieves under 125 µm. 0,05 g of sample was placed in a container and 30 ml of saturated $Ca(OH)_2$ aqueous solution was added, and the container was covered tightly. A container having only saturated $Ca(OH)_2$ solution without samples was used as a blank sample. These solutions were left for 14 days to allow for the pozzolanic active sample powder to react with Ca^{2+} ion (TS EN 196-5, 2002). After 14 days, the remaining Ca^{2+} ion concentration in the solution was determined by titration with 0.01M EDTA solution. 10 ml of solution from each container was taken and put into a beaker, 100 ml distilled water, 1 ml of %10 NaOH solution and 1 drop of Calgon indicator were added. %10 NaOH solution kept the pH at about 12 (Black, 1965). The color of the solution was pink at the beginning of the titration, after the titration when EDTA consumed all the Ca2+ ions the color turned into blue and the titration ended. EDTA solution was standardized with pure saturated $Ca(OH)_2$ solution prepared as blank solution. Since the pozzolanic active aggregates reacted with Ca^{2+} ions, the concentration difference of Ca^{2+} ions between the blank solution and the sample solution was used to calculate the pozzolanic activity of the sample powder.

3.5. Quantitative Analyses of Soluble Salts

The amount of salt in the mudbrick and mud-plaster samples were determined as percent by weight. For the experiment, samples were powdered, 1 g sample was taken and mixed with 50 ml distilled water. The mixture was left for settlement of suspended particles for 24 hours and then filtered. The amount of soluble salt in the samples was determined by the conductivity measurements of the solution. The measurements were done using a conductometer of Metrohm AG Herisau, Kondoktometer E382. The percentage of the salt in the sample was calculated with the Equation 3.10 and Equation 3.11 (Black, 1965):

$$EC = \left[\frac{0.001411 * Rstd}{Rext}\right] \text{ , (mmho.cm}^{-1}\text{)} \qquad \textbf{(3.10)}$$

where;

EC: Electrical conductivity, (mmho.cm^{-1})

R_{std}: The cell resistance with standard solution (0.01 N KCl)

R_{ext}: The cell resistance with extract solution

$$\text{\% Salt in sample} = \left[A * \frac{V_{ext}}{1000}\right] * \left[\frac{100}{Ws}\right] \text{, \%} \qquad \textbf{(3.11)}$$

where;

34

A: Salt concentration (mg/l) = 640 x EC (mmho cm^{-1})

V_{ext}: Volume of the extract solution (ml)

Ws: Weight of sample (mg)

3.6. Microstructural Analyses

The microstructural characteristics of the samples were examined by thin section analyses of the samples with polarized microscope, X-Ray Diffraction analyses and FTIR analyses of the powdered samples and image analyses of cross-sections.

3.6.1. Thin Section Analysis

The thin sections of selected samples were prepared in MTA Petrography Laboratory. Samples were placed into plastic molding boxes which had 1.5cm x 3cm x 1cm dimensions. Epoxy resin was prepared by mixing the resin and its hardener and poured on the samples in the molding boxes. After the samples were hardened, they were removed from boxes and cut into slices as thin as possible. Those thin slices were fixed on microscope slides and then they were reduced to 30μ thickness for the analyses. Thin sections analyses were carried out by using a polarizing microscope of Leica DM EP equipped with photographic attachment. Mineralogical and morphological properties of samples matrix, shape, size, and distribution of aggregates in the matrix were examined.

3.6.2. X-Ray Diffraction Analyses (XRD)

Mineralogical content and clay type of the powdered samples were examined in detail with the XRD analyses. All samples as a whole as well as the clay and silt parts of the slightly burnt and unburnt samples were examined by XRD analyses.

XRD analyses of the overall sample including binder (clay and silt portion) and the aggregates (sand portion) were carried out for all (twenty) samples. Each sample was first powdered in an agate mortar and XRD traces of the whole unoriented sample was taken.

The clay and silt size of the 7 samples were extensively examined by XRD for their clay minerals identification. During the particle size analyses, after the separation of clay and silt portion, a few drops of the suspension was put on a microscopic glass slide and dried in the desiccator. XRD trace of that oriented sample was taken. The same sample on the microscopic glass slide was exposed to the ethylene glycol vapor for 24 hours and its XRD trace was taken. After that, the sample was heated at 500 ºC for one hour in an oven. XRD trace of that sample was also taken. Apart from this, the clay and silt fraction of the samples were treated with 2% aqueous solution of acetic acid (CH_3CO_2H) to dissolve all and samples free of $CaCO_3$ were examined by XRD. Mineral phases were identified in detail by those XRD traces.

The instrument used was a Bruker X-Ray Diffraction D8-Discover. Analyses were done by using CuKα radiation, adjusted to 40kV and 40mA. The XRD traces were recorded for the 2θ values from about 5º to 70º.

3.6.3. Fourier Transform Infrared Spectroscopic Analysis (FTIR)

Powdered samples were also analyzed by FTIR to obtain supportive results for other methods of microstructural analyses. The analyses were carried out with a Bruker

FTIR spectrometer for the characteristic absorption bands between 400-4000cm^{-1}. Very small amount (a few milligrams) of homogeneous powdered sample was placed on the crystal part of the instrument and pressed be observed in FTIR instrument.

3.6.4. Image Analyses of Cross Sections

The general texture of the samples and the presence and size of pores, fibers and gravels were analyzed with the cross-section images of the samples. Cross sections of samples were prepared by cutting the samples with a fine Buehler saw. Images were taken at 10x magnifications with stereo binocular microscope (Leica Stereo Optic Microscope).

CHAPTER 4

EXPERIMENTAL RESULTS

The results of the various analyses carried on physical, physicomechanical, raw material composition and microstructural properties of mudbrick samples are given under the following sections of this chapter.

4.1. Basic Physical Properties

The results of laboratory analyses on bulk density (ρ), porosity (φ), water absorption capacity (θ) and water vapor diffusion resistance index (μ) and color identification are given as a physical property in this section.

4.1.1. Bulk Density, Total Porosity, Water Absorption Capacity Properties

In this section, the data of bulk density, total porosity and water absorption capacity characteristics of mudbrick, interior/exterior mud plaster, and floor covering mortar samples are given (Table 4.1)

- For mudbrick samples their bulk density, effective porosity and water absorption capacity were determined to be in the range of 0,75±0,09 and 1,75±0,02 g/cm^3, 33,1±0,19 and 56,7±0,54 by volume, 18,9±0,29 and 51,4±1,60 by weight, respectively. The average values of these characteristics (ρ, φ, θ) were found to be 1.22±0.35 g/cm^3, 45.7±10.0 by volume, 35.3±13.6 by weight, respectively.

Table 4.1. Physical properties of mudbrick, mud plaster, floor covering mortar samples: bulk density (ρ), porosity (φ), water absorption capacity (θ)

Sample Code	**ρ,**	**φ,**	**θ**
	g/cm^3	**% by volume**	**% by weight**
IV.B65.FCM.34.CB	1,17±0,02	51,8±2,73	44,2±3,02
IV.B65.MB.38.PB	0,75±0,09		
IVb.B13.FCM.21A.NB	1,28±0,22		
IVb.B13.IWP.18.CB	1,22±0,01	51,7±0,22	42,4±0,57
IVb.B13.MB.19.CB	1,44±0,31	43,5±12,39	31,9±15,49
IVb.B48.EWP.26.CB	1,32±0,04	48,4±1,89	36,6±2,62
IVb.B48.FCM.24.CB	1,39±0,00	46,2±0,12	33,3±0,02
IVb.B48.MB.17.CB	1,75±0,02	33,1±0,19	18,9±0,29
IVb.B52.EWP.28.CB	1,41±0,01	45,9±0,45	32,5±0,60
IVb.B52.FCM.29.CB	1,3±0,03	50,2±3,16	38,8±3,34
IVb.B52.IWP.30.CB	1,31±0,05	49±0,97	37,4±2,15
IVb.B52.MB.27.PB	0,99±0,05		
IVc.B55.EWP.1.CB	1,28±0,02	50,00±0,88	39,1±1,44
IVc.B55.FCM.2.NB	1,41±0,07		
IVc.B55.IWP.3.PB	1,48±0,11	42,8±3,48	29,2±4,49
IVc.B62.FCM.14.PB	0,96±0,13		
IVc.B62.IWP.13.CB	1,24±0,00	51,7±0,61	41,8±0,62
IVc.B62.MB.16.CB	1,10±0,02	56,7±0,54	51,4±1,60
Vb.B51.MB.43.CB	1,27±0,03	49,41±1,32	38,94±1,85
Vd.B54.IWP.39.NB	1,12±0,21		

- For interior wall plaster samples their bulk density, effective porosity and water absorption capacity were determined to be in the range of 1.12±0.21 and

1.48±0.11 g/cm3, 42.8±3.48 and 51.7±0.61 by volume, 29.2±4.49 and 42.4±0.57 by weight, respectively. The average values of these characteristics (ρ, φ, θ) were found to be 1.27±0.13 g/cm3, 49.0±4.2 by volume, 37.7±6.1 by weight, respectively.

- For exterior wall plaster samples their bulk density, effective porosity and water absorption capacity were determined to be in the range of 1.28±0.02 and 1.41±0.01 g/cm^3, 45.9±0.45 and 50.0±0.88 by volume, 32.5±0.60 and 39.1±1.44 by weight, respectively. The average values of these characteristics (ρ, φ, θ) were found to be 1.34±0.07 g/cm3, 48.1±2.1 by volume, 36.1±3.3 by weight, respectively.
- For floor covering mortar samples their bulk density, effective porosity and water absorption capacity were determined to be in the range of 0.96±0.13 and 1.41±0.07 g/cm^3, 46.2±0.12 and 51.8±2.73 by volume, 33.3±0.02 and 44.2±3.02 by weight, respectively. The average values of these characteristics (ρ, φ, θ) were found to be 1.25±0.17 g/cm3, 49.4±2.9 by volume, 38.8±5.5 by weight, respectively.

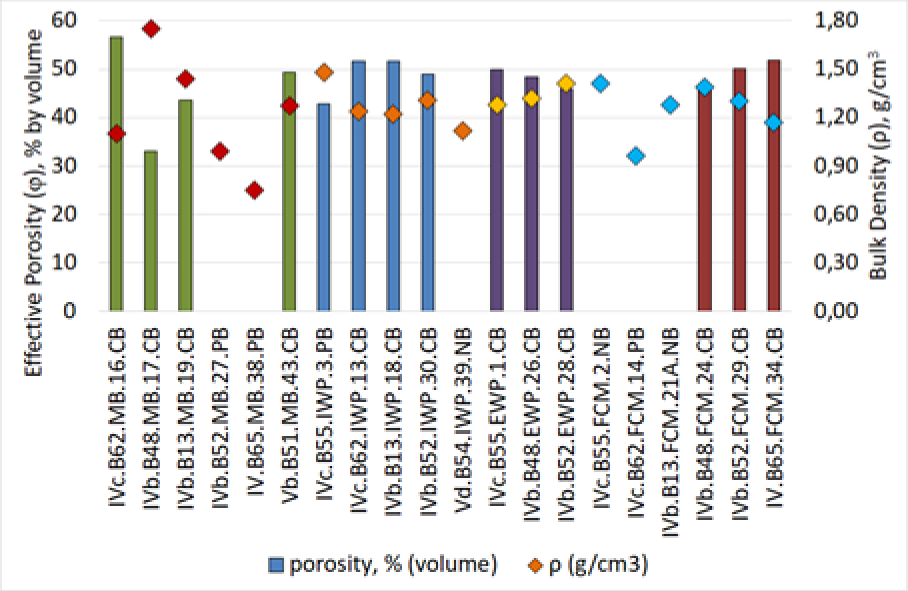


Figure 4.13 Bulk density (ρ) and porosity (φ), characteristics of samples.

In terms of porosity and bulk density values interior wall plasters, exterior wall plasters and floor covering mortars have similar physical properties independently of spatial and temporal changes. The physical properties of mudbrick samples are different from each other (Figure 4.1). Higher porosity values indicate that the mud samples have breathable characteristic.

4.1.2. Color Identification

The colors of the mudbrick, mud plaster and floor covering mortar samples are presented in Table 4.2. Samples were analyzed with Munsell color charts and their color were defined by hue, value and chroma of each sample.

The results show that the general color of the samples varying from very pale brown and pink/light red. The pink and light red color indicates the hematite presence and the deterioration rate of the samples.

Table 4.2. Color identification of the samples by their hue, value and chroma.

Samples	Hue	Value	Chroma	Colour
IV.B65.MB.38.PB	7.5Y	7	4	Pink
IV.B65.FCM.34.CB	7.5Y	7	4	Pink
IVb.B13.MB.19.CB	7.5Y	7	4	Pink
IVb.B13.FCM.21A.NB	10YR	7	3	Very pale brown
IVb.B48.MB.17.CB	2.5YR	7	4	Pale yellow
IVb.B48.FCM.24.CB	10YR	7	4	Very pale brown
IVb.B52.MB.27.PB	10YR	7	4	Very pale brown
IVb.B52.FCM.29.CB	10YR	7	4	Very pale brown
IVc.B62.MB.16.CB	2.5YR	6	8	Light red
IVc.B62.FCM.14.PB	7.5YR	8	4	Pink

4.2. Basic Physicomechanical Properties

The physicomechanical properties of the samples were determined in terms of ultrasonic pulse velocity and modulus of elasticity values.

4.2.1. Ultrasonic Pulse Velocity and Modulus of Elasticity

The UPV_{DIRECT} values of mudbrick samples with an average bulk density value of 1.14±0.30 g/cm^3 were determined to be in a range of 488±188 and 751±139 m/s with an average of 595±128 m/s. E_{mod} values of the mudbrick samples were determined to be in a range of 0.18±0.13 and 0.77±0.28 GPa with an average of 0.43±0.27 GPa.

The UPV_{DIRECT} values of interior wall plaster samples with an average bulk density value of 1.28±0.13 g/cm^3 were determined to be in a range of 454±92 and 1067±248 m/s with an average of 809±272 m/s. E_{mod} values of the mudbrick samples were determined to be in a range of 0.24±0.10 and 1.57±0.22 GPa with an average of 0.90±0.57 GPa.

The UPV_{DIRECT} values of exterior wall plaster samples with an average bulk density value of 1.34±0.07 g/cm^3 were determined to be in a range of 651±266 and 987±444 m/s with an average of 827±169 m/s. E_{mod} values of the mudbrick samples were determined to be in a range of 0.56±0.45 and 1.40±1.14 GPa with an average of 0.95±0.42 GPa.

The UPV_{DIRECT} values of floor covering mortar samples with an average bulk density value of 1.23±0.07 g/cm^3 were determined to be in a range of 389±97 and 1110±242 m/s with an average of 750±510 m/s. E_{mod} values of the mudbrick samples were determined to be in a range of 0.17±0.08 and 1.48±0.63 GPa with an average of 0.83±0.93 GPa.

Table 4.3. Physicomechanical properties of mudbrick, mud plaster, floor covering mortar samples: UPV$_{DIRECT}$ (m/s) and E$_{mod}$

Samples	ρ, (g/cm^3)	UPV$_{DIRECT}$ (m/s)	E$_{mod}$ (GPa)
IV.B65.FCM.34.CB	1,17	389±97	0.17±0.08
IV.B65.MB.38.PB	0,75	488±188	0.18±0.13
IVb.B13.FCM.21A.NB	1,28	1110±242	1.48±0.63
IVb.B13.IWP.18.CB	1,26	1067±248	1.57±0.22
IVb.B13.MB.19.CB	1,44	751±139	0.77±0.28
IVb.B48.EWP.26.CB	1,32	843±190	0.89±0.39
IVb.B52.EWP.28.CB	1,41	987±444	1.40±1.14
IVb.B52.IWP.30.CB	1,30	583±5	0.41±0.01
IVc.B55.EWP.1.CB	1,28	651±266	0.56±0.45
IVc.B55.IWP.3.PB	1,48	988	1,33
IVc.B62.IWP.13.CB	1,24	454±92	0.24±0.10
IVc.B62.MB.16.CB	1,10	491±80	0.25±0.08
Vb.B51.MB.43.CB	1,27	649±104	0.50±0.16
Vd.B54.IWP.39.NB	1,12	955±58	0.95±0.12

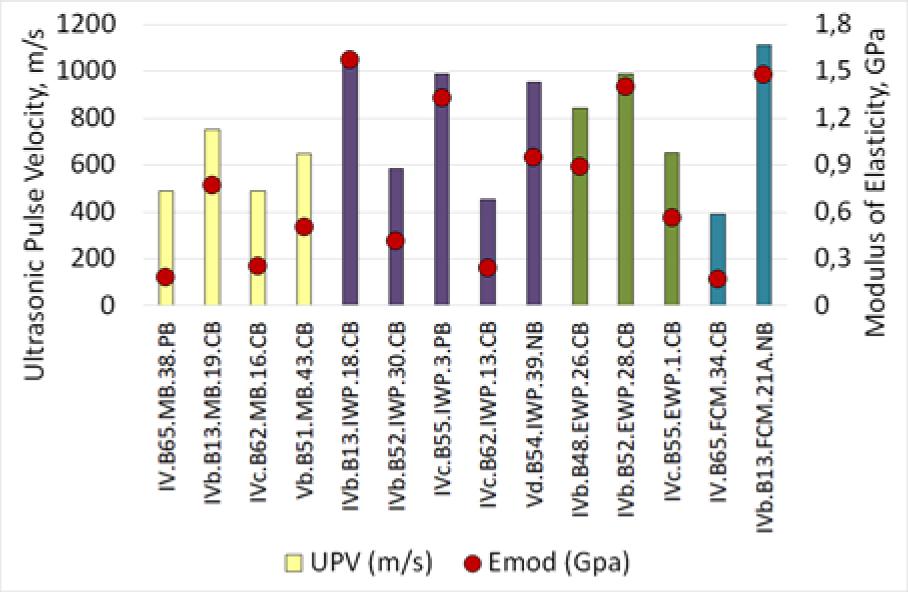


Figure 14.2. Ultrasonic pulse velocity (m/s) and modulus of elasticity (GPa) characteristics of samples.

