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## **A physicochemical study of Medieval and Post-Medieval ceramics from the Aegean**

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## **CHAPTER 9 X-RAY POWDER DIFFRACTION**

### **9. INTRODUCTION**

Several analytical techniques are used to characterize the composition of the materials. One of these is X-ray powder Diffraction (XRD), which is widely used for the determination and identification of crystalline phases that compose various materials. This method is considered a useful tool for the ceramic analysis since it provides significant information both for their composition and the firing temperature of the ceramic materials. X-ray Diffraction (XRD) provided qualitative analysis of the chemical compounds through the study of the ceramics crystalline structure. XRD was used for the characterization of the mineral content of ceramic materials and to investigate inclusions, as well as the matrix. Information regarding the raw materials and the firing technology were studied from the mineralogical transformations (Gliozzo 2020; Grammatikakis 2019; Ouahabi 2015; Trindade et al 2009; Lee et al. 2008; Hajjaji 2004; Rodriguez 2003; Cultrone 2001; Dondi 1999; Hillier 1999; Duminuco 1998; Gonzelez-Garcia et al. 1990; Chen 1977).

During the present study, 90 bulk samples of the 245 samples were analyzed using a SIEMENS X-ray diffractometer of the National Centre for Scientific Research 'NCSR Demokritos'. The determination of the new mineralogical phases, as well as the primary and secondary minerals, is performed through the evaluation of X-ray powder diffractograms. The obtained results were compared to the ones revealed through the petrographic study (OM) and the microtextural analysis (SEM). The identification of neo-formed minerals is significant enough, because their chemical composition and their formation conditions provide information on:

- The chemical composition of the raw materials used
- The atmosphere prevailing in the furnace (oxidized / reducing conditions)
- The equivalent firing temperature

### **9.1 X-RAY POWDER DIFFRACTOMETRY (XPRD)**

Diffraction effects are observed when electromagnetic radiation impinges on period instructors with geometrical variations on the length scale of the wavelength of the radiation. The distances between atoms in crystals and molecules amount to 0.15–0.4 nm which correspond to the electromagnetic spectrum with the wavelength of x-rays having photon energies between 3 and 8 keV (Cowley 1993; Janssens 2004). X-rays have high energies and short wavelengths on the order of the atomic spacing for solids. When a beam of x-rays falls against on a solid material, a portion of this beam will be scattered in all directions by the electrons associated with each atom or ion hit upon (Cullity 1956; Callister 2007). One common diffraction technique employs a powdered or polycrystalline specimen consisting of many fine and randomly oriented particles that are exposed to monochromatic x-radiation. Each powder particle (or grain) is a crystal, and has a large number of them with random orientations which ensures that some particles are properly oriented in such a way that every possible set of crystallographic planes will be available for diffraction (Burhke et al. 1997). Constructive interference of the rays occurs at an angle  $\theta$  if the path length difference is

equal to a whole number of wavelengths,  $n\lambda$ , where  $n$  is an integer and  $\lambda$  is the wavelength. The fundamental relationship in XRD is the Bragg equation:

$$n\lambda = 2d \sin\theta$$

where  $d$  is the distance between the layers of atoms (Janssens 2004; Callister 2007).

The firing temperature and the firing conditions in the interior of the kiln can be estimated by means of the mineral reactions. The mineralogical assemblages of the clay paste can be revealed through X-Ray Powder Diffraction. Archaeologically discovered ceramics contain minerals which may be classified on the basis of their origin in the following categories:

- **Primary minerals:** mineralogical phases which pre-existed in sediments rich in clay and which proved resilient enough to sustain the firing conditions and to survive (e.g. quartz or feldspars). The classification of these minerals is that of clastic grains when participating in clay sediment naturally. Some of these inclusions exhibit features demonstrating that they were added intentionally in the original clay paste by potter in antiquity, who wanted thereby to improve the technical characteristics of the mass; in some cases, their intention was simply to perform a standard and somewhat “ritual” procedure, usually referred to as tempering.
- **‘Firing’ minerals:** the neo-formed phases which are crystallized after the primary minerals react with the clay paste, during the increasing firing temperature conditions under which the primary mineralogical phases become unstable. According to the synthesis of the raw materials, the firing minerals could comprise diopside, gehlenite, anorthite, wollastonite, enstatite, mullite, hematite, magnetite, spinel and many others. The glassy phase is included in this category.
- **Secondary minerals:** they formed after firing and particularly during the time of the burial of the ceramic or their interaction with the atmospheric chemical elements (e.g. calcite, hydrogrossular, zeolites). Their crystallization depends mainly on the degree of dampness/dryness soil; pH and chemical composition of the solutions penetrate the ceramic (Xanthopoulou 2019).

Except for the minerals present as inclusions in the clay paste of ceramics, the contribution of the clay minerals which constitute the clay paste is extremely significant, since the type of the clay minerals and their quantity imparts to the clay paste different properties and consequently affect and many times settle the typology of the end ceramic product. The determination of clay minerals through X-ray Diffraction needs a pretreatment of the raw material in which the clay minerals are separated by extracting a fine enough particle-size fraction ( $<2 \mu\text{m}$ ), minimizing the non-clay impurities.

During the present study, 90 bulk samples were analyzed using a SIEMENS X-ray diffractometer of the National Centre for Scientific Research ‘NCSR Demokritos’. The diffractometer is equipped with Cu-K $\alpha$  radiation and holds a system of single-chroming/ monochromatic application of a secondary beam of pyrolytic graphite operating at 40kV and 35mA. The system of fractures/slashes in the course of the initial and the diffracted beam  $1.0^\circ$ . Bulk samples were scanned from  $3$  to  $60^\circ 2\theta$ , with scanning angle step of  $0.03/2\theta$  and a step time of  $0.1\text{s}$  for  $1\text{h}$  and  $30\text{ min}$ . Qualitative analysis was performed using the DIFFRACplus EVA software (Bruker-AXS, USA) based on the ICDD Powder Diffraction File (2006 version).

## 9.2 RESULTS AND DISCUSSION OF XPRD

When the significant data of the XRD spectroscopy were received and compared to the different archaeological regions as well as the chronological periods, many conclusions were extracted.

**Champlevé Ware** were constructed only in **Chalcis in Euboea**. They consisted of Quartz, Plagioclase, K-Feldspars and Diopside. Also, the presence of Hematite was observed apart from one sample (CH148). Also, small amount of Muscovite/Illite was detected at samples CH84, CH92, CH124 (Figure 133, Tables 29,32). This pottery type was fired at a high temperature  $T > 950$  °C due to presence of Diopside, during the Middle Byzantine to Late Byzantine/Frankish periods (Table 35).

Fragments of **Incised Sgraffito Ware** from **the Athenian Agora in Attica** contained Quartz, Plagioclase, K-Feldspars and Hematite as well as sample AGBZY841 Diopside, Gehlenite; and sample AGBZY846 Cristobalite. They were fired at a high Temperature  $T > 950-1000$  °C due to detection of Diopside, Gehlenite and Cristobalite. In **Chalcis in Euboea**, two different firing groups were found. The first one consisted of Quartz, Plagioclase, K-Feldspars, Muscovite/Illite and Hematite (apart from CH150). Second group consisted of two other samples and specifically of CH133 with Cristobalite and CH153 with Diopside, except for Quartz, Plagioclase, K-Feldspars and Hematite that they were also detected (Figure 134, Tables 29,30,32). The first group was fired at a lower temperature than samples of the Athenian Agora in Attica, because Muscovite/Illite was observed. The second one was fired at a higher temperature due to the presence of Cristobalite and Diopside. It seems that the potters of the Athenian Agora in Attica used only a high temperature for this pottery type during the Middle Byzantine/Frankish period. Opposed to this, potters of Chalcis used a lower temperature about  $T$  800-950 °C for this typology during the Middle Byzantine period and a higher  $T > 950-1000$  °C (CH133, CH153) during the Late Byzantine/Frankish period (Tables 35,36).