The UPV values of the mudbricks and exterior wall plaster samples are in the same range in their group while the interior wall plaster and floor covering mortar samples have different values with each other (Figure 4.2). Although the mud samples were affected by different degrees from the fire, the obtained physico-mechanical results indicate that these samples have good durability properties

4.3. Compositional Properties

The compositional properties of the samples were determined in terms of loss on ignition, binder-aggregate proportion, particle size distribution and pozzolanic activity values.

4.3.1. Loss on Ignition

Loss on ignition experiment were performed both the all of the whole samples and the finest aggregates of the seven samples. The results of the loss on ignition analyses of the whole samples are given in the Table 4.4. The amount of organic matter in the samples ranged from 3,30±0,18% to 8,43±0,07% by mass. Considering that the samples were damaged at various levels due to fire, the results obtained are considered to have a certain margin of error. The amount of calcium carbonate ($CaCO_3$) in the samples ranged from 17,01±0,46% to 46,09±4,34% by mass. The mud samples of the Building 52 (IVb sublayer), Building 55 and Building 62 (IVc sublayer) have similar calcium carbonate percentage (Figure 4.3). The mud samples of the Building 65 (IV layer), Building 48 and Building 13 (IVb sublayer) have in different range of calcium carbonate percentage. The results indicate that 75% of the samples have calcium carbonate more than 20% percentage.

Table 4.4. % organic matter and % $CaCO_3$ values of the whole samples.

Sample Name	Organic matters, %	$CaCO_3$, %
	% by mass	% by mass
IV.B65.MB.38.PB	4,20±0,07	35,37±1,83
IV.B65.FCM.34.CB	8,39±1,50	25,46±3,52
IVb.B13.MB.19.CB	4,03±0,18	26,17±0,72
IVb.B13.FCM.21A.NB	5,13±0,01	46,09±4,34
IVb.B13.IWP.18.CB	8,43±0,07	17,01±0,46
IVb.B48.MB.17.CB	4,11±0,03	35,66±0,70
IVb.B48.FCM.24.CB	3,30±0,18	40,62±2,02
IVb.B48.EWP.26.CB	6,44±0,09	18,01±0,78
IVb.B52.MB.27.PB	4,40±0,08	28,31±0,19
IVb.B52.FCM.29.CB	5,50±0,03	26,88±0,13
IVb.B52.IWP.30.CB	5,20±0,10	30,58±0,49
IVb.B52.EWP.28.CB	5,95±0,03	23,36±0,17
IVc.B55.FCM.2.NB	7,43±0,04	31,45±0,06
IVc.B55.IWP.3.PB	4,19±0,14	33,24±0,56
IVc.B55.EWP.1.CB	6,04±0,28	31,40±0,78
IVc.B62.MB.16.CB	4,19±0,08	20,51±4,05
IVc.B62.FCM.14.PB	5,31±0,77	27,90±1,64
IVc.B62.IWP.13.CB	4,22±0,10	20,57±0,45
Vb.B51.MB.43.CB	4,51±0,03	19,26±0,88
Vd.B54.IWP.39.NB	3,26±0,06	37,07±0,47
IV.B65.MB.38.PB	4,20±0,07	35,37±1,83

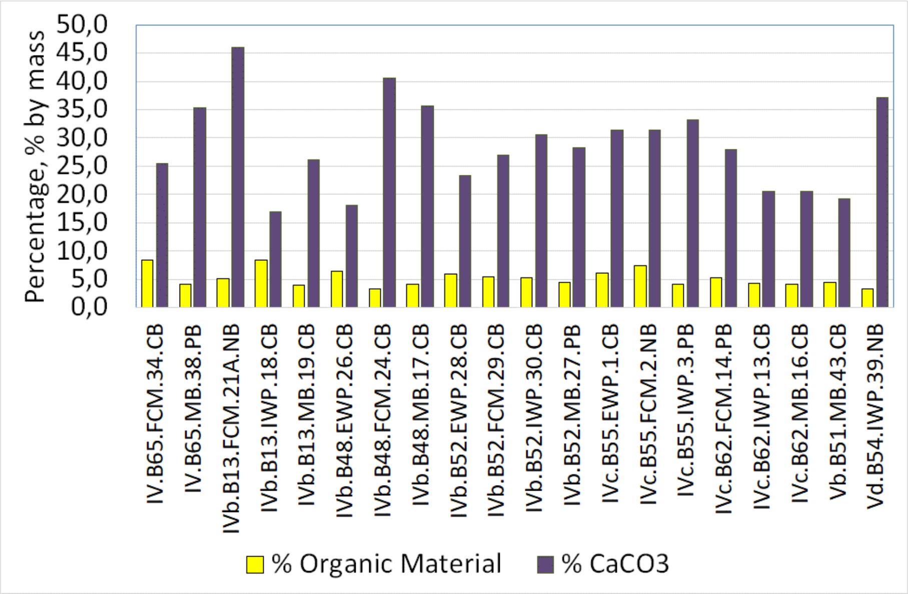


Figure 4.3. % organic matter and % $CaCO_3$ values of the whole samples.

The loss on ignition experimental results of the finest part of the seven samples are given in the Table 4.5. The amount of organic matter in the samples ranged from 2,54±1,30% to 9,91±0,15% by mass. The amount of calcium carbonate in the samples ranged from 21,03±0,75% to 54,79±16,61% by mass. The results indicate that the finest part of the interior wall plaster and floor covering mortar samples in the IVc sublayer (IVc.B55.FCM.2.NB, IVc.B55.IWP.3.PB and IVc.B62.FCM.14.PB) have similar calcium carbonate (average 36,5%) and organic matter content (average 6,7%) (Figure 4.4.). The finest part of the mudbrick samples (IV.B65.MB.38.PB and IVb.B52.MB.27.PB) have similar calcium carbonate (average 22,9%) and organic matter content (average 8,5%) (Figure 4.4.).The floor covering sample IVb.B13.FCM.21A.NB have the highest calcium carbonate (54,8%) and lower organic matter (2,54%). IVb sublayer samples results show varieties in terms of calcium carbonate and organic matter content. The finest parts of the samples have higher ratio of calcium carbonate and similar ratio of organic matter than the whole samples.

Table 4.5. % organic matter and % clay-sized $CaCO_3$ values of the samples.

Sample Name	Organic matter, %	$CaCO_3$, %
	% by mass	% by mass
IV.B65.MB.38.PB	9,25±0,03	24,75±0,14
IVb.B13.FCM.21A.NB	2,54±1,30	54,79±16,61
IVb.B52.MB.27.PB	7,68±0,21	21,03±0,75
IVc.B55.FCM.2.NB	6,64±0,42	34,7±8,75
IVc.B55.IWP.3.PB	5,71	38,09
IVc.B62.FCM.14.PB	5,77	36,84
Vd.B54.IWP.39.NB	9,91±0,15	29,79±0,30

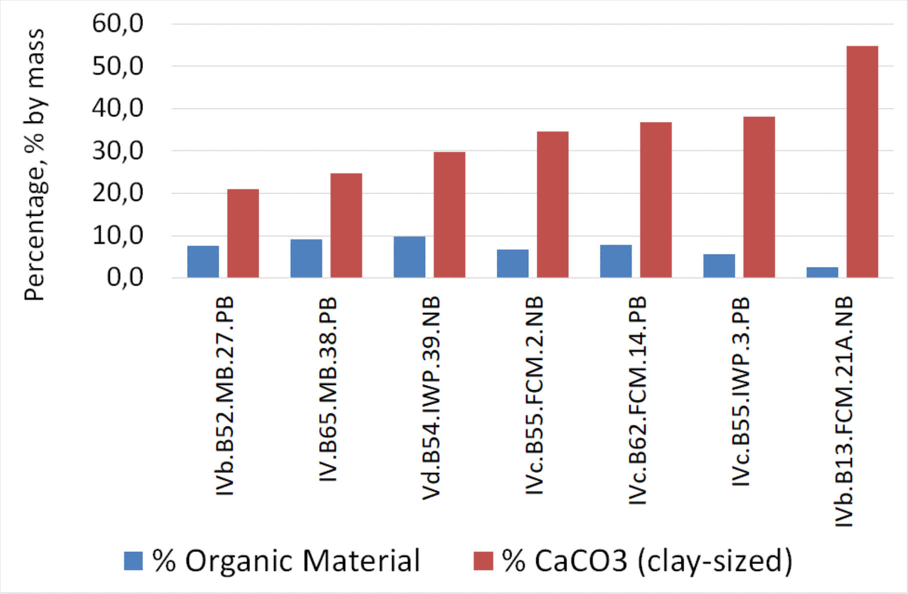


Figure 4.4. % organic matter and % $CaCO_3$ (clay-sized) values of the silt-clay mixture of the samples.

4.3.2. Particle Size Distribution of Aggregates

Particle size distribution analyses were performed only six samples (2 mudbrick samples, 3 floor covering mortar and 1 interior wall plaster) which were partially burnt and not burnt. In all the examined samples, clay and silt size portions are in the range of 17% - 71% by weight (Figure 4.5.). Clay and silt-sized parts are accepted to act as "binders" in mud samples and the presence of 20% -30% binder in adobe materials is important in terms of ensuring the durability of adobe (Brown and Clift, 1979). The floor covering mortar IVc.B62.FCM.14.PB has the lowest binder part and interior wall plaster sample Vd.B54.IWP.39.NB has the highest binder part (Figure 4.5.). Almost all samples have sufficient silt-clay part in terms of good quality of mud materials.

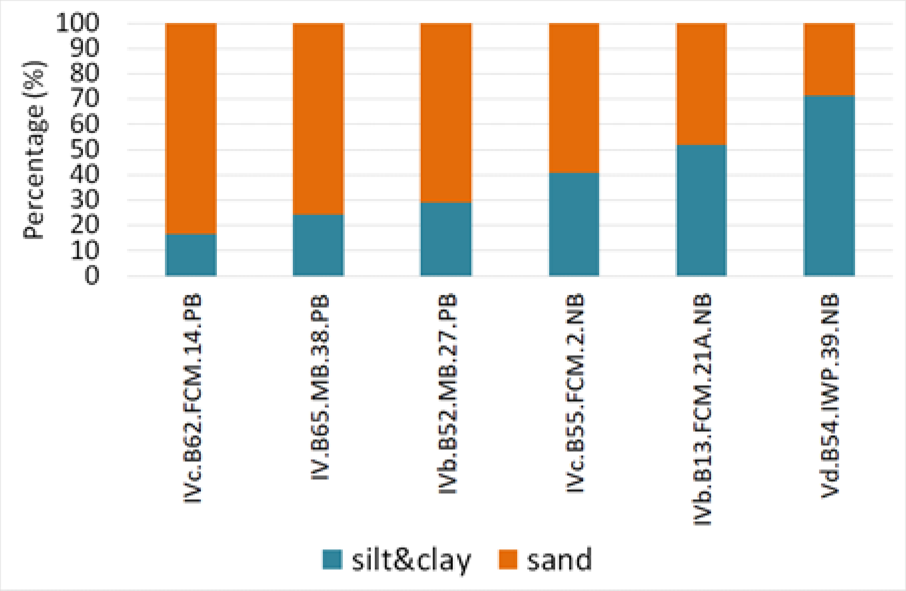


Figure 4.15. The percentage of silt&clay part and sand part of the samples.

The particle size distribution of the samples indicates that there are three different compositional properties in terms of the usage of the samples as a mudbrick, interior wall plaster and floor covering mortar. (Figure 4.6. and Figure 4.7.). The interior wall plaster sample Vd.B54.IWP.39.NB has the 87% percentage of the fine particles below 0,5mm. This ratio is very high, and compatible with the interior wall plaster properties. The floor covering mortar samples IVc.B55.FCM.2.NB and IVb.B13.FCM.21A.NB have 68-70% percent fine particles below 0.5 mm (Figure 4.6. and Figure 4.7.). These two samples show similar aggregates distribution although they belong to different time period and building. The mudbrick samples IV.B65.MB.38.PB, IVb.B52.MB.27.PB and the floor covering mortar IVc.B62.FCM.14.PB have 46-52% percent fine particles below 0.5mm (Figure 4.6. and Figure 4.7.). The mudbrick samples have similar aggregate distribution and they have approximately 50% fine and 50% coarse sand. This type of aggregate distribution for the mudbrick samples is evaluated as a good composition in terms of the durability, porosity and strength properties. The floor covering mortar IVc.B62.FCM.14.PB shows different properties from the other two floor covering mortars that indicates this sample could be a lower flooring layer of the building.

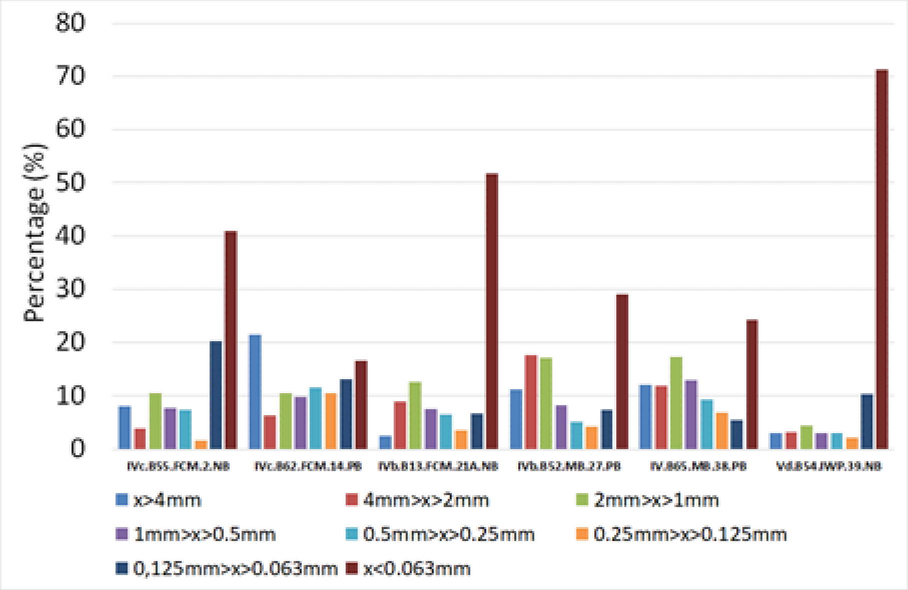


Figure 4.16. Particle size distribution of the Ulucak Höyük mud-based construction materials.

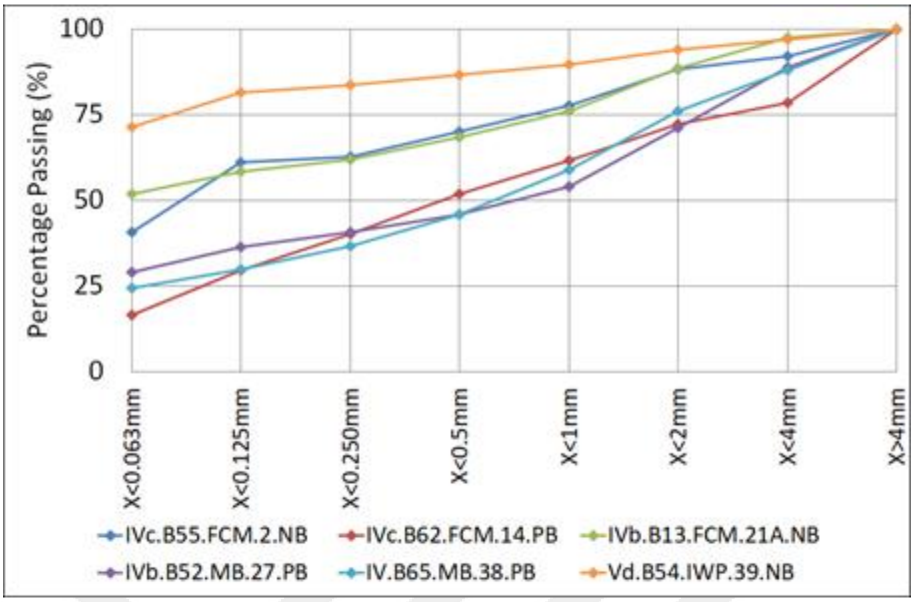


Figure 17. Cumulative particle size distribution of the samples.

4.3.3. Pozzolanic Activity

The pozzolanic activity experiment were performed for the burnt and partially burnt samples and the results are given in the Table 4.6. The consumed amount of EDTA of mudbricks were found to be between 12.7 and 16.8 ml. These values are inversely proportional to their pozzolanic activities. The consumed amount of EDTA of the blank solution, which is not pozzolan, is 19.7 ml. According to the blank solution, the consumed amount of $Ca(OH)_2$ solution of samples was found to be between 124.2 and 303.3 mg.

Although all samples are determined to have high pozzolanic activity value the average pozzolanic activity of the IVc sublayer samples are lower (175±20 mg) than the average pozzolanic activity of the IVb sublayer samples (208±62 mg) (Figure 4.8.). Since clay minerals gain high pozzolanic activity by the firing between 600-900°C, pozzolanic activity of the samples also indicates the firing temperature of the samples.

Table 4.6. Pozzolanic activity values of the burnt and partially samples.

Sample	Consumed EDTA (ml)	Consumed $Ca(OH)_2$ (mg)
IV.B65.FCM.34.CB	12,7	303,3
IV.B65.MB.38.PB	14,7	217,1
IVb.B13.IWP.18.CB	15,6	179,4
IVb.B13.MB.19.CB	13,15	282,7
IVb.B48.EWP.26.CB	15,1	204,5
IVb.B48.FCM.24.CB	16,1	152,4
IVb.B48.MB.17.CB	12,8	303,1
IVb.B52.EWP.28.CB	15,3	189,6
IVb.B52.FCM.29.CB	13,3	277,3
IVb.B52.IWP.30.CB	16,4	143,8
IVb.B52.MB.27.PB	16,8	124,2
IVc.B55.EWP.1.CB	15,8	168,3
IVc.B55.IWP.3.PB	15,4	186,0
IVc.B62.IWP.13.CB	16,2	150,2
IVc.B62.MB.16.CB	15,1	196,7
Vb.B51.MB.43.CB	14,6	218,9
Blank Sample	19,7	

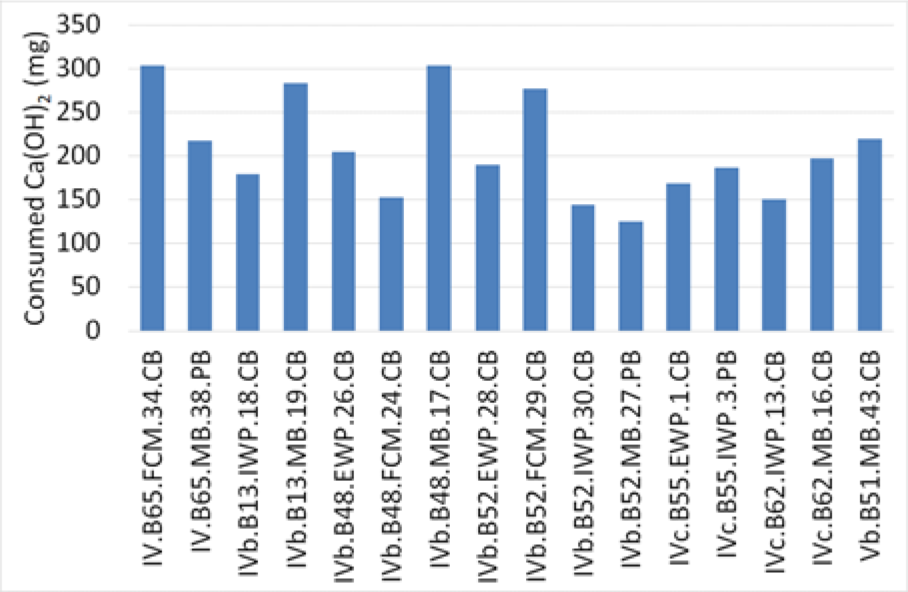


Figure 18. Pozzolanic activity values of the burnt and partially burnt mud samples.

4.4. Quantitative Analysis of the Soluble Salts

The results for determining the presence of the quantity of soluble salts in mudbrick samples were shown in Table 4.7. The results show that the amount of salt in Ulucak Höyük mudbrick dwellings materials is negligible and that there is no problem caused by salt in the dwellings.

Table 4.7. The salt amount of the samples.

Sample	Amount of Salt, (%)
IV.B65.FCM.34.CB	0,18
IV.B65.MB.38.PB	0,19
IVb.B13.FCM.21A.NB	0,21
IVb.B13.IWP.18.CB	0,29
IVb.B13.MB.19.CB	0,23
IVb.B48.EWP.26.CB	0,18
IVb.B48.FCM.24.CB	0,19
IVb.B48.MB.17.CB	0,16
IVb.B52.EWP.28.CB	0,20
IVb.B52.FCM.29.CB	0,20
IVb.B52.IWP.30.CB	0,21
IVb.B52.MB.27.PB	0,17
IVc.B55.EWP.1.CB	0,21
IVc.B55.FCM.2.NB	0,17
IVc.B55.IWP.3.PB	0,18
IVc.B62.FCM.14.PB	0,21
IVc.B62.IWP.13.CB	0,20
IVc.B62.MB.16.CB	0,18
Vb.B51.MB.43.CB	0,21
Vd.B54.IWP.39.NB	0,21
IV.B65.FCM.34.CB	0,18

4.5. Microstructural Analyses

The microstructural analyses of the samples were determined in terms of thin section analyses, XRD analyses, FTIR analyses and cross section images of the samples. Results are given under respective subheadings.

4.5.1. Thin Section Analysis

Petrographic analyses were carried out for five different samples that were collected three different building. One of the mudbrick sample belongs to Building 51 (Vb.B51.MB.43.CB), one mudbrick and one floor covering mortar samples belongs to building 48 (IVb.B48.MB.17.CB and IVb.B48.FCM.24.CB) and one mudbrick and one floor covering mortar belong to building 62 (IVc.B62.MB.16.CB and IVc.B62.FCM.14.PB). Those samples represent the three different sublayers that one of early Neolithic Layer Vb and two of late Neolithic Layer IVb and IVc. Thin section analyses of individual samples were described below with their sample codes. The matrix of the sample was examined with respect to their mineralogical characteristics.

The general texture image of the samples (Figure 4.9.) and the image of the cracks/fiber voids (Figure 4.10.) are given below together to illustrate the difference between the textures. The rock and mineral variety of the samples are given separately under the relevant sub-headings.

The matrix of the mudbrick sample Vb.B51.MB.43.CB consist of aggregates majority of are crystals with fewer rock fragments (quartzite). These aggregates cemented with a clay binder containing very small mica flakes, which are intensely dyed with opaque minerals such as hematite/magnetite (Figure 4.9. - a). Pores and cracks were observed in thin section and the partially filled parts of the pores consist of a mixture of calcite and aragonite. Firing of organic additives like straw may have caused these pores (Figure 4.10. - a).

The matrix of the mudbrick sample IVb.B48.MB.17.CB consist of various sizes crystal and rock fragments which cemented with a micritic calcite and clay binder (Figure 4.9. - b). Binder is dyed with an opaque mineral. Pores were observed which may occur from burnt organic additives or falling of small pebbles (Figure 4.10.-b).

The matrix of the floor covering mortar sample IVb.B48.FCM.24.CB consist of crystals, rock fragments and mud lamps cemented with a very fine grained clayey-calcareous binder which painted with iron oxide (Figure 4.9. - c). Pores and cracks were observed; pore walls are plastered with calcite (Figure 4.10. - c).

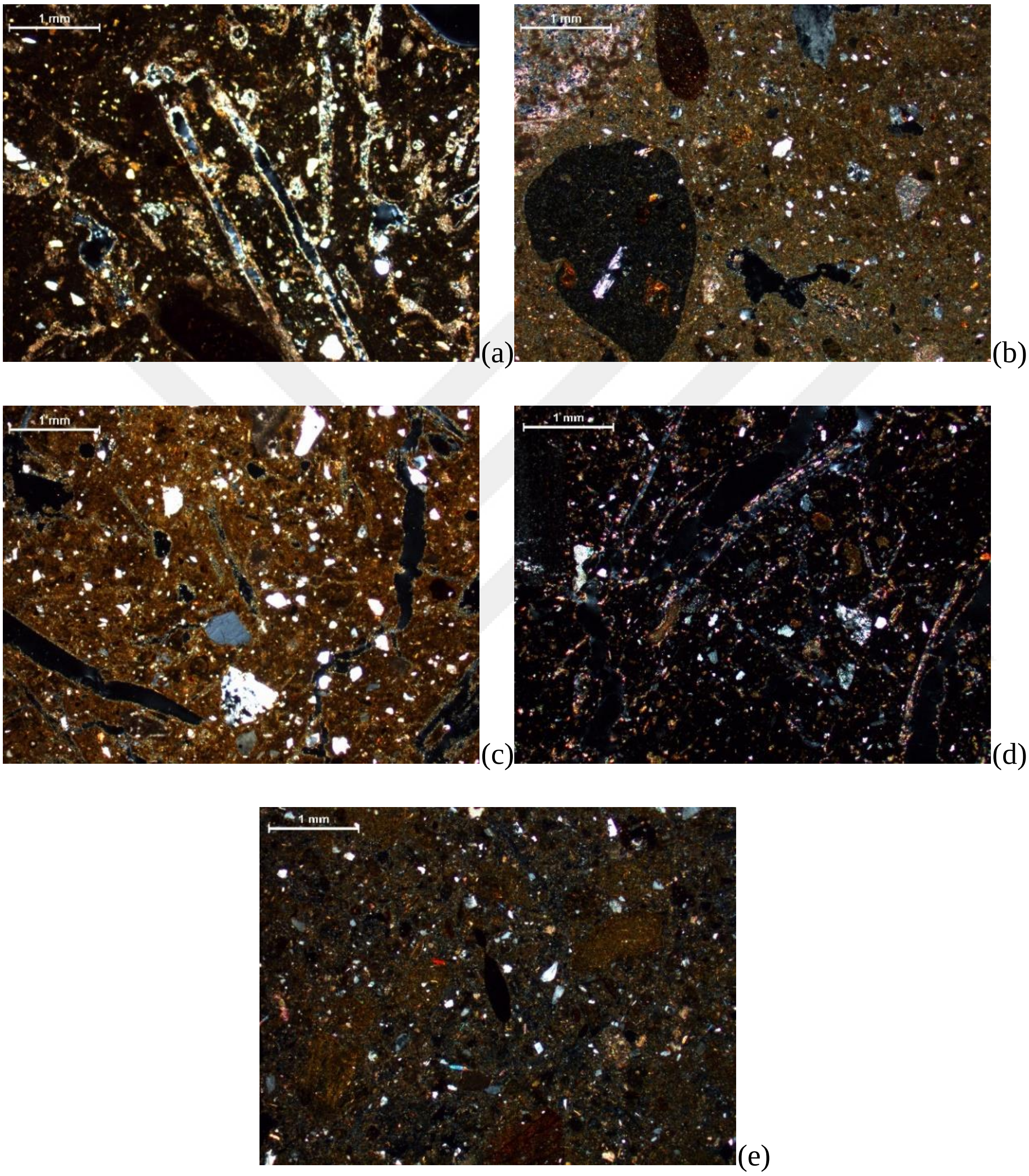


Figure 19. Thin section images of the general texture of the samples at 2.5x magnification (a) Mudbrick (Vb.B51.MB.43.CB), (b) Mudbrick (IVb.B48.MB.17.CB), (c) Floor Covering Mortar (IVb.B48.FCM.24.CB), (d) Mudbrick (IVc.B62.MB.16.CB), (e) Floor Covering Mortar (IVc.B62.FCM.14.PB)

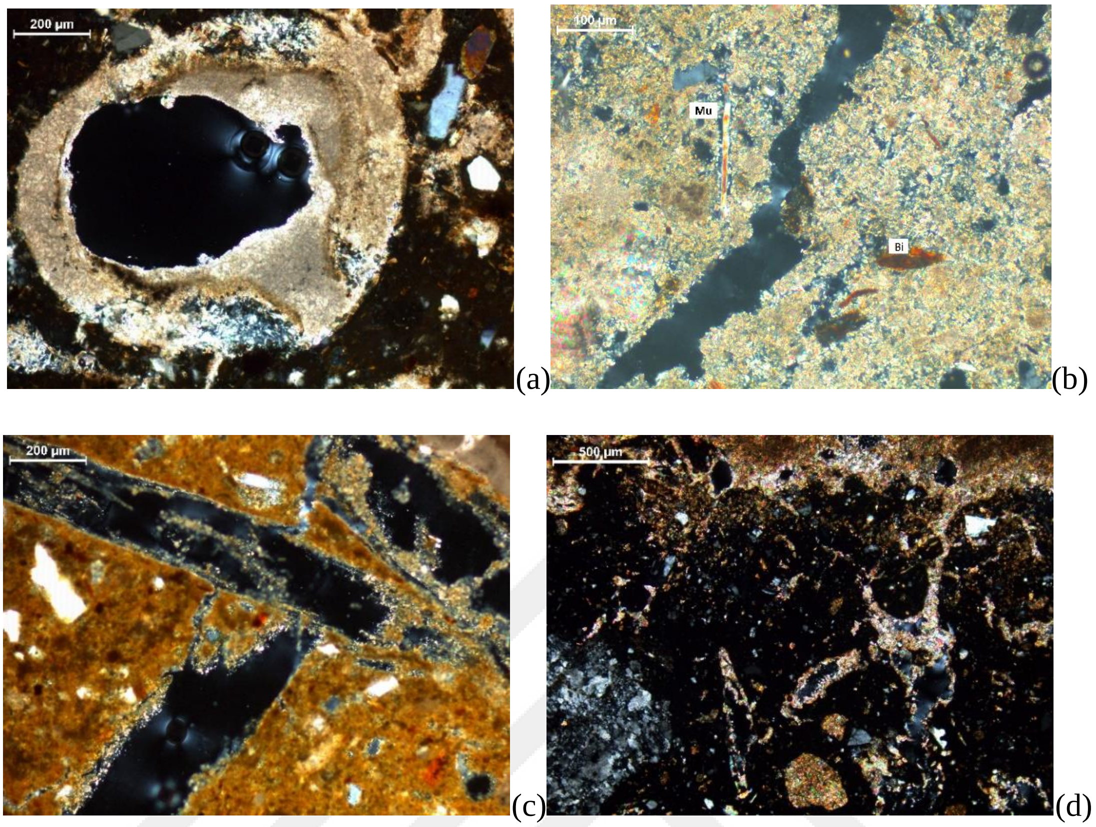


Figure 4.20. Thin section images of the pores and fibre voids of the samples (a) Mudbrick (Vb.B51.MB.43.CB)_10x magnification, (b) Mudbrick (IVb.B48.MB.17.CB)_20x magnification (c) Floor Covering Mortar (IVb.B48.FCM.24.CB)_10x magnification, (d) Mudbrick (IVc.B62.MB.16.CB)_5x magnification.

The matrix of the mudbrick sample IVc.B62.MB.16.CB consist of ferrous opaque mineral fragments and very small quartz grains are present in the clayey binder painted with iron oxide and hydroxides (Figure 4.9. - d). Pores and cracks are filed with calcite crystals (Figure 4.10. - d).

The matrix of floor covering mortar sample IVc.B62.FCM.14.PB is consist of the aggregates and mud lamps which dimensions varied between micron size to 6 mm. This sample have large amount of aggregates with fewer clayey-calcareous binder (Figure 4.9. - e). Pores or cracks are not observed in this sample.

The Rock Fragments and Crystals of Mudbrick Vb.B51.MB.43.CB: Quartz, calcite, biotite, muscovite, sanidine, tourmaline and opaque minerals were the most common crystals observed in the matrix. In addition, quartzite was observed as a metamorphic rock fragments, flintstone were observed as a sedimentary rock fragment.