All the three types of **Amphorae** fragments (A8A, Günsenin 2, Günsenin 3) from **Chalcis in Euboea** were composed by Quartz, Plagioclase, K-Feldspars and Hematite. They contained also Muscovite/Illite apart from two samples of Günsenin 3 Amphorae (CH158, CH160). Günsenin 3 Amphora (CH160) and Günsenin 2 Amphora (CH163) also consisted of Diopside as well as Günsenin 3 Amphora (CH159) of Cristobalite (Figure 135, Tables 29,32). At A8A Amphora, potters used a lower temperature  $T$  800-900 °C, at Günsenin 2 Amphorae a few higher temperature  $T > 900$  °C, and at Günsenin 3 Amphorae a quite higher temperature about  $T > 950-1000$  °C. At A8A Amphora, a significant amount of Muscovite/Illite was detected in contrast to the less amount of this mineral at Günsenin 2 Amphorae. Finally, the higher temperature of Günsenin 3 Amphorae was evidenced by the presence of Diopside and Cristobalite (Table 35). Amphorae (A8A, Günsenin 2, Günsenin 3) were constructed in Chalcis in Euboea during the Middle Byzantine/Frankish period.

**Splashed Ware** sample (CH89) from **Chalcis in Euboea** was composed of Quartz, Plagioclase, Calcite and Muscovite/Illite. But, Splashed Ware sample CH145 was made up of Quartz, Plagioclase, K-Feldspars, and Cristobalite (Figure 136, Tables 29,32). Sample CH89 was fired at a lower temperature about  $T$  800-950 °C due to the presence of Muscovite/Illite than sample

CH145 with higher temperature  $T > 1000$  °C in which Cristobalite was detected. This typology was constructed during the Late Byzantine/Frankish period in Chalcis (Table 35).

**Plain Glazed Ware** sample (CH88) from **Chalcis in Euboea** contained Quartz, Plagioclase, Hematite and Muscovite/Illite. But, sample CH125 contained Quartz, Plagioclase, Hematite, Diopside and Cristobalite (Figure 137, Tables 29,33). At sample CH88, Muscovite/Illite was observed and this means that was fired at low temperature about  $T$  800-950 °C. But, sample CH125 consisted of Diopside, Cristobalite and this means that was fired at higher temperature  $T > 1000$  °C. This pottery type was manufactured in Chalcis during the Early Byzantine to Middle Byzantine periods (Table 35).

Regarding **Slip-Painted Ware**, both samples from **the Athenian Agora in Attica** (AAG59) and from **Chalcis in Euboea** (CH94, CH138) contained Quartz, K-Feldspars, Plagioclase, Hematite (apart from CH138) and Muscovite/Illite. In contrast to them, sample CH121 from Chalcis contained Quartz, Plagioclase, K-Feldspars, Hematite, Diopside and Cristobalite (Figure 138, Tables 29,30,32). These ceramics from the Athenian Agora in Attica and from Chalcis in Euboea mainly were fired at a low temperature about  $T$  800-950 °C due to the presence of Muscovite/Illite. Only one sample from Chalcis (CH121) contained Diopside and Cristobalite and the temperature was about  $T > 1000$  °C. Slip-Painted Ware from Chalcis were manufactured during the Middle Byzantine period and these from the Athenian Agora in Attica during the Late Byzantine/Frankish period (Tables 35,36).

At fragments of **Zeuxippus Ware Subtype** from **the Athenian Agora in Attica**, Quartz, Plagioclase, K-Feldspars (apart from AAG47) and Hematite were found. Muscovite/Illite also existed at samples AAG43, AAG50 and Calcite at samples AAG47, AAG50. Furthermore, in sample AAG50 was detected Hallite. Imported **Zeuxippus Ware** (CH101) from **Chalcis in Euboea** contained Quartz, Plagioclase, Hematite, Diopside and Cristobalite (Figure 139, Tables 29,30,32). Regarding fragments of Zeuxippus Ware Subtype from the Athenian Agora in Attica, all of them were fired at a low temperature  $T$  800-950 °C because Muscovite/Illite was detected. But, at Zeuxippus Ware in Chalcis, that came from Western Turkey, Diopside and Cristobalite were found. Imported sample was fired at higher temperature  $T > 1000$  °C than the lower temperature  $T$  800-950 °C of the Subtype. Both sub Zeuxippus Ware from the Athenian Agora and Zeuxippus Ware from Western Turkey were manufactured during the Late Byzantine/Frankish period (Tables 35,36).

At fragments of **Polychrome Painted Ware/Maiolica** from **the Athenian Agora in Attica**, Quartz, Plagioclase, Diopside and Gehlenite were found. Two samples AAG29, AAG66 also consisted of Hematite and sample AAG66 of Muscovite/Illite (Figure 140, Tables 30,33). They were fired at a high temperature  $T > 950$  °C due to the presence of Diopside and Gehlenite during the Turkish/Venetian period (Table 36).

In imported **Maiolica** fragment (AAG70), that was found in **the Athenian Agora in Attica**, Quartz, Plagioclase, Hematite, Diopside and Gehlenite were observed. Imported Maiolica fragments that were found in **Chalcis in Euboea** (CH154) and in **the Castle of Mytilene in Lesbos** (MYT227), were made up of the same minerals apart from the presence of Hematite (Figure 140,

Tables 29,30,31,33). At imported Maiolica fragments from these three studied areas, Diopside and Gehlenite were detected and that means that they were fired at a high temperature  $T > 950$  °C during the Turkish/Venetian period (Tables 35,36,37).

At fragments of **Polychrome Sgraffito Ware** from **the Athenian Agora in Attica** (AAG37, AAG60, AAG62), Quartz, Plagioclase and Diopside were found. At AAG60 was observed also K-Feldspars, Gehlenite and Cristobalite; at AAG62 Hematite; and at AAG37 Cristobalite. Polychrome Sgraffito Ware samples from **the Castle of Mytilene in Lesvos** consisted of Quartz, Plagioclase and K-Feldspars (apart from MYT209) and Hematite (apart from MYT217). Sample MYT217 had also Diopside, Gehlenite and Cristobalite; sample MYT209 Diopside and Gehlenite; and sample MYT194 Sanidine (Figure 141, Tables 30,31,33). Polychrome Sgraffito Ware from the Athenian Agora and from the Castle of Mytilene were fired at a high temperature  $T > 950-1000$  °C as Diopside, Gehlenite or Cristobalite were detected during the Turkish/Venetian period (Tables 36,37).