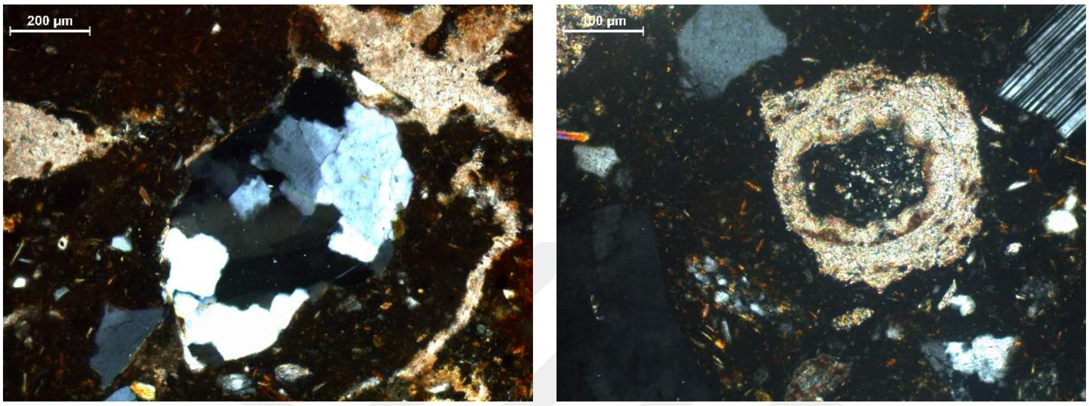


Figure 4.11. The rock fragments and crystals of mudbrick Vb.B51.MB.43.CB a. quartzite, quartz, cracks with filling calcite. b. orthoclase, quartz, schist, cracks with filling calcite

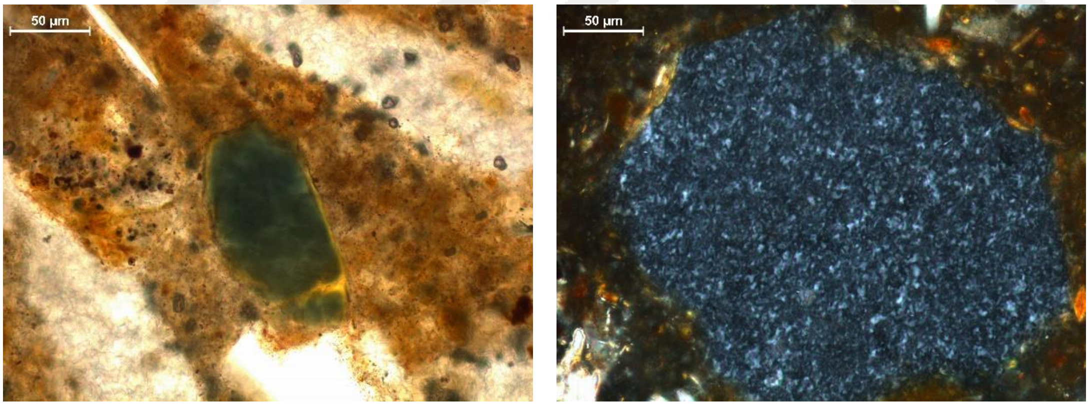


Figure 4.12. The rock fragments and crystals of mudbrick Vb.B51.MB.43.CB a. *tourmaline*. b. flintstone clast

The Rock Fragments and Crystals of Mudbrick IVb.B48.MB.17.CB: Cataclastic quartz, calcite, plagioclase, biotite, muscovite, opaque minerals were the most common crystals observed in the matrix. In addition, olivine basalt (igneous rock fragment), biotite-quartz schist and quartzite (metamorphic rock fragment), limestone and dolomitic clay stone (sedimentary rock fragment) were observed.

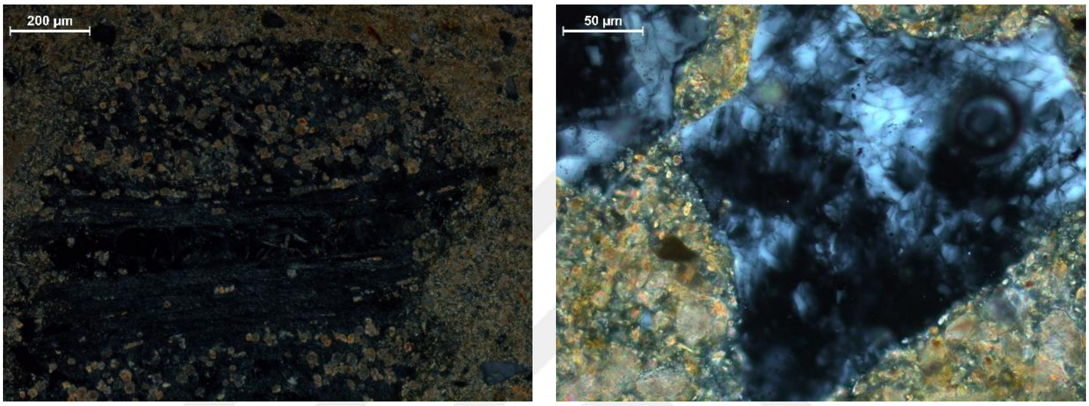


Figure 4.13. The rock fragments and crystals of mudbrick IVb.B48.MB.17.CB a- dolomitic clay stone b. cataclastic quartz

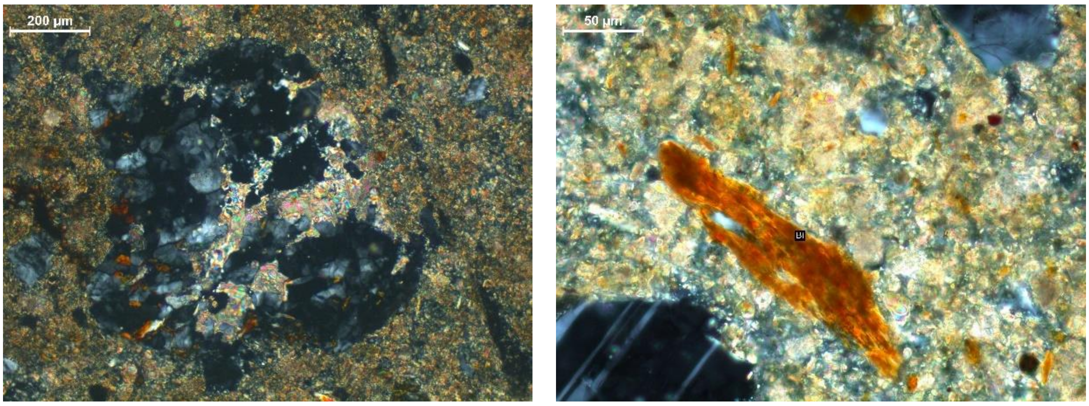


Figure 4.14. The rock fragments and crystals of mudbrick IVb.B48.MB.17.CB a- quartzite clast, its cracks filled with calcite b. quartz, biotite, plagioclase

The Rock Fragments and Crystals of Floor Covering Mortar IVb.B48.FCM.24.CB: Cataclastic quartz, calcite, plagioclase, biotite, muscovite, sanidine were the most common crystals. In addition, rock clast with cataclastic quartz (igneous rock fragment), cataclastic quartzite (metamorphic rock fragment), dolostone, flintstone, crystalline lime clast (sedimentary rock fragments) were also frequently observed in thin section images.

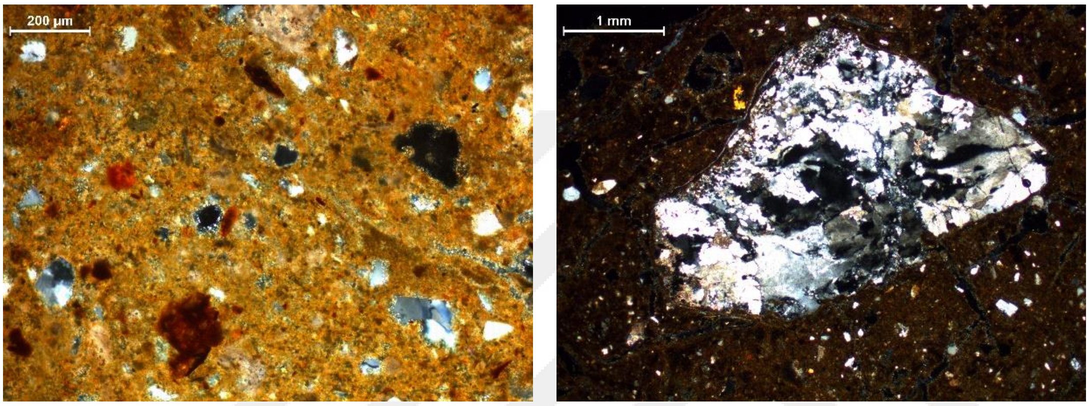


Figure 4.215. The rock fragments and crystals of mudbrick IVb.B48.FCM.24.CB a- quartz. b. cataclastic quartzite

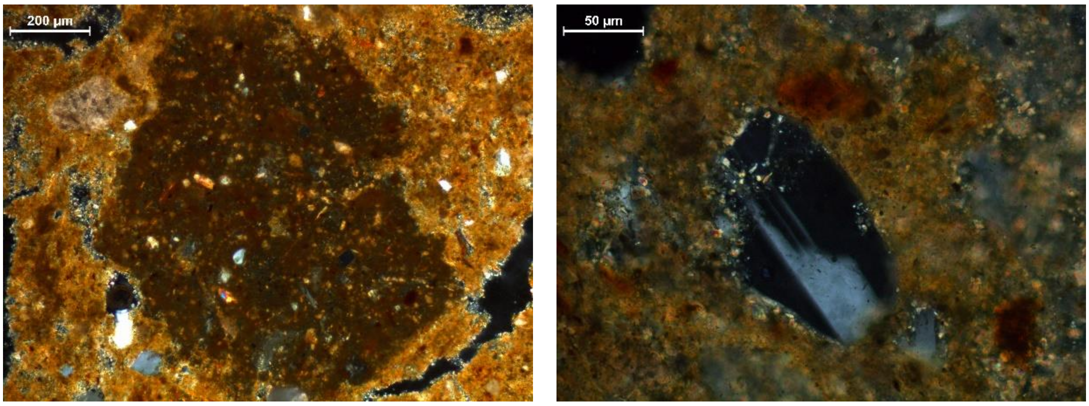
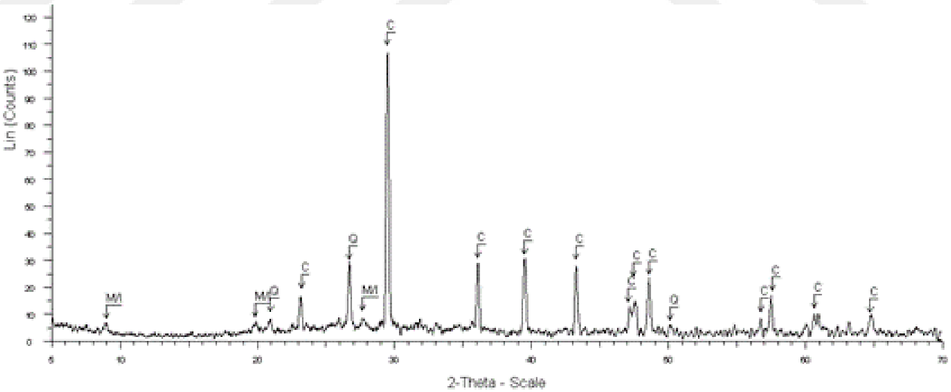


Figure 4.16. The rock fragments and crystals of mudbrick IVb.B48.FCM.24.CB a- mud lamp. b. plagioclase

4.5.2. X-Ray Diffraction Analyses (XRD)

XRD analyses were performed both the whole of all samples and the clay size of the seven unburnt and partially burnt samples. XRD traces of the whole samples show the major mineralogical composition that gives information about the raw material resources. XRD traces of the silt-clay size of the samples represents the major clay type of the samples.

Clay Size XRD Traces of the Samples: Calcite, quartz and mica/illite group were determined in XRD traces of the clay sample IV.B65.MB.38.PB. The exposure of the clay sample to ethylene glycol and the temperature of 500 degrees did not cause any change in the composition. It is observed that the material contains a significant proportion of calcite in clay size. It is thought that this may be caused by limestone, which is widely used as raw material in the region (Figure 4.17).



a

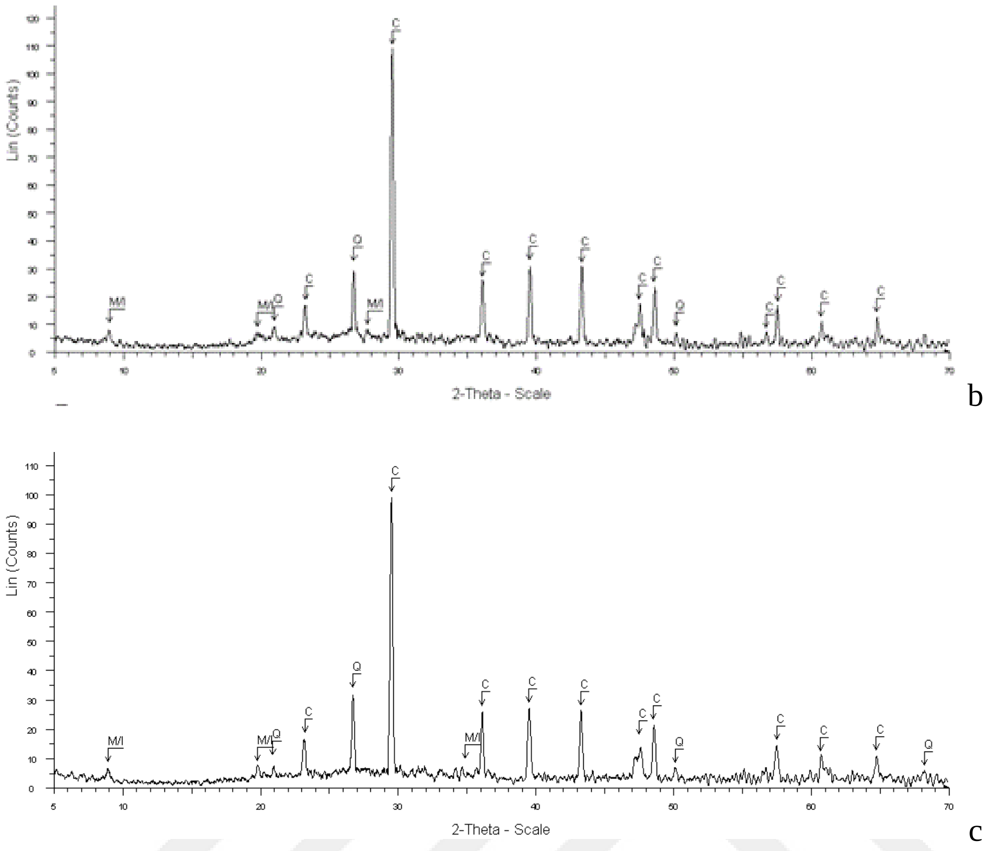


Figure 4.17. XRD traces of the sample IV.B65.MB.38.PB a-oriented clay part. b- after ethylene glycol. c- after exposed 500ºC. (C: Calcite, Q: Quartz, M/I: Mica/Illite)

Calcite, quartz, mica/illite group and kaolinite peaks were determined in XRD traces of the clay sample IVb.B13.FCM.21A.NB. The exposure of the clay sample to ethylene glycol did not cause any change in the composition. It is observed that the material contains a significant proportion of calcite in clay size. After exposing the sample with acetic acid, the calcite part was removed from the sample therefore the mica/illite and kaolinite group peaks were become more visible. Also, smectite group peaks and feldspar group peaks came in sight (Figure 4.18.).

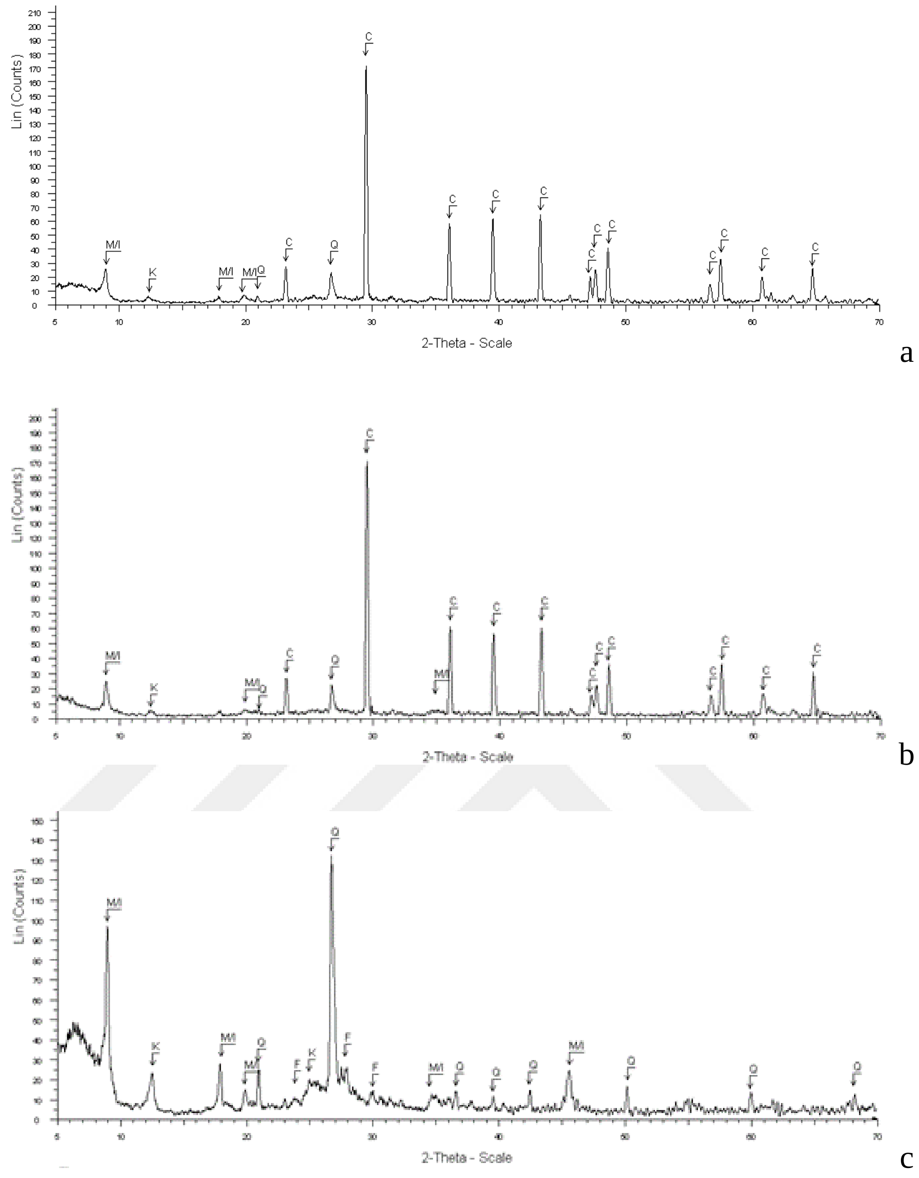


Figure 4.18. XRD traces of the sample IVb.B13.FCM.21A.NB a-oriented clay part. b- after ethylene glycol. c- after exposed acetic acid. (C: Calcite, Q: Quartz, M/I: Mica/Illite, K: Kaolinite, F: Feldspar)

Calcite, quartz and mica/illite group were determined in XRD traces of the clay sample IVb.B52.MB.27.PB. The exposure of the clay sample to ethylene glycol and the temperature of 500 degrees did not cause any change in the composition. It is observed that the material contains a significant proportion of calcite in clay size. It is thought that this may be caused by limestone, which is widely used as raw material in the region.

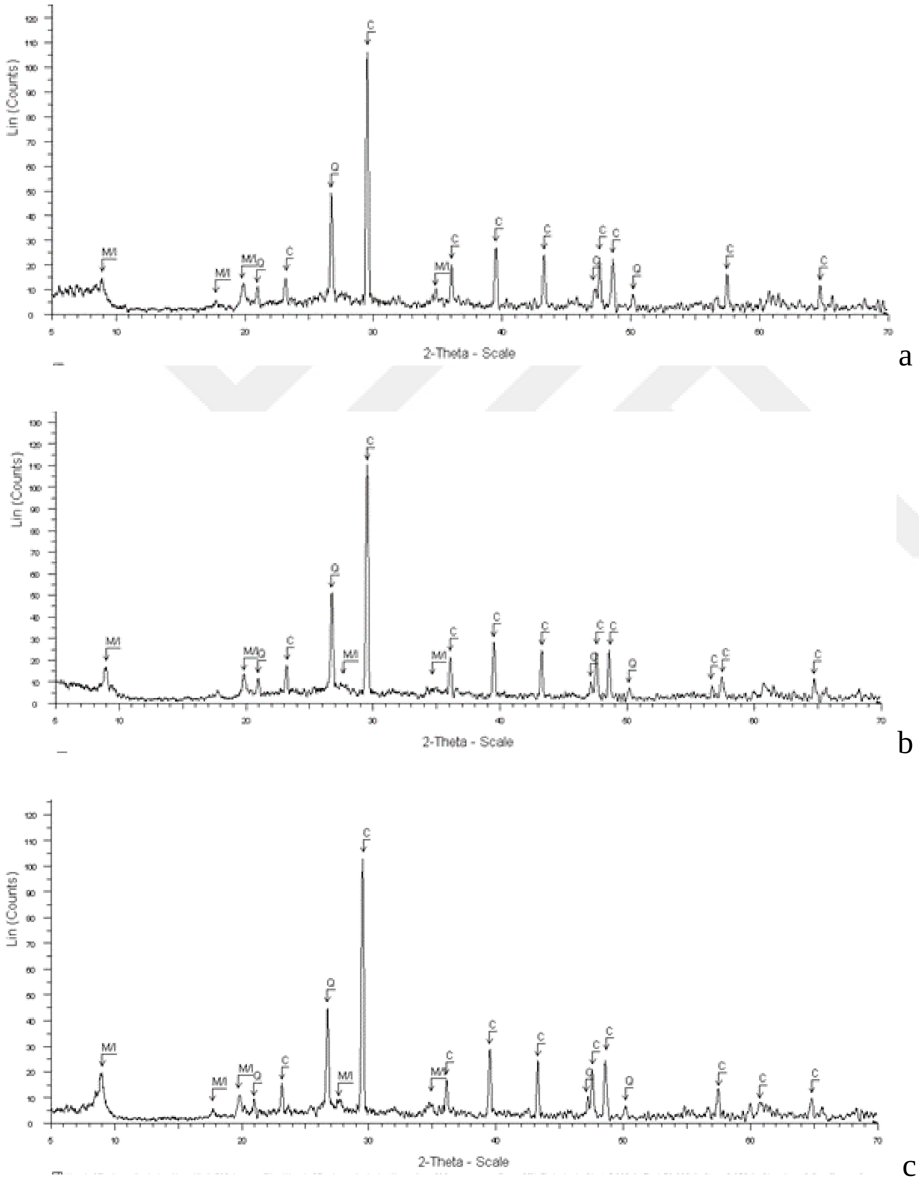
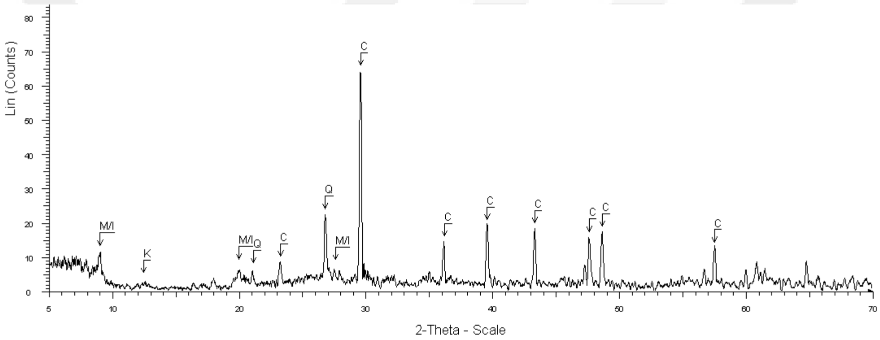
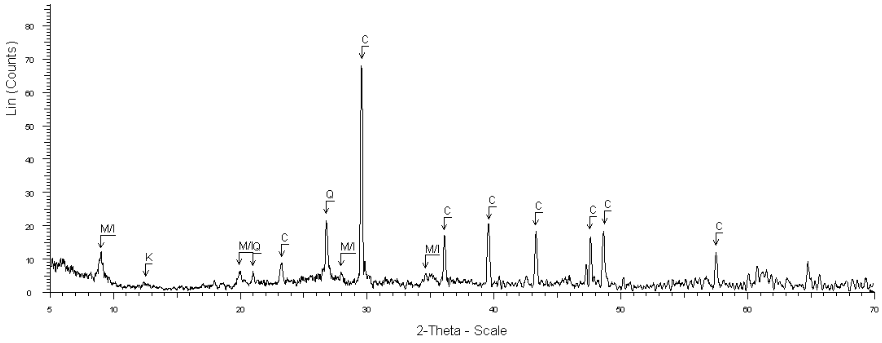


Figure 4.19. XRD traces of the sample IVb.B52.MB.27.PB a-oriented clay part. b- after ethylene glycol. c- after exposed 500ºC. (C: Calcite, Q: Quartz, M/I: Mica/Illite)

Calcite, quartz and mica/illite and kaolinite peaks were determined in XRD traces of the clay sample IVc.B55.FCM.2.NB. The exposure of the clay sample to ethylene glycol did not cause any change in the composition. Heating the clay sample to the temperature of 500 degrees makes the mica/illite peaks more visible and kaolinite peaks disappear. It is observed that the material contains a significant proportion of calcite in clay size. After exposing the sample with acetic acid, the calcite part was removed from the sample therefore the mica/illite and group peaks were become more visible. Also, kaolinite, feldspar group and group peaks came in sight



(a)



(b)

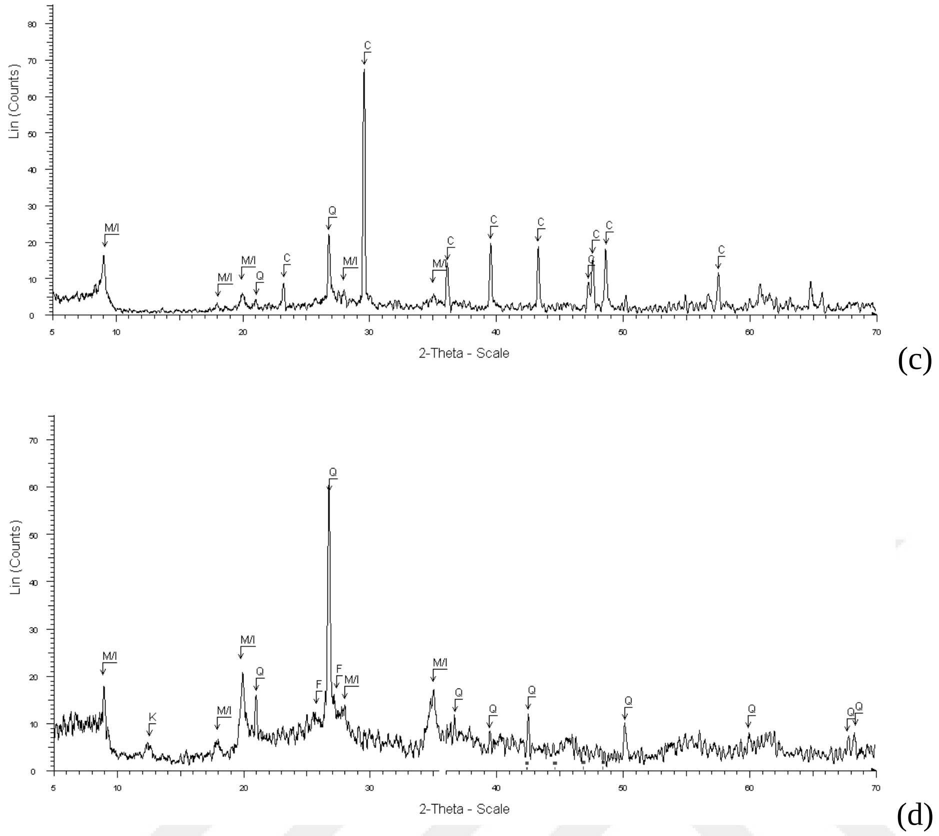
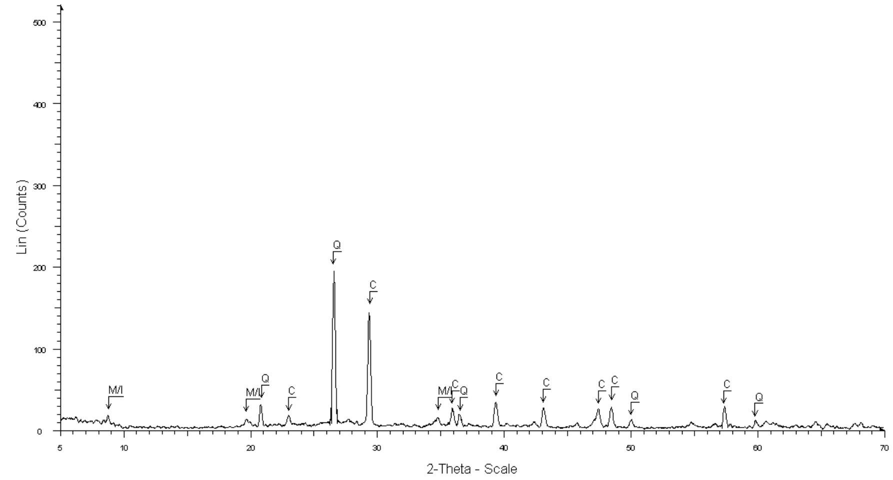


Figure 22.20. XRD traces of the floor covering mortar finest aggregates (IVc.B55.FCM.2.NB). a- oriented. b- ethylene glycolated. c- heated at 500ºC. d- treated with acetic acid. (C: Calcite, Q: Quartz, M/I: Mica/Illite, K: Kaolinite, F: Feldspar)

Whole part of the Samples' XRD Traces: XRD traces of some representative samples are given in the Figure 4.21. The main minerals are calcite, quartz and mica/illite for all samples. Some samples also have feldspar, anorthite and hematite peaks. All the presence of minerals is shown detailed in the Table 5.1.


Lin (Counts)
2-Theta - Scale
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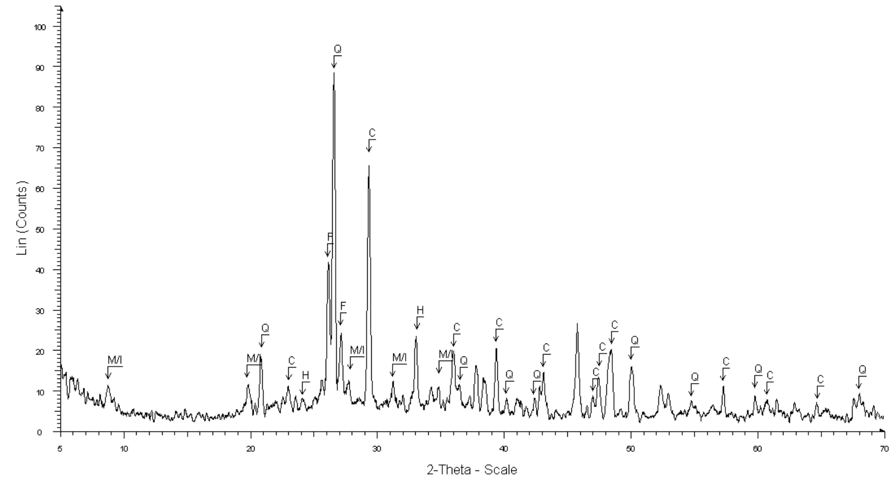
(a)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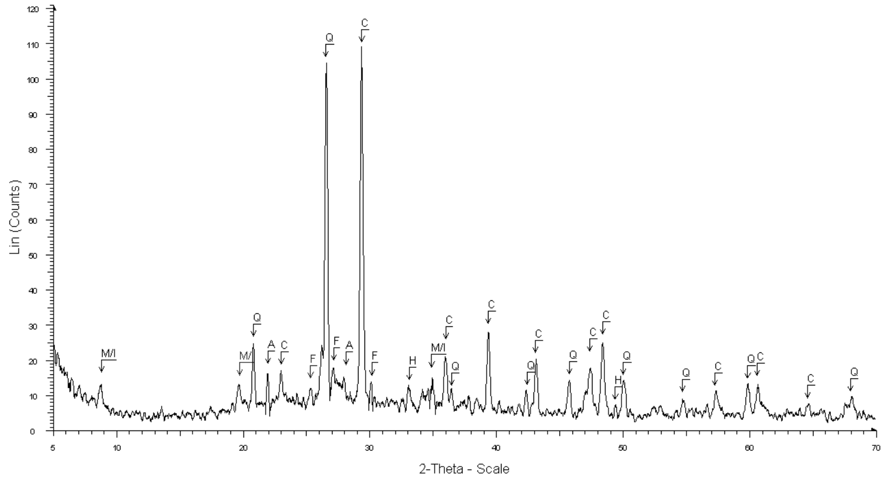
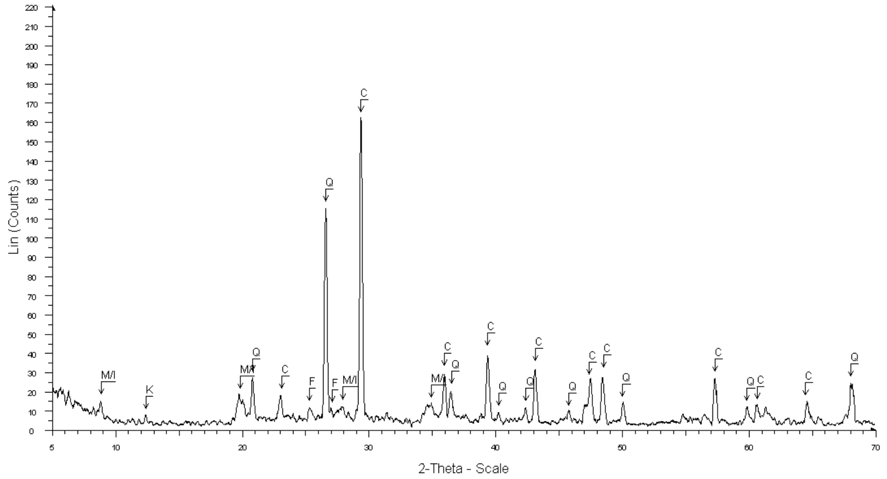
(b)