**Monochrome Glazed Ware** fragments that were analysed came from **the Athenian Agora in Attica** and **the Castle of Mytilene in Lesvos**. Samples from the Athenian Agora contained Quartz, Plagioclase (apart from AAG26), K-Feldspars (only at AAG26, AAG54) and Hematite (apart from AAG53, AAG54, AAG71). Most of them had Diopside (apart from AAG54) and Gehlenite (at AAG20, AAG26) or Cristobalite (at AAG71). Only at one sample (AAG54), Muscovite/Illite was detected. In contrast to the Athenian Agora, samples from the Castle of Mytilene consisted of Quartz, Plagioclase (apart from MYT231) and also K-Feldspars and Hematite that were detected only at two samples (MYT199, MYT213). Furthermore, MYT213 consisted of Quartz, Plagioclase, Muscovite/Illite, Hematite and Analcime. Finally, one sample (MYT206) from the Castle of Mytilene is non Calcareous. At this sample, the presence of Mullite was detected, apart from Quartz and Plagioclase (Figure 142, Tables 30,31,33). At Monochrome Glazed Wares from the Athenian Agora, Diopside, Gehlenite or Cristobalite were detected, so they were fired at a high temperature  $T > 950-1000$  °C during the Late Byzantine/Frankish to Turkish/Venetian periods. Only one sample (AAG54) was fired at a lower temperature  $T 800-950$  °C because Muscovite/Illite existed. Samples from the Castle of Mytilene were fired at a lower temperature  $T 800-950$  °C than the Athenian Agora during the Turkish/Venetian period. One sample (MYT206) from the Castle of Mytilene is non Calcareous and was fired at a higher temperature  $T > 1000$  °C due to the presence of Mullite (Tables 36,37).

Fragments of **Green and Brown Painted Ware** from **the Athenian Agora in Attica** contained Quartz, Plagioclase, Hematite, Diopside and Cristobalite (Figure 143, Tables 30,32). They were fired at a high temperature  $T > 1000$  °C during the Middle Byzantine period (Table 36).

Fragments of **Polychrome Painted Ware** from **the Castle of Mytilene in Lesvos** (MYT228) consisted of Quartz and Cristobalite (Figure 144, Tables 31,34). The presence of Cristobalite means that it was fired at a high temperature  $T > 1000$  °C during the Turkish/Venetian period. **Painted Ware** MYT16187 from the Castle of Mytilene in Lesvos consisted of Quartz, Plagioclase, Diopside, Gehlenite and Cristobalite and sample MYT191 of Quartz, Plagioclase, K-Feldspars, Hematite, Mullite and Dollomite. They were fired also at a high temperature  $T > 1000$  °C during the Turkish/Venetian to Early Modern periods. At **Unglazed Painted Ware** fragment (MYT207)

from the Castle of Mytilene in Lesvos, Quartz, Plagioclase, K-Feldspars, Muscovite/Illite and Calcite were analysed (Figure 144, Tables 31,34). Specifically, at the first sample, Diopside, Gehlenite and Cristobalite; and at the second sample Mullite and Dollomite were detected. The presence of Muscovite/Illite and Calcite means that was fired at a low temperature T 800-850 °C (Table 37). They were dated during the Turkish/Venetian period.

Fragments of **Polychrome Marbled Ware** from **Chalcis in Euboea** (CH102) was constituted by Quartz, Plagioclase, K-Feldspars, Hematite and Diopside. Samples from **the Castle of Mytilene in Lesvos** consisted of Quartz, Plagioclase, K-Feldspars (apart from MYT211). Samples MYT197, MYT208, MYT211 from the Castle of Mytilene contained also Diopside and Cristobalite; and MYT211 Gehlenite. At sample MYT197 was also observed Hematite. In contrast to them sample MYT165 contained Quartz, Plagioclase, K-Feldspars and Calcite (Figure 145, Tables 29,31,34). At Polychrome Marbled Ware in Chalcis, that came from Italy, Diopside was detected. As well as at samples from the Castle of Mytilene, Diopside, Cristobalite and Gehlenite were observed. This pottery type from Chalcis seemed to be fired at a high temperature T>950 °C and also from the Castle of Mytilene at T>950-1000 °C, during the Turkish/Venetian period. Apart from sample MYT165 that was fired at a lower temperature about T 800-850 °C (Tables 35,37).

Fragments of **Roulette/Veneto Ware** from **Chalcis in Euboea** (CH82) and from **the Castle of Mytilene in Lesvos** (MYT178) contained Quartz, Plagioclase, Hematite and Muscovite/Illite. Sample CH82 also contained K-Feldspars and Calcite (Figure 146, Tables 29,31,34). At Roulette/Veneto Ware that came from Italy and were found in Chalcis and in Mytilene, Muscovite/Illite and Calcite (MYT178) were observed. This pottery type seemed to be fired at a low temperature about T 800-900 °C during the Late Byzantine/Frankish period (Tables 35,37).

Fragments of **Spanish Lustre Ware** from **the Athenian Agora in Attica** (AAG68), which came from Spain, was made up of Quartz, Plagioclase, Hematite, Diopside and Gehlenite. The presence of Diopside and Gehlenite means that it was dated during the Turkish/Venetian period. **Lustre Ware** sample from **Chalcis in Euboea** (CH110), which came from Egypt, contained Quartz, Plagioclase, K-Feldspars, Hematite, Diopside and Gehlenite (Figure 147, Tables 29,30,34). Diopside and Gehlenite were detected and thus it was dated in the Islamic period. Both of these pottery typologies seemed to be fired at a high temperature T>950 °C (Tables 35,37).

At fragments of **Iznik Ware** that came from Western Turkey and were found at **the Castle of Mytilene in Lesvos**, Quartz and Cristobalite were detected (Figure 148, Tables 31,34). Cristobalite was found and as a result they were fired at a high temperature T>1000 °C (Table 37). They were dated during the Turkish/Venetian period.

Fragments of **Kütahya Ware**, which came from Western Turkey, were found at **the Castle of Mytilene in Lesvos** and were made up of Quartz and Cristobalite and sample MYT179 of Diopside also (Figure 149, Tables 31,34). Cristobalite as well as Diopside (MYT179) were detected and thus they were fired at a high temperature T>1000 °C (Table 37). They were dated during the Turkish/Venetian period.

Fragments of **Miletus Ware**, which came from Western Turkey, were found at **the Castle of Mytilene in Lesvos**, Quartz, Plagioclase, K-Feldspars (apart from MYT226) were observed.

Samples MYT167, MYT226 contained Muscovite/Illite and sample MYT226 of Calcite also (Figure 150, Tables 31,34). They were dated during the Turkish/Venetian period. They were fired at a low temperature T 800-950 °C as Muscovite/Illite and Calcite (only at MYT226) were observed (Table 37).

Fragment of **Glazed White Ware II** from **Chalcis in Euboea** (CH107) contained Quartz, Plagioclase and K-Feldspars, whereas fragment of **Glazed White Ware IV** from **the Castle of Mytilene in Lesvos** (MYT203) contained only Quartz and Calcite. Fragment of **Polychrome White Ware** from Chalcis in Euboea (CH108) contained only Quartz (Figure 151, Tables 29,31,34). **Polychrome White Ware** and **Glazed White Ware II** from Chalcis in Euboea were dated in the Middle Byzantine period and **Glazed White Ware IV** from the Castle of Mytilene in Lesvos in the Late Byzantine/Frankish period. They seemed to be fired at a low temperature about T 800-850 °C. Both of these typologies that probably came from Western Turkey (Tables 35,37).

Fragment of **Glazed Frit Ware** (MYT182) from **the Castle of Mytilene in Lesvos** contained Quartz, Plagioclase, K-Feldspars and Diopside. Fragment of **Glazed Frit Ware** (CH106) from **Chalcis in Euboea** contained Quartz, Plagioclase, K-Feldspars and Cristobalite. Finally, fragment of **Frit Ware** (CH147) from Chalcis contained Quartz, Plagioclase, K-Feldspars, Cristobalite and also Mullite (Figure 152, Tables 29,31,34). Glazed Frit Ware sample from the Castle contained Diopside and Glazed Frit Ware sample from Chalcis Cristobalite. All of these seemed to be fired at a high temperature T>950 °C. Glazed Frit Ware was dated during the Turkish/Venetian period and Glazed Frit Ware during the Islamic period. Frit Ware sample from Chalcis was non Calcareous and seemed to have been fired at T >1000 °C due to the existence of Cristobalite and Mullite. This was dated in the Islamic period. All these typologies probably came from Syria or Egypt (Tables 35,37).