(c)



(d)

Figure 4.21. XRD traces of the samples' whole part a) mudbrick IVb.B52.MB.27.PB b) mudbrick IV.B65.MB.38.PB c) Interior wall plaster IVb.B13.IWP.18.CB d) Interior wall plaster Vd.B54.IWP.39.NB. (C: Calcite, Q: Quartz, M/I: Mica/Illite, K: Kaolinite, F: Feldspar, H: Hematite, A: Albite)

Table 5.1. The minerals determined by XRD analyses of Ulucak Höyük mud-based samples (C: Calcite, Q: Quartz, M/I: Mica / Illite group H, F: Feldspar, Hematite, A: Albite, Mn: Montmorillonite, K: Kaolinite)

Sample Code	C	Q	M/I	F	H	A	K
IV.B65.MB.38.PB	+	+	+	+	+		
IV.B65.FM.34.CB	+	+	+	+	+		
IVb.B13.MB.19.CB	+	+	+				
IVb.B13.CEL.21A.NB	+	+	+				
IVb.B13.IWP.18.CB	+	+	+	+	+	+	
IVb.B48.MB.17.CB	+	+	+	+			
IVb.B48.FM.24.CB	+	+	+				
IVb.B48.EWP.26.CB	+	+	+				
IVb.B52.MB.27.PB	+	+	+				
IVb.B52.FM.29.CB	+	+	+	+	+		
IVb.B52.IWP.30.CB	+	+	+				
IVb.B52.EWP.28.CB	+	+	+	+	+		
IVc.B55.CEL.2.NB	+	+	+	+			
IVc.B55.IWP.3.PB	+	+	+	+			
IVc.B55.EWP.1.CB	+	+	+	+			
IVc.B62.MB.16.CB	+	+		+			
IVc.B62.FM.14.PB	+	+	+				
IVc.B62.IWP.13.CB	+	+		+			
Vb.B51.IWP.43.PB	+	+	+	+	+		
Vd.B54.IWP.39.NB	+	+	+	+			+

4.5.3. Fourier Transform Infrared Spectroscopic Analysis (FTIR)

The FTIR analysis of all binder (silt-clay) and aggregate parts of mudbrick materials shows that the main mineralogical composition of the samples consists of calcite, quartz and mica / illite composition.

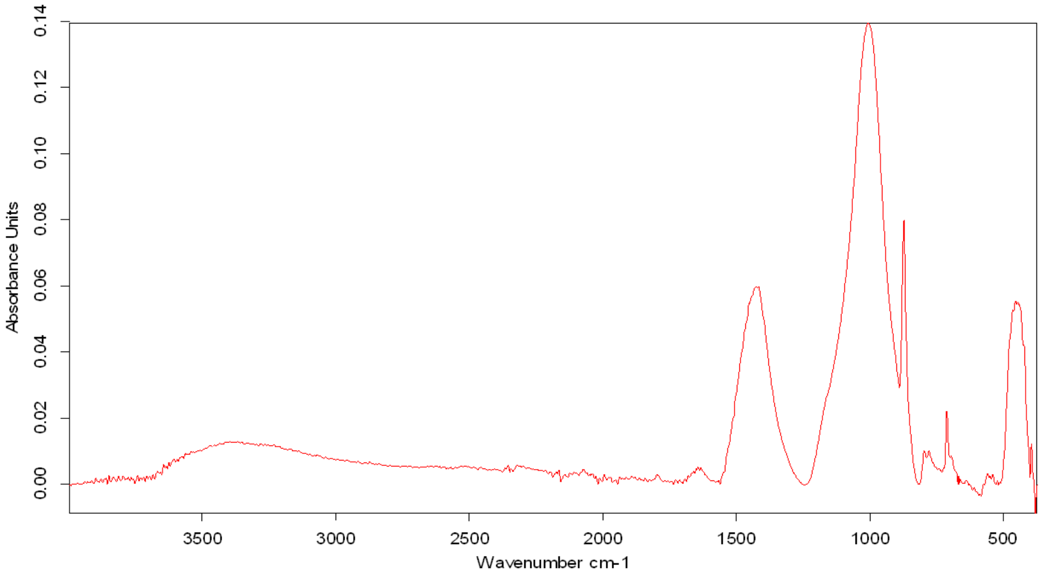


Figure 4.22. FTIR traces of the sample IVc.B55.EWP.1.CB

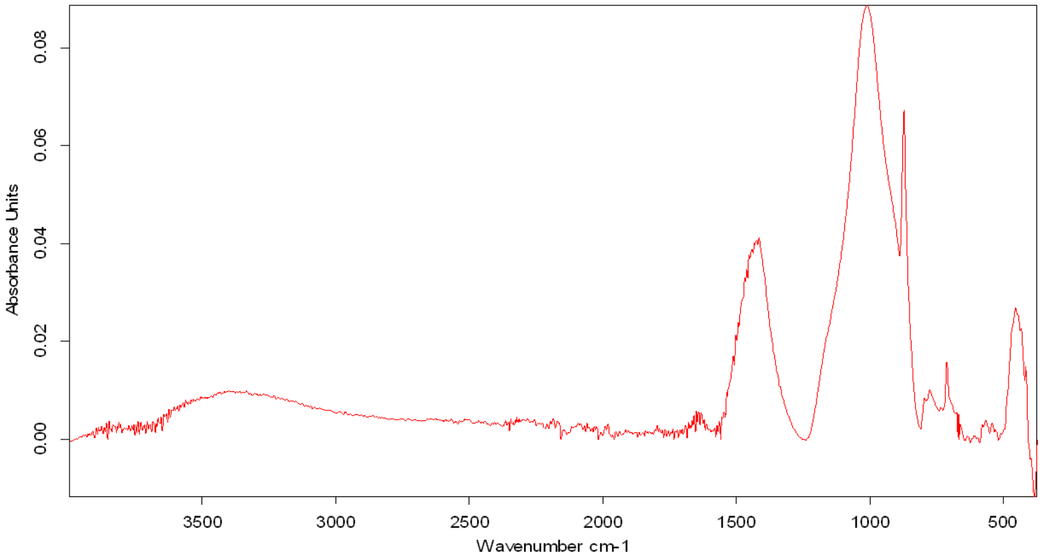


Figure 4.23. FTIR traces of the sample IVc.B62.IWP.13.CB

4.5.4. Image Analyses of Cross Sections

Cross section images of the samples were taken in 10x magnification by stereomicroscope. In cross section images the matrix of the samples could be analyzed in terms of their cracks, fiber pores and aggregate composition. These images could be evaluated with the thin section analyses.

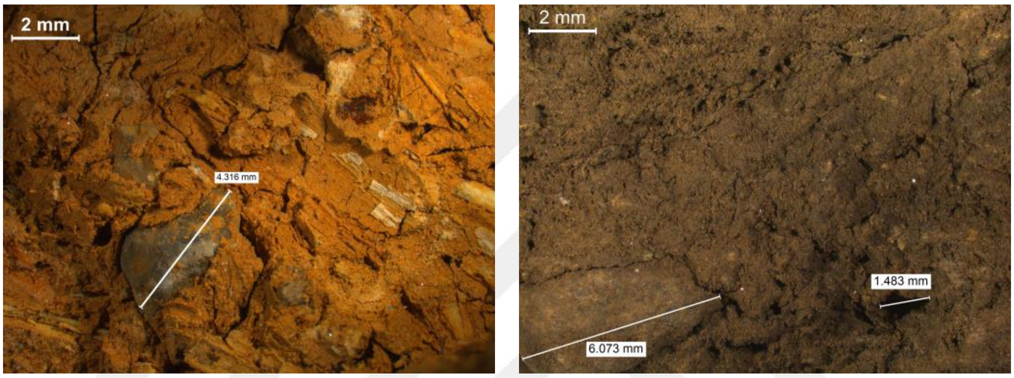


Figure 4.24. Cross section images at 10x by *stereomicroscope* of the sample IVc.B55.EWP.1.CB-left IVc.B55.FCM.2.NB -right

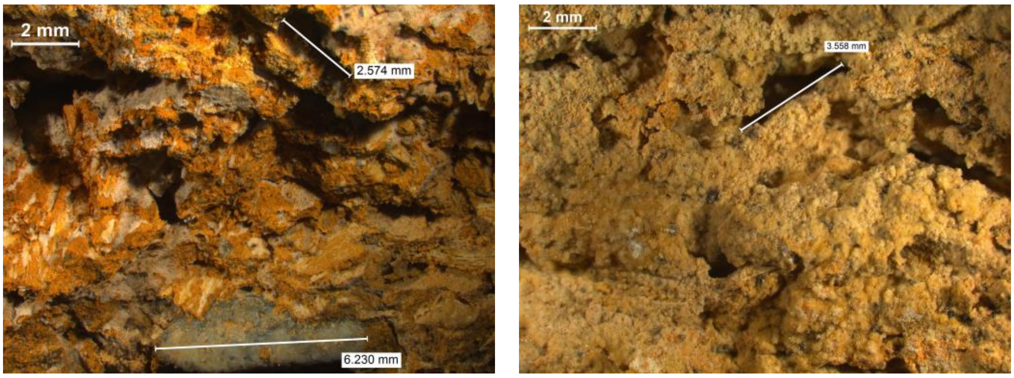


Figure 4.25. Cross section images at 10x by *stereomicroscope* of the sample IVc.B55.IWP.3.PB-left IVc.B62.IWP.13.CB-right

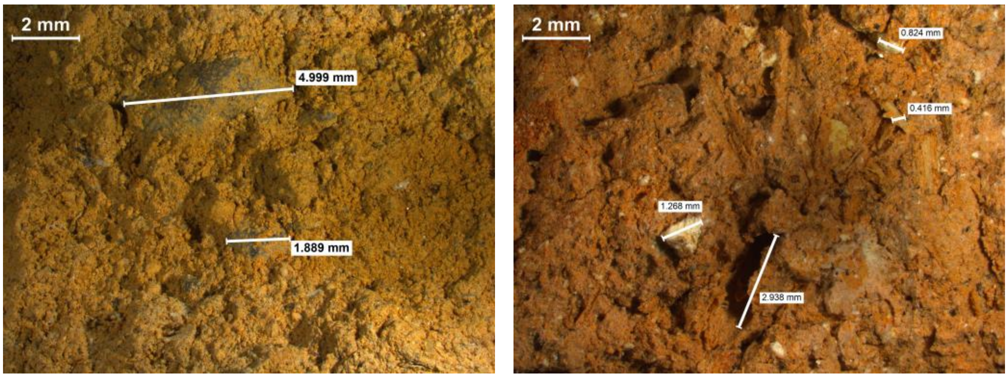


Figure 4.26. Cross section images at 10x by *stereomicroscope* of the sample IVc.B62.FCM.14.PB-left IVc.B62.MB.16.CB-right

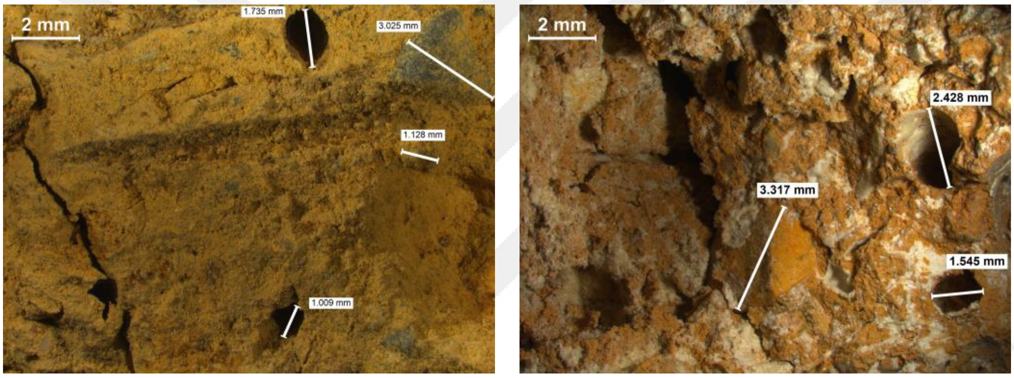


Figure 4.27. Cross section images at 10x by *stereomicroscope* of the sample IVb.B48.MB.17.CB-left IVb.B13.MB.19.CB-right

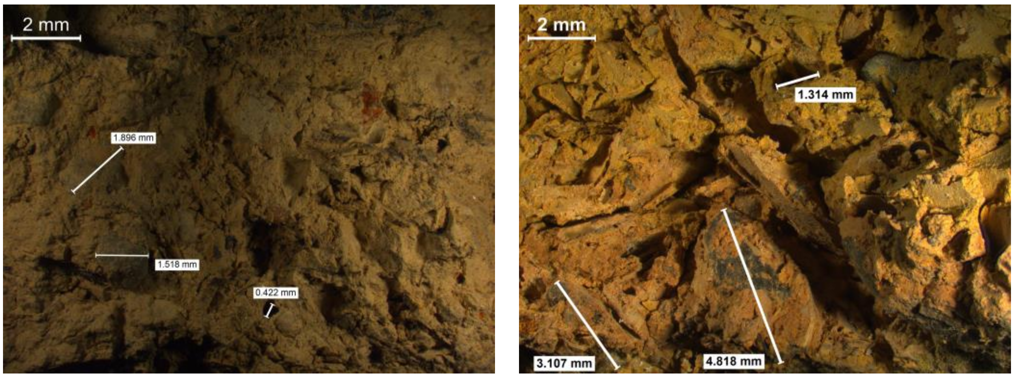


Figure 4.28. Cross section images at 10x by *stereomicroscope* of the sample IVb.B13.FCM.21A.NB-left IVb.B48.FCM.24.CB-right

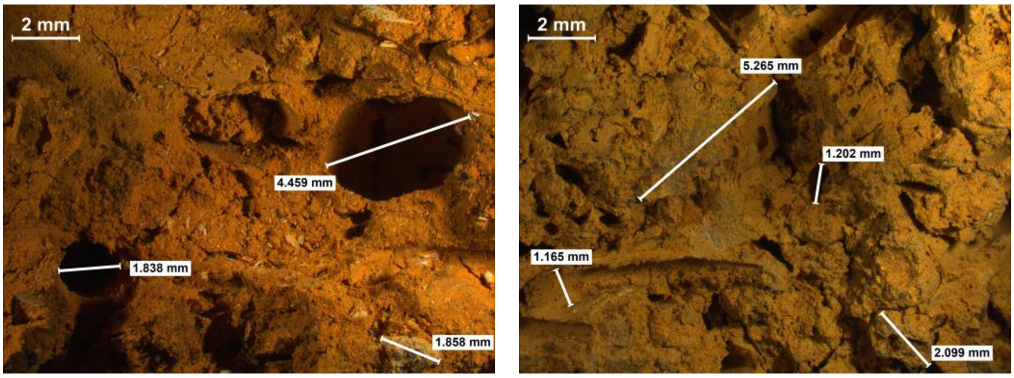


Figure 4.29. Cross section images at 10x by *stereomicroscope* of the sample IVb.B48.EWP.26.CB-left IVb.B52.MB.27.PB-right