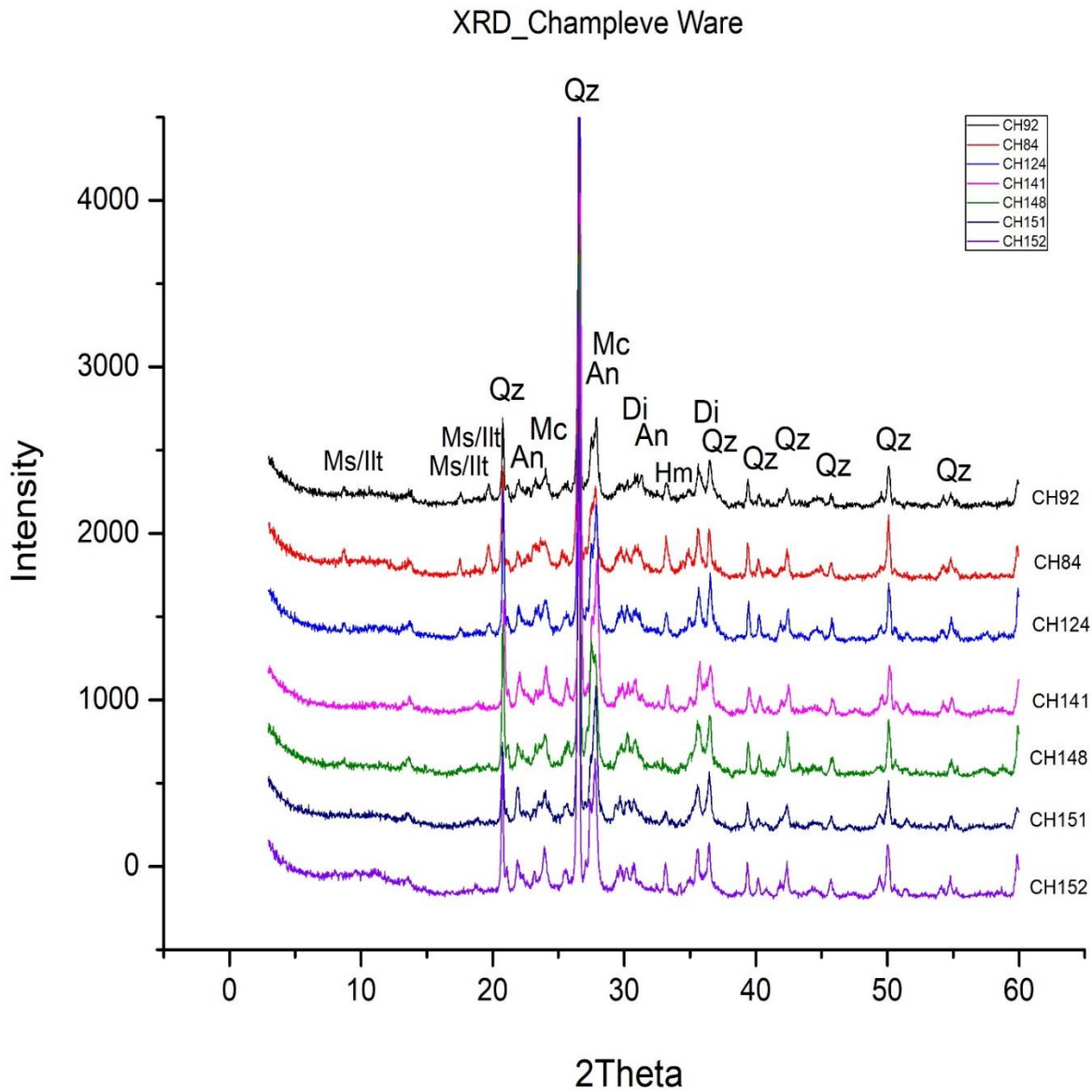


Figure 133 X-Ray Diffraction (XRD) diagrams of Champlevé Ware fragments.

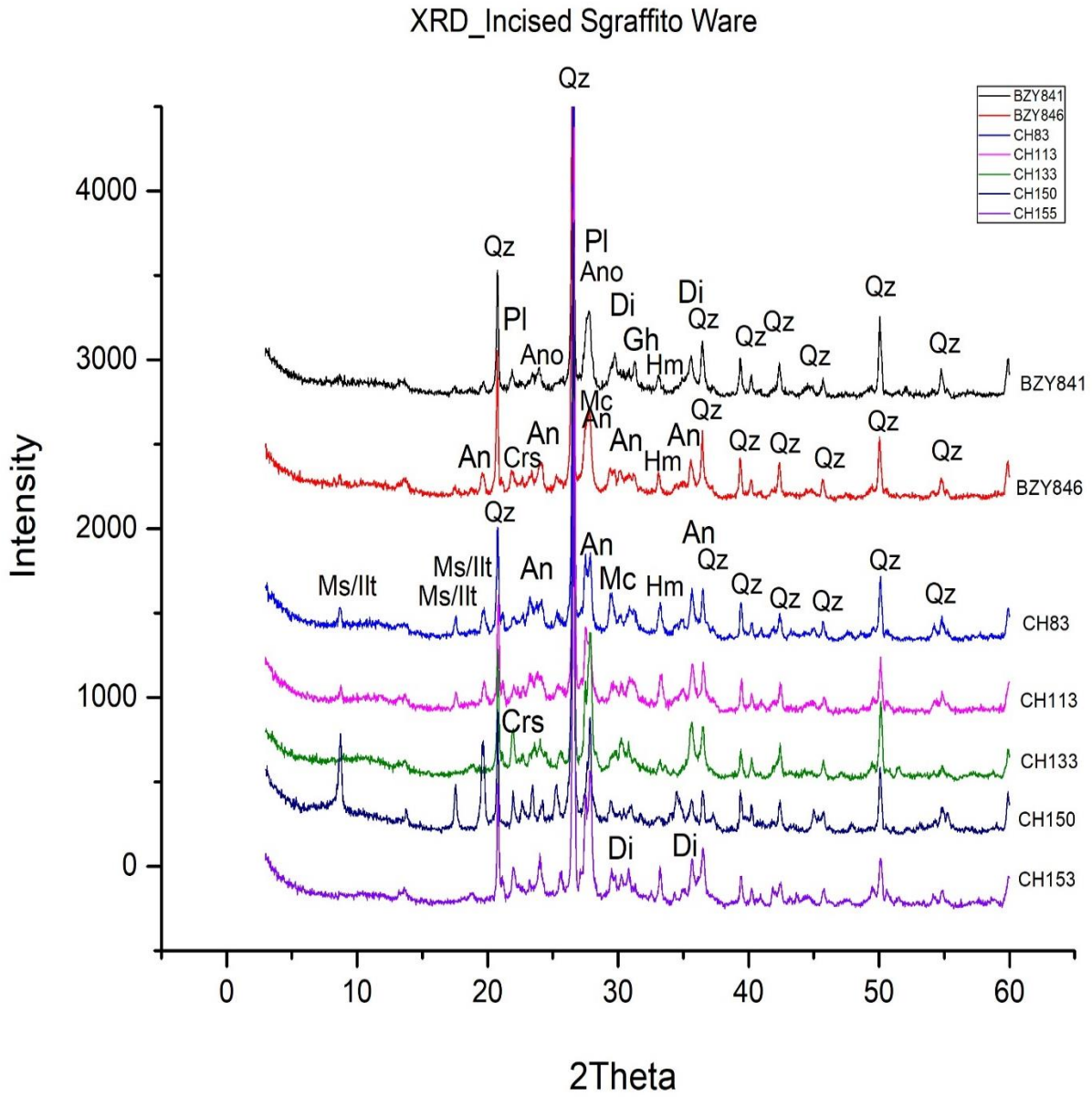


Figure 134 X-Ray Diffraction (XRD) diagrams of Incised Sgraffito Ware fragments.