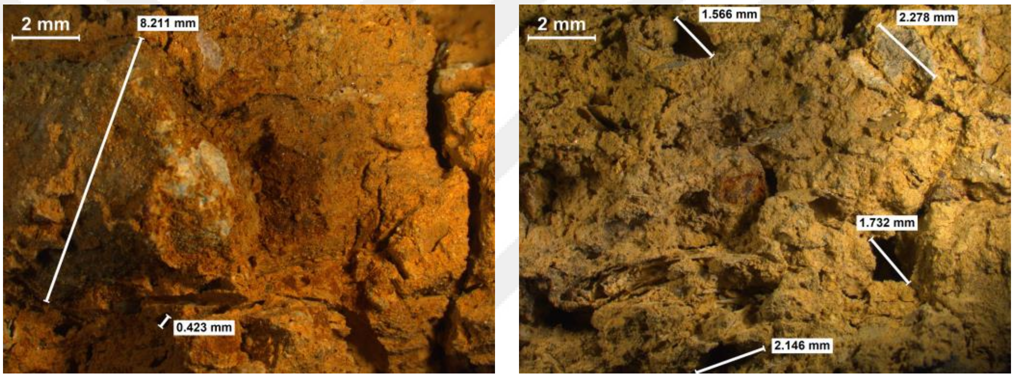


Figure 4.30. Cross section images at 10x by *stereomicroscope* of the sample IVb.B52.EWP.28.CB-left IVb.B52.FCM.29.CB-right

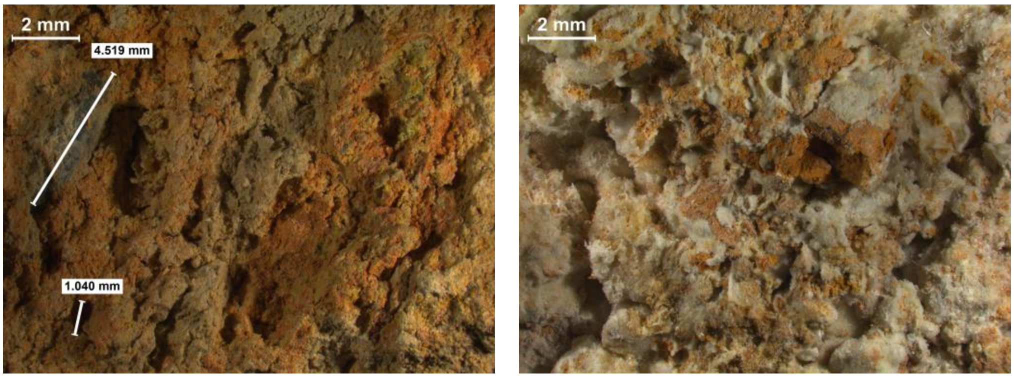


Figure 4.31. Cross section images at 10x by *stereomicroscope* of the sample IV.B65.FCM.34.CB-left IV.B65.MB.38.PB-right

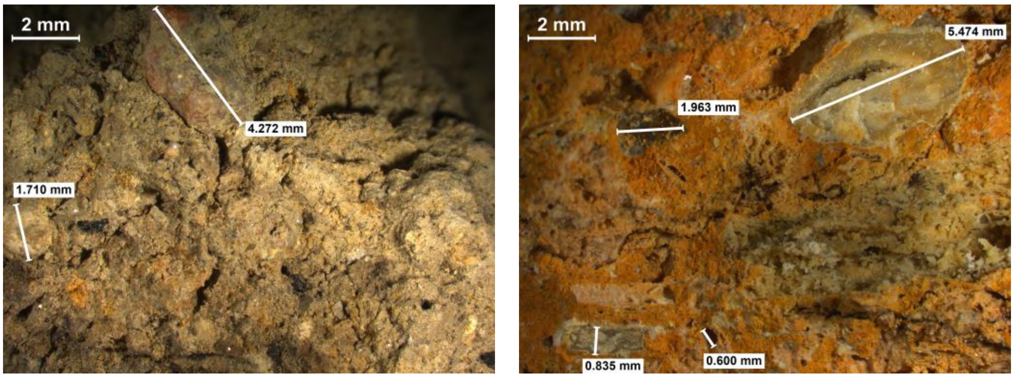


Figure 4.32. Cross section images at 10x by *stereomicroscope* of the sample Vd.B54.IWP.39.NB-left Vb.B51.IWP.43.PB-right

CHAPTER 5

DISCUSSION

The joint interpretation of the data achieved on performance, compositional and raw materials properties of mud brick, mud mortar and mud plaster samples belonging to four periods of Ulucak Höyük Neolithic settlement is done in order to:

- to better-understand their particular *compositional properties,*
- to define *indicators* specific to mud-based construction materials:
  - for the *assessment of their technological properties* and
  - for the *evaluation of results in relation to archaeological questions*.

The indicators introduced as measurable parameters are used:

- to discover the technological differences among mud-based construction materials, and
- to find out relationships between spatial, periodical and functional characteristics in the archaeological settlement.

5.1. Compositional Properties Assessment of Ulucak Höyük Neolithic Mud-Based Construction Materials

Raw material analyses were performed on seven unburnt and partially burnt mud samples. These samples are mainly three types of mud-based materials specifically:

- mudbrick as load-bearing masonry unit (IV.B65.MB.38.PB, IVb.B52.MB.27.PB),

- mud plaster applied to the interior surfaces of the exterior walls (IVc.B55.IWP.3.PB, Vd.B54.IWP.39.NB), and
- mud mortar as floor covering layer (IVb.B13.FCM.21A.NB, IVc.B55.FCM.2.NB, IVc.B62.FCM.14.PB).

The analyses on the compositional properties of mud-based construction materials, specifically their particle size distribution, silt-clay amount in their mud mixture and clay type as binder of the mixture, were conducted on seven unburnt samples and slightly-burnt mud samples. The petrographical analyses, specifically image analyses of thin section samples, were conducted on five burnt samples for raw materials identification and the data were evaluated in reference to the geological formations of the neighbouring district given in literature.

The results of particle size distribution analyses show that:

- The total silt-clay contents (x<63 micron) in the soil mixtures of mud-based materials vary in the range of 17% and 71% while the clay-sized $CaCO_3$-free portion of their silt-clay content are in the range of 11% and 49%. The silt-clay content of mudbrick (IV.B65.MB.38.PB, IVb.B52.MB.27.PB), floor covering mud mortar (IVb.B13.FCM.21A.NB, IVc.B55.FCM.2.NB) and interior mud plaster (Vd.B54.IWP.39.NB) are 21%, 25% and 49% in average, respectively (Figure xxx). Those ranges fall into the acceptable ranges of silt-clay amount to sustain the durable and compact form for mud materials (Jimenez Delgado, M. C. & Guerrero, I. C., 2007). One of the floor covering mortar (IVc.B62.FCM.14.PB) have similar particle size distribution with the mudbrick samples. This result may indicate that this sample could belong to a lower floor layer of the building (Figure 4.6. and Figure 4.7.).
- The very coarse aggregate (x>2mm) content in the mixtures of mudbrick, floor covering mud mortar and interior mud plaster are 25%, 11% and 6% in average, respectively (Figure 4.6. and Figure 4.7.). The presence of very coarse

aggregate has vital importance to provide volume stability and long-term durability of mud materials.

- Each type of mud-based materials, specifically mudbrick, floor covering mud mortar and interior mud plaster has different grain size distribution which result in different bulk densities (Figure 4.7.). That signals the conscious preparation of mud-based materials depending on their functions.

The results of XRD analyses of unburnt and slightly burnt samples showed that:

- The main binder of the Ulucak Höyük mud-based construction materials is mica-illite group clay (Table 5.1.).
- Some of the sample also have kaolinite group clay. The data indicate that the kaolinite is used in finishing materials such as floor covering mortars and interior wall plasters (Table 5.1.).

Table 5.1. The minerals determined by XRD analyses of the finest part of the seven unburnt and partially burnt samples (C: Calcite, Q: Quartz, M/I: Mica / Illite group, F: Feldspar, K: Kaolinite)

Sample Code	C	Q	M/I	F	K
IV.B65.MB.38.PB	+	+	+		
IVb.B13.FCM.21A.NB	+	+	+	+	+
IVb.B52.MB.27.PB	+	+	+		
IVc.B55.FCM.2.NB	+	+	+	+	+
IVc.B55.IWP.3.PB	+	+	+		+
IVc.B62.FCM.14.PB	+	+	+	+	
Vd.B54.IWP.39.NB	+	+	+		+

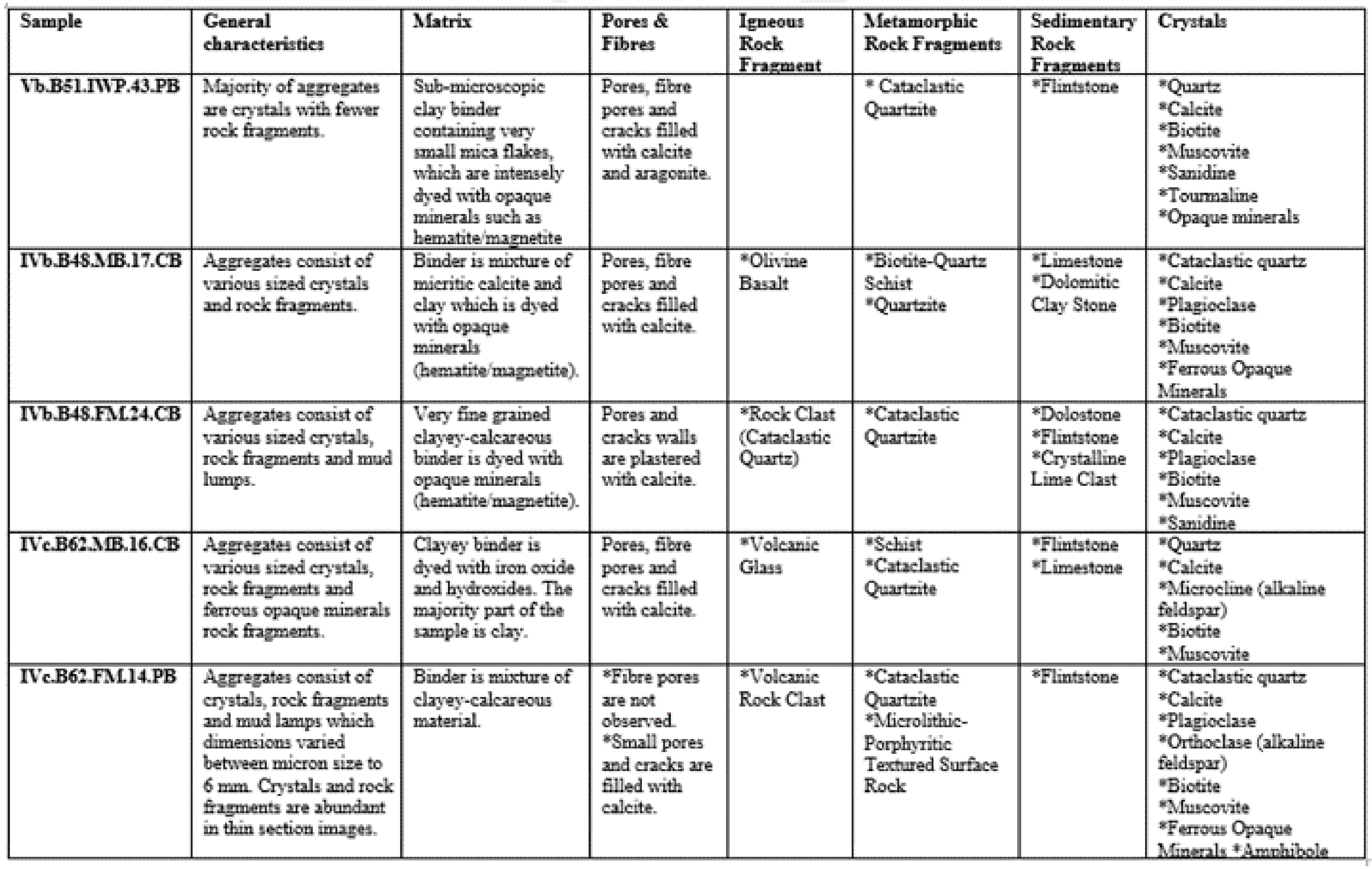
- There is high amount clay-sized $CaCO_3$ particles in the silt-clay mixture of the samples (Figure 4.4.).

- That qualified clay types have small swelling behaviour contributing to their long term durability of Neolithic house construction. These types of clays led to the formation of a plastic mixture which could be easily shaped by hand and traditional tools (Livingstoon, 1988). The presence of clay sized calcium carbonate particles in clay mixture has potential to stabilize the clay-silt mixture, therefore contribute to their dimensional stability (Ngowi, 1997).

The results of thin section analyses of five burnt samples showed that (Figure 5.1.):

- The texture of the samples is differentiated from each other in terms of the types of the aggregates, the size of aggregates, presence of fibres etc. (Figure 4.9. and Figure 4.10.).
- The crystal composition of the five samples mainly composed of quartz some of them being cataclastic quartz, calcite, muscovite, biotite, and plagioclase feldspar.
- Igneous (olivine basalt), metamorphic (quartzite, cataclastic quartzite, schist, microlithic porphyric surface rocks) and sedimentary (limestone, flintstone, dolomite claystone, dolostone, crystalline limestone) rock fragments were observed.
- Thin section images of the samples also showed some mud-lump fragments (Figure 4.16.-a) in the matrix, this may indicate that the usage of previous mud materials in the new mud materials preparation.

Mud-based materials preparation process includes the choice of qualified clay resources with certain amount, adding very coarse gravels to all samples, and calcite additives apart from the silt-clay sized presence. This process had also performed depending on function of mud-based construction material. Conscious material production shows the experience on technological knowledge achieved centuries and their continuation.



Sample	General characteristics	Matrix	Pores & Fibres	Igneous Rock Fragment	Metamorphic Rock Fragments	Sedimentary Rock Fragments	Crystals
Vb.B51.IWP.43.PB	Majority of aggregates are crystals with fewer rock fragments.	Sub-microscopic clay binder containing very small mica flakes, which are intensely dyed with opaque minerals such as hematite/magnetite	Pores, fibre pores and cracks filled with calcite and aragonite.		* Cataclastic Quartzite	*Flintstone	*Quartz *Calcite *Biotite *Muscovite *Sanidine *Tourmaline *Opaque minerals
IVb.B48.MB.17.CB	Aggregates consist of various sized crystals and rock fragments.	Binder is mixture of micritic calcite and clay which is dyed with opaque minerals (hematite/magnetite).	Pores, fibre pores and cracks filled with calcite.	*Olivine Basalt	*Biotite-Quartz Schist *Quartzite	*Limestone *Dolomitic Clay Stone	*Cataclastic quartz *Calcite *Plagioclase *Biotite *Muscovite *Ferrous Opaque Minerals
IVb.B48.FM.24.CB	Aggregates consist of various sized crystals, rock fragments and mud lumps.	Very fine grained clayey-calcareous binder is dyed with opaque minerals (hematite/magnetite).	Pores and cracks walls are plastered with calcite.	*Rock Clast (Cataclastic Quartz)	*Cataclastic Quartzite	*Dolostone *Flintstone *Crystalline Lime Clast	*Cataclastic quartz *Calcite *Plagioclase *Biotite *Muscovite *Sanidine
IVc.B62.MB.16.CB	Aggregates consist of various sized crystals, rock fragments and ferrous opaque minerals rock fragments.	Clayey binder is dyed with iron oxide and hydroxides. The majority part of the sample is clay.	Pores, fibre pores and cracks filled with calcite.	*Volcanic Glass	*Schist *Cataclastic Quartzite	*Flintstone *Limestone	*Quartz *Calcite *Microcline (alkaline feldspar) *Biotite *Muscovite
IVc.B62.FM.14.PB	Aggregates consist of crystals, rock fragments and mud lamps which dimensions varied between micron size to 6 mm. Crystals and rock fragments are abundant in thin section images.	Binder is mixture of clayey-calcareous material.	*Fibre pores are not observed. *Small pores and cracks are filled with calcite.	*Volcanic Rock Clast	*Cataclastic Quartzite *Microlithic-Porphyritic Textured Surface Rock	*Flintstone	*Cataclastic quartz *Calcite *Plagioclase *Orthoclase (alkaline feldspar) *Biotite *Muscovite *Ferrous Opaque Minerals *Amphibole

Figure 5.1. The summary of thin section analyses results in terms of matrix characteristics, igneous rock fragments, metamorphic rock fragments, sedimentary rock fragments, crystals, pores and fibers presence.

Considering all, the mudbricks, floor covering mortars and interior plasters belong to the Neolithic period of Ulucak Höyük archaeological site are qualified mud-based materials which exhibit the conscious use of natural adobe soil resource by the people of that region in Neolithic period and the advanced adobe technology achieved in Ulucak Höyük Neolithic Settlement.