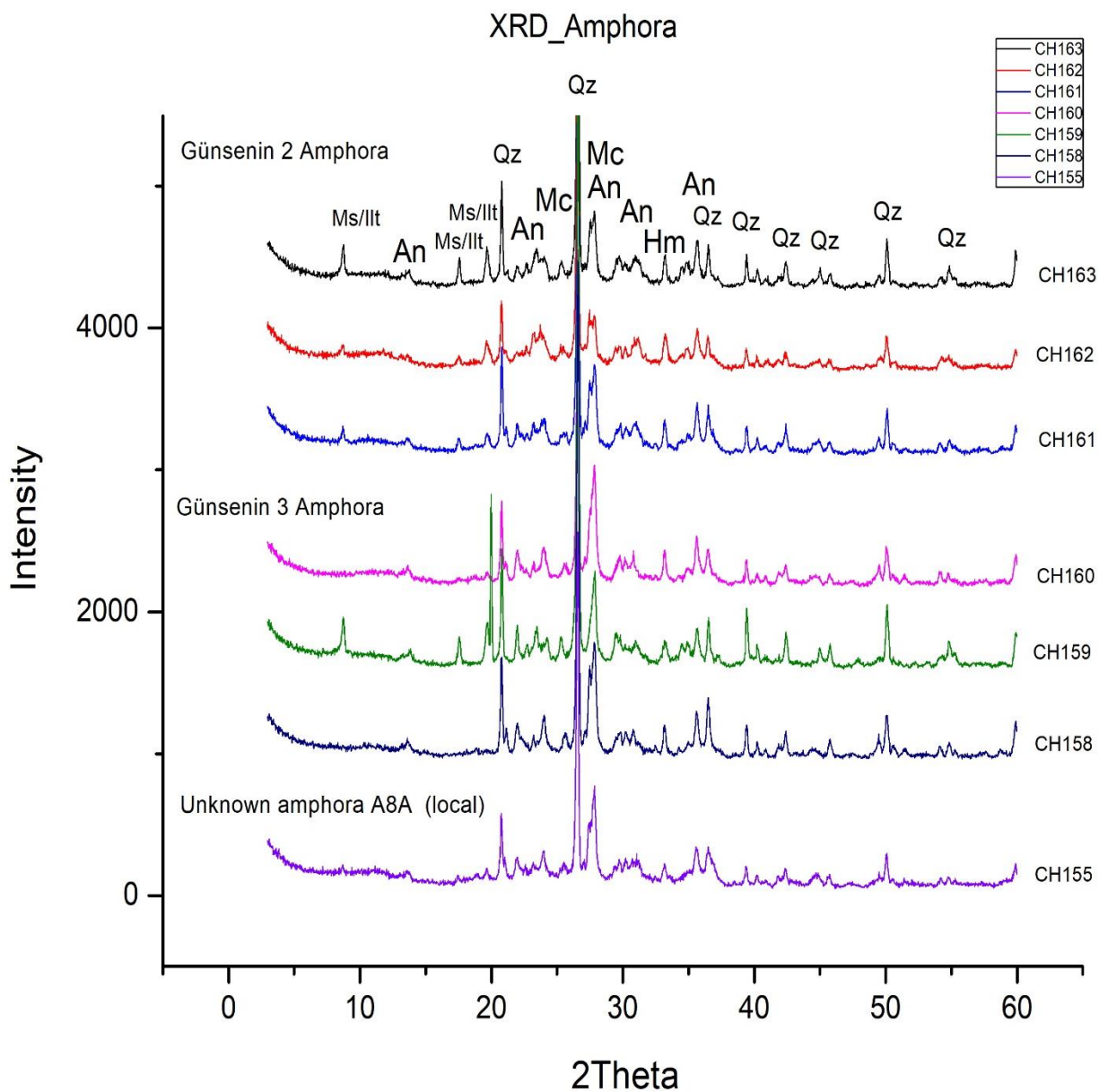


Figure 135 X-Ray Diffraction (XRD) diagrams of Amphora fragments.





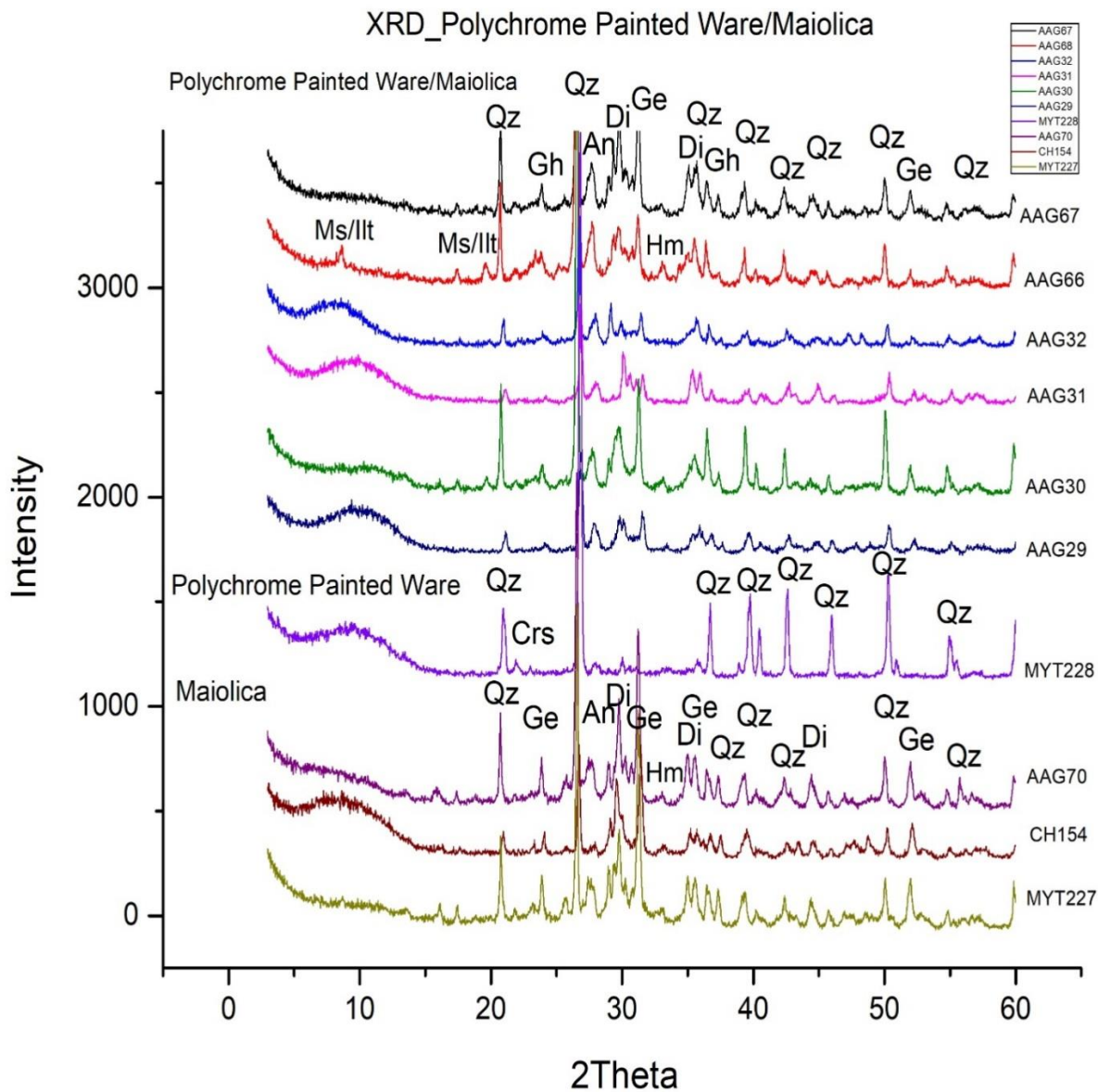


Figure 140 X-Ray Diffraction (XRD) diagrams of Polychrome Painted Ware/Maiolica fragments, Polychrome Painted Ware fragments and Maiolica fragments.

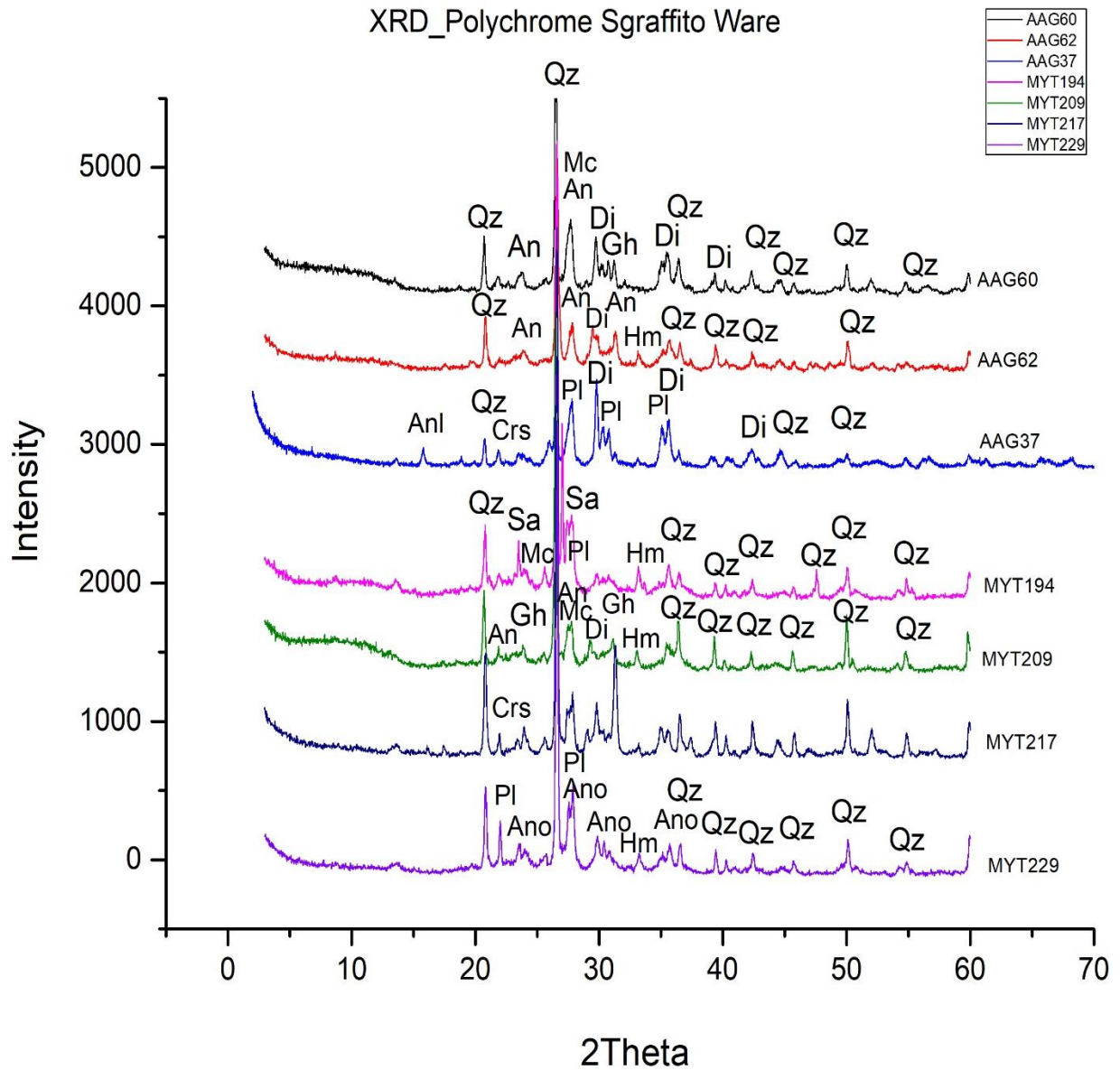


Figure 141 X-Ray Diffraction (XRD) diagrams of Polychrome Sgraffito Ware fragments.



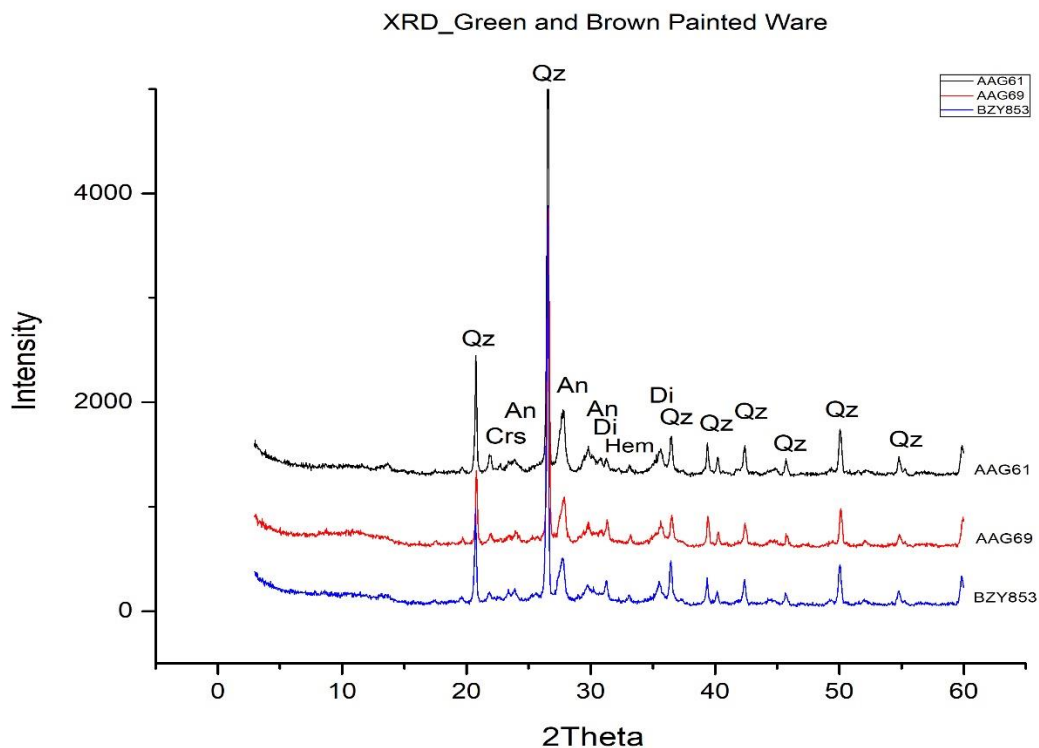


Figure 143 X-Ray Diffraction (XRD) diagrams of Green and Brown Painted Ware fragments.