5.2. Indicators Introduced as Measurable Parameters for Materials' Technological Properties Assessment

The indicators are determined by the certain experiments as measurable parameters to better understand the technological properties, preparation techniques and raw material resources of the mud-based materials of Ulucak Höyük Neolithic Settlement. The indicators which mainly determined for the unburnt or slightly burnt seven mud-based materials are described below:

- *Particle size distribution:* Particle size distribution is an indicator for technological identification of mud-based materials in terms of the raw materials composition and preparation of the mud-based materials by their function. This indicator is determined by the particle size distribution analysis including both sedimentation and sieving procedures (Teutenico, 1988). Ulucak Höyük mud-based materials have different aggregates distributions depending on the type/function of materials. In this study, *particle size distribution indicates that there are three types of mud mixtures for their specific use in the Ulucak Höyük buildings such as the mudbricks for the construction of the walls, mud plasters on the surface of the mudbrick walls and floor covering mortars* (Figure 4.7.). These mixtures were prepared by conscious use of natural adobe soil resource.
- *Percentage of silt-clay and very coarse aggregate:* These indicators are used to evaluate the composition properties and qualification of the mud-based

materials and they are determined by the particle size distribution analysis. The percentage of silt-clay and very coarse aggregates of Ulucak Höyük mud-based materials are different depending on the type/function of materials. The interior wall plaster sample (Vd.B54.IWP.39.NB) has the highest silt-clay portion with 49% while still having very coarse aggregates with %6. The floor covering mortar samples (IVb.B13.FCM.21A.NB, IVc.B55.FCM.2.NB) have around %25 silt-clay portion and very coarse aggregates with %10. The mudbrick samples (IV.B65.MB.38.PB, IVb.B52.MB.27.PB) have the lowest silt-clay portion around %21 and very coarse aggregates with %25 (Figure 4.6 and Figure 4.7.). *The percentage of silt-clay particles and very coarse aggregates for each type of mud-based samples are evaluated as a good composition for their positive contribution to the durability, porosity, and strength properties of the material* (Moropoulou, 2000).

- *Presence of clay-sized calcium carbonate ($CaCO_3$):* Clay-sized $CaCO_3$ is an indicator for the identification of raw material resources and qualification of the mud-based construction materials. The presence of this indicator is determined by the XRD traces of the silt-clay portion and its amount is determined by the loss on ignition experiment (at 900℃). The mud-based materials of Ulucak Höyük have clay-sized calcium carbonate in the silt-clay mixtures in the range of 21% - 54.8% (Figure 4.4.). *The ratio of clay-sized $CaCO_3$ varying in the silt-clay mixture may indicate the use of various adobe resources or the changes in layers of soil resources occurred in time in the same resources.*
- *Clay type (mica-illit and/or kaolinite):* Clay type is an indicator for the raw materials sources and determined by the XRD traces. The main clay type of the Ulucak Höyük mud based construction materials is mica-illite group clay. The finishing materials such as floor covering mortar and interior mud plaster also have kaolinite group clay. *This result indicates that there are two different clay sources that used in Ulucak Höyük buildings. The finishing mud materials such as floor covering mortar and interior plaster have mica-illite and*

kaolinite group clay, while the mudbrick samples have only mica-illite group clay.

- *Bulk density:* Bulk density is an indicator for the technological identification of materials in terms of the physical properties. Bulk density data of the Ulucak Höyük mud based construction materials indicate that mudbrick, floor covering mortar and interior wall plaster materials have different bulk densities. These values are also in coherence with the grain size distribution of these mud-based samples (Figure 5.2.).

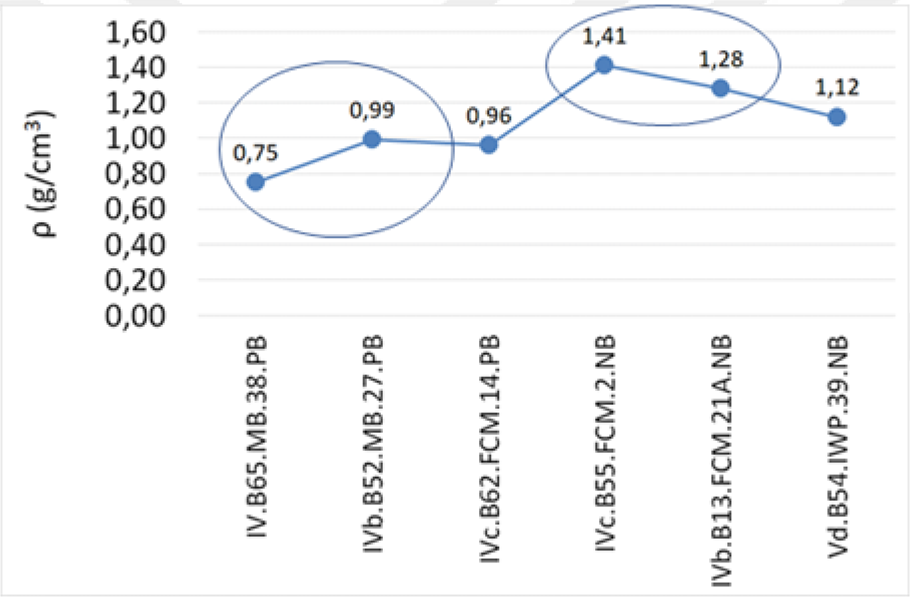


Figure 5.2. The bulk density values of the unburnt and partially burnt samples.

5.3. Evaluation of Technological Properties Data on Mud Construction Materials in Terms of Archaeological Questions

The combined interpretation of all data on mud-based construction materials belonging to Ulucak Höyük Neolithic Period exhibited the materials characteristics of mud brick, floor covering mud mortars and interior mud plasters. Their properties are described briefly below:

- The mudbrick samples (IV.B65.MB.38.PB and IVb.B52.MB.27.PB) belonging to the IVth level of Neolithic period have mica-illite type of clay as a binder without kaolinite. The average percentage of silt-clay aggregate is 26% by weight with 3/4th of the aggregate is clay content and 1/4th of the aggregate is clay-sized $CaCO_3$ (Table 5.2.). The average very coarse aggregate (x>2mm) is 25%. Their average bulk density is 0.87 g/cm^3.
- The floor covering mud mortar samples (IVb.B13.FCM.21A.NB, IVc.B55.FCM.2.NB) belonging to IVb and IVc sublayers have mica-illite and kaolinite mixed clay type as a binder. The average percentage of silt-clay aggregate is 47% by weight, for the sample IVc.B55.FCM.2.NB 2/3rd of the aggregate is clay content and 1/3rd of the aggregate is clay-sized $CaCO_3$ and for the sample IVb.B13.FCM.21A.NB 1/2nd of the aggregate is clay content and 1/2nd of the aggregate is clay-sized $CaCO_3$ (Table 5.2.). The average very coarse aggregate (x>2mm) is 11%. Their average bulk density is 1.35 g/cm^3.
- The floor covering mud mortar sample (IVc.B62.FCM.14.PB) differs from the other floor covering mud mortars in terms of its grain size distribution, bulk density, clay type and clay content. This sample has similar compositional properties with the mudbricks. The sample has mica-illite type of clay as a binder without kaolinite. The percentage of silt-clay aggregate is 17% by weight with 2/3rd of the aggregate is clay content and 1/3rd of the aggregate is clay-sized $CaCO_3$ (Table 5.2.). The very coarse aggregate (x>2mm) is 26%. Its bulk density is 0.96 g/cm^3.

- The interior mud wall plaster sample (Vd.B54.IWP.39.NB) belonging to Vd sublayer have mica-illite and kaolinite mixed clay type as a binder. The percentage of silt-clay aggregate is 71% by weight with 2/3rd of the aggregate is clay content and 1/3rd of the aggregate is clay-sized $CaCO_3$ (Table 5.2.). The average very coarse aggregate (x>2mm) is 6%. Its bulk density is 1.12 g/cm^3.

Table 5.2. Clay type and the portion of silt-clay and clay-sized $CaCO_3$

	Portion of silt-clay in the silt-clay mixture	Portion of clay-sized $CaCO_3$ in the silt-clay mixture	Clay type
Mudbrick IV.B65.MB.38.PB	3/4	1/4	Mica-illite
Mudbrick IVb.B52.MB.27.PB	3/4	1/4	Mica-illite
Floor covering mortar IVc.B55.FCM.2.NB	2/3	1/3	Mica-illite and kaolinite
Floor covering mortar IVb.B13.FCM.21A.NB	1/2	1/2	Mica-illite and kaolinite
Floor covering mortar IVc.B62.FCM.14.PB	2/3	1/3	Mica-illite
Interior wall plaster Vd.B54.IWP.39.NB	2/3	1/3	Mica-illite and kaolinite

- *The joint interpretation of the clay type and the portion of clay-sized $CaCO_3$ in silt-clay mixture show that two different clay sources were used in Ulucak Höyük mud-based construction materials preparation.* One of the source have mica-illite type clay and the other source have mica-illite with kaolinite group clay. *The differences in the portion of $CaCO_3$ is due to the different layers of the source used at various periods since deeper layers could be reached as the source was used. The source preferences change in terms of the function of mud-based materials, the mudbrick samples have mica-illite group clay and the finishing materials such as floor covering mortar and interior wall plaster have mica-illite group clay with kaolinite.*
- The raw materials and technological properties of the unburnt and slightly burnt mudbrick, floor covering mud mortar and interior wall mud plaster

samples of the Ulucak Höyük Neolithic settlement are described specifically. *In case of the conservation studies, new mud-based materials can be prepared by using the similar raw materials and compositional properties as described. In addition, salt analysis show that the salt amount is negligible therefore the samples are not suffering from the salt problem* (Table 4.7.). *For this reason, the raw materials which could be used in the conservation studies should not contain high amount of salt.*

- The burnt mud-based construction materials of the Ulucak Höyük have a high pozzolanic activity values that can be due to the presence of kaolinite. (Table 4.6. and Figure 4.8.). If the kaolinite group clay fire at around 600-650ºC up to 900°C, it gains high pozzolanic activity (Baronio and Binda, 1996). Besides this, thin section analyses of the samples indicate that the presence of the volcanic rock fragment, which is the natural pozzolanic aggregates. The high pozzolanic activity properties due to the firing of kaolinites and the presence of volcanic rock fragments. *In case of the conservation studies, those burnt samples can be treated with the pozzolanic lime mortar and colloidal calcium hydroxide solution.*

CHAPTER 6

CONCLUSION

Ulucak Höyük is one of the important Neolithic settlement of the Aegean Turkey with eight meters of Neolithic deposits represents a thousand years. Ulucak Höyük has well-preserved mudbrick remains that provide comprehensive investigation on the architectural structure of each level, the building material, and the properties of these materials.

This research is performed to reveal the compositional and technological properties, and raw material properties of the Ulucak Höyük Neolithic settlement mud-based construction materials. These properties were evaluated in terms of the similarities/differences with the buildings, periods and materials function to better understand the technological knowledge and social organization of that period.

The samples collected from eight different houses that represent the Vth, and IVth levels of the settlement and the maximum time difference between the samples is 700 years. Twenty mud-based samples mainly mudbricks as a load-bearing materials, floor covering mud mortars and interior/exterior wall mud plasters were investigated in the study. Due to the samples were affected heavy fire, thirteen samples were not evaluated comprehensively. Only partially burnt and not burnt seven samples were investigated in detail.

In the study, the main indicators were obtained from seven unburnt and partially burnt samples. These indicators are measurable parameters for better understand the technological properties, preparation techniques and raw material resources of the mud-based materials in Ulucak Höyük Neolithic Settlement. These indicators are particle size distribution, percentage of silt-clay, percentage of very coarse aggregates, presence of clay-sized $CaCO_3$ in silt-clay mixture, clay type and bulk density.

- Particle size distribution of the Ulucak Höyük mud-based samples show that three types of the construction material such as mudbrick, floor covering mortar and interior wall plaster have their specific aggregate distribution. The mudbrick samples have approximately 26% silt-clay size particles and 25% very coarse aggregate. The floor covering mortar samples have approximately 47% silt-clay size particles and 11% very coarse aggregate. The interior wall plaster sample have 71% silt-clay size particles and 6% very coarse aggregate. This result show that the mud-based construction materials of the Ulucak Höyük were prepared by different techniques in terms of their function. Conscious material production shows the experience on technological knowledge achieved centuries and their continuation.
- The percentage of silt-clay content (without clay-sized $CaCO_3$) of mudbricks, floor covering mud mortars and interior mud plaster are 21%, 25% and 49% in average, respectively. These silt-clay ratios are proper ranges to sustain the durable and compact form for mud materials. The presence of very coarse aggregates in all type of mud samples is provide a good composition for the durability, porosity, and strength properties of the materials.
- The mud-based materials of Ulucak Höyük have clay-sized calcium carbonate in the silt-clay mixtures in the range of 21% - 54.8%. The high ratio of clay-sized calcium carbonate is due to the geological formation of the Ulucak region. The presence of clay-sized calcium carbonate particles in silt-clay mixture has potential to stabilize the mud-based materials, therefore contribute to their dimensional stability.
- The main clay type of the Ulucak Höyük Neolithic mud-based materials is mica-illite with or without kaolinite. These clay types are qualified in terms of having small swelling behavior contributing to their long term durability. In

addition, because of the plasticity properties of these clay types the mud materials could be easily shaped.

- The bulk density values of the unburnt and partially burnt mud samples represent their physical properties. The approximate bulk density values of the mudbricks, floor covering mortars and interior wall plaster are 0.87, 1.35 and 1.12 g/cm^3, respectively. These three types of construction materials have their specific bulk density values.

These indicators were determined with the unburnt samples and the useful experiments are particle size distribution, XRD analysis, loss on ignition experiment and bulk density analyses.

The indicators showed that results:

- The mudbricks, floor covering mortars and interior plasters belong to the Neolithic period of Ulucak Höyük archaeological site are qualified mud based materials in terms of:

    having mica-illite clay type,
    presence of clay-sized $CaCO_3$
    presence of very coarse aggregates

- The qualified raw material preferences and preparation techniques (particle size distribution & bulk density) of the mud-based materials depending on their function exhibit the conscious use of natural adobe soil resource of that region in Neolithic period and the advanced adobe technology.
- There were two different clay sources, one of the source had only mica-illite group clay which was used for mudbrick preparation, the other source had

mica-illite and kaolinite group clay which was used for finishing elements such as floor covering mortar and interior wall plaster.

- The portion of clay-sized $CaCO_3$ in the mud-based materials belonging to IVb, IVc and Vd sublayers are different. This is due to the presence of two different clay sources and period differences because the different layers of the sources were used in time.

For the conservation studies:

- The definitions of the compositional and technological properties of the mud-based construction materials of Ulucak Höyük were determined for the mudbrick, floor covering mortar and interior wall plaster by the help of the indicators. In case of the conservation studies, new mud-based construction materials could be prepared with these definitions.
- The Ulucak Höyük mud-based samples have very little salt amount (between 0.16% - 0.29%), so the new conservation materials should not have high amount of salt because presence of salt in the construction materials cause the material loss in time.
- The burnt mud samples of the Ulucak Höyük have high pozzolanic activity values which due to the presence of kaolinite and volcanic rock fragments. These samples can be treated with the pozzolanic lime mortar and colloidal calcium hydroxide solution in case of the conservation studies.

The mud-based specimens of Ulucak Höyük were affected to various degrees by fires occurring in the Neolithic period, and their physical and compositional properties were also changed. The bulk density and porosity values of the burnt mud-based samples indicate that the mudbrick, floor covering mortar and interior/exterior wall plaster samples have porous and breathable characteristic.

The porous structure, presence of fibers and coarse aggregates of the mud-based samples is visible in cross section images of the samples.

Thin section analyses were performed on five burnt samples and their results show that the raw material of the Ulucak Höyük mud-based materials are compatible with the geological structure of the region (Kayan, 1999). The crystal composition of the five samples mainly composed of quartz some of them being cataclastic quartz, calcite, muscovite, biotite, and plagioclase feldspar. Igneous (olivine basalt), metamorphic (quartzite, cataclastic quartzite, schist, microlithic porphyric surface rocks) and sedimentary (limestone, flintstone, dolomite claystone, dolostone, crystalline limestone) rock fragments were observed.

Further researches related with the study can be suggested as follows:

- In this study only seven unburnt samples provide a comprehensive result for the properties of the mud-based samples. More unburnt mudbrick, floor covering mortar and wall plaster samples which represent the different buildings and layers of the Ulucak Höyük Neolithic settlement could be studied in terms of the particle size distribution, percentage of silt-clay and coarse aggregates, portion of clay-sized $CaCO_3$, clay type and bulk density values to better understand the compositional, technological properties and raw material resources of the construction materials.
- The different clay source (including kaolinite) preferences of finishing construction materials could be studied in detail.
- For the conservation studies, firstly visual analyses should be done, and then the deterioration rate of the site can be determined by non-destructive analyses (IR termography, UPV).
- The usage of high amount $CaCO_3$ both in clay-sized and additive aggregates can be studied in term of the hygienic and decorative functions (Clarke, 2012).

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