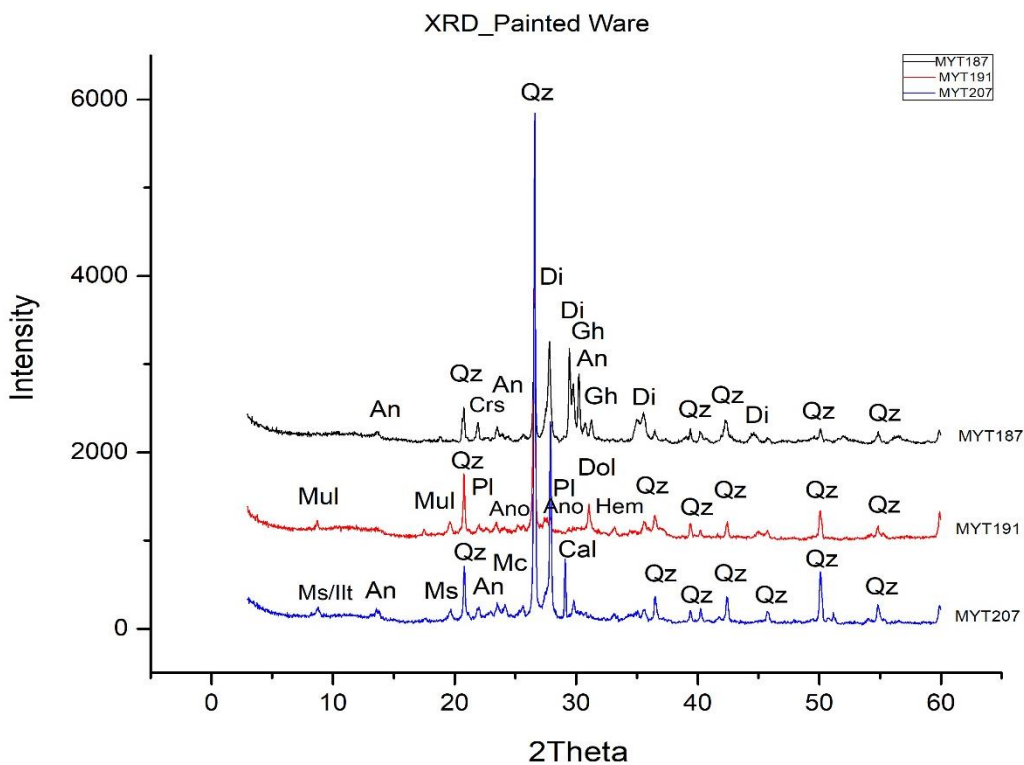


Figure 144 X-Ray Diffraction (XRD) diagrams of Painted Ware fragments.

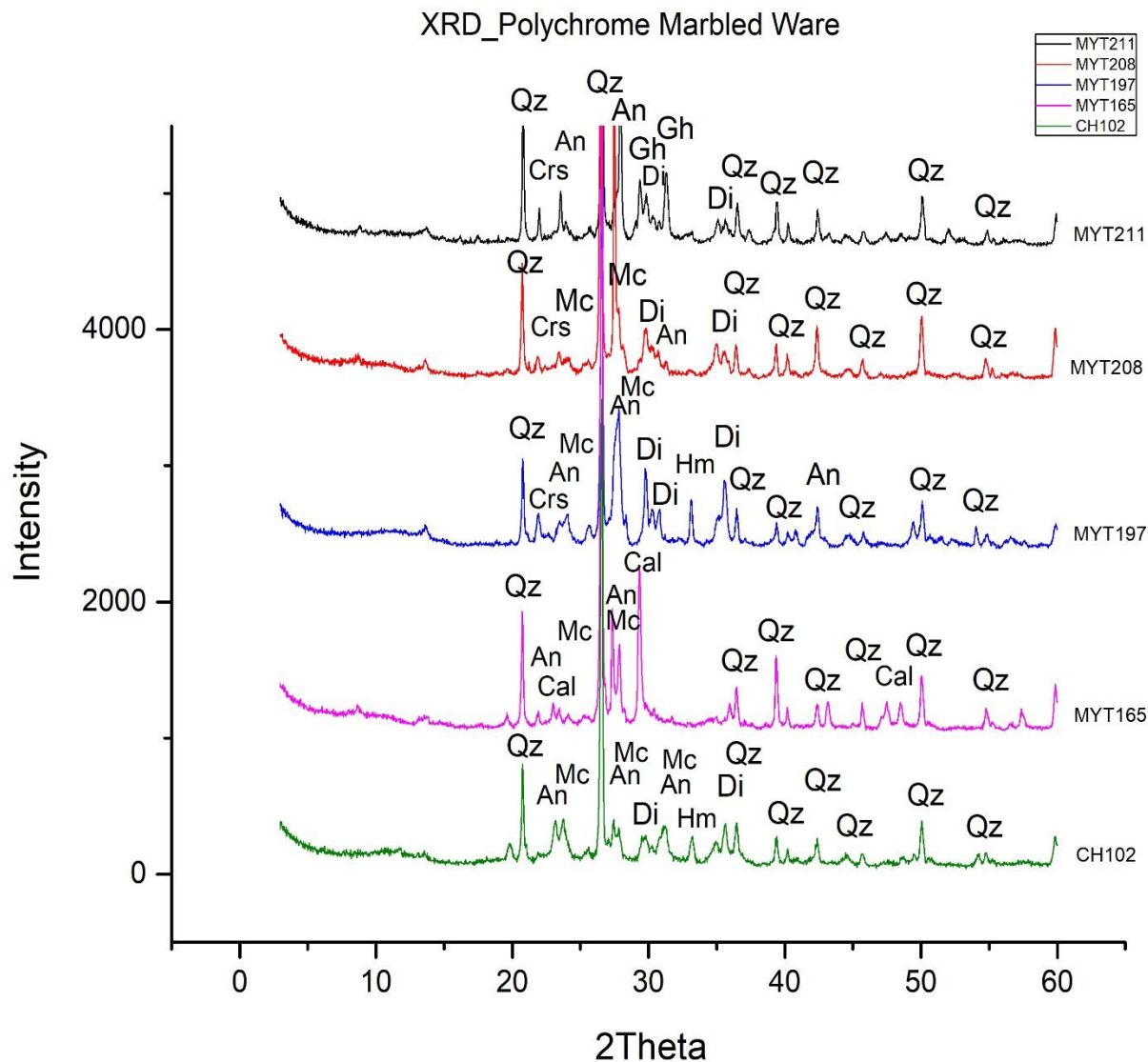


Figure 145 X-Ray Diffraction (XRD) diagrams of Polychrome Marbled Ware fragments.



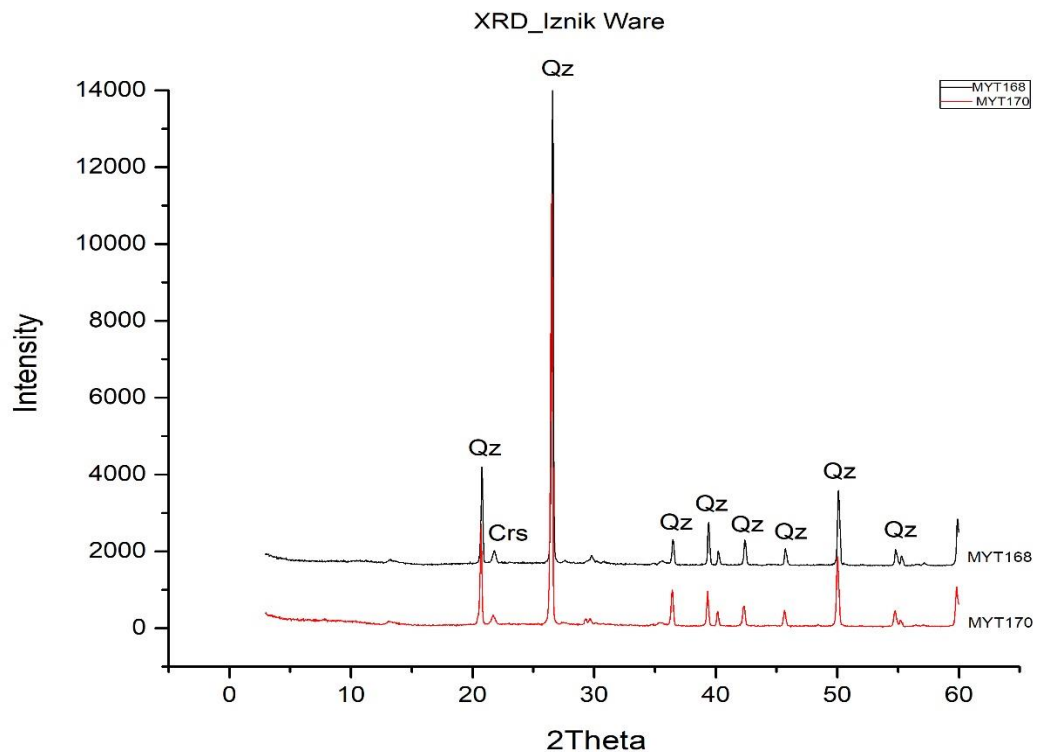


Figure 148 X-Ray Diffraction (XRD) diagrams of Iznik Ware fragments.

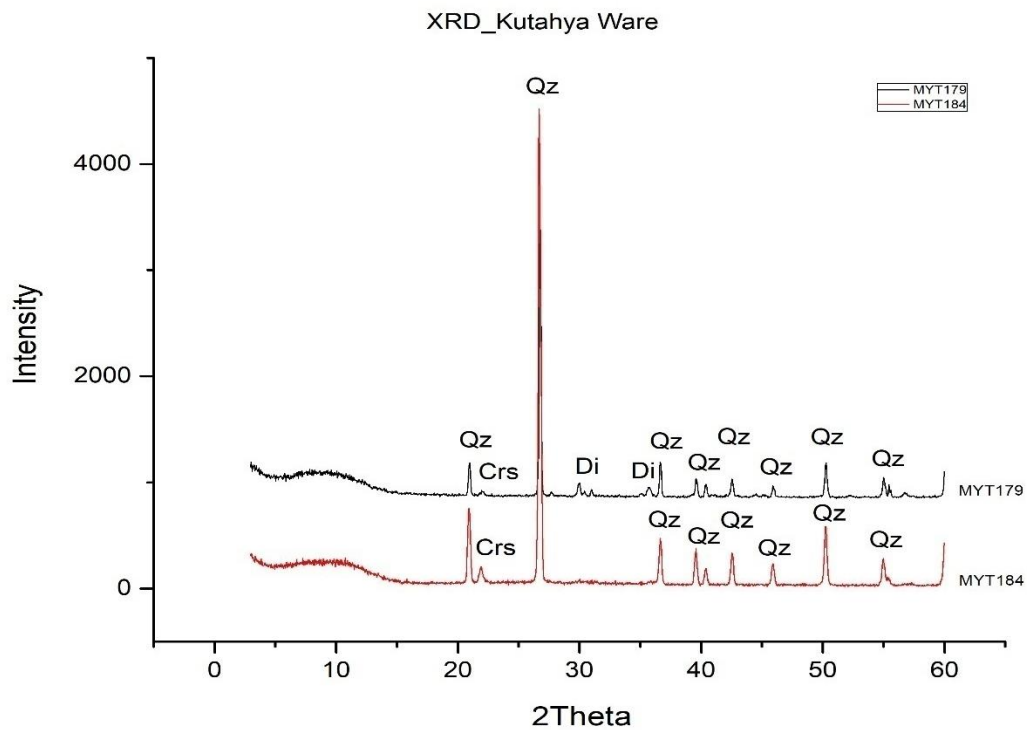


Figure 149 X-Ray Diffraction (XRD) diagrams of Kütahya Ware fragments.

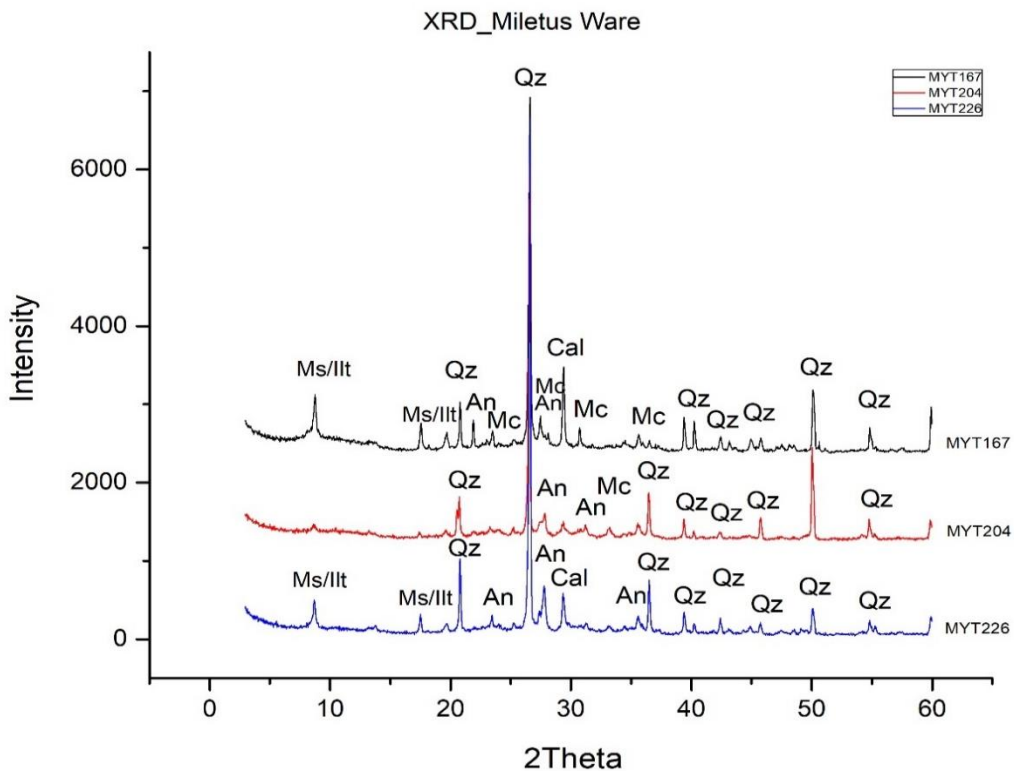


Figure 150 X-Ray Diffraction (XRD) diagrams of Miletus Ware fragments.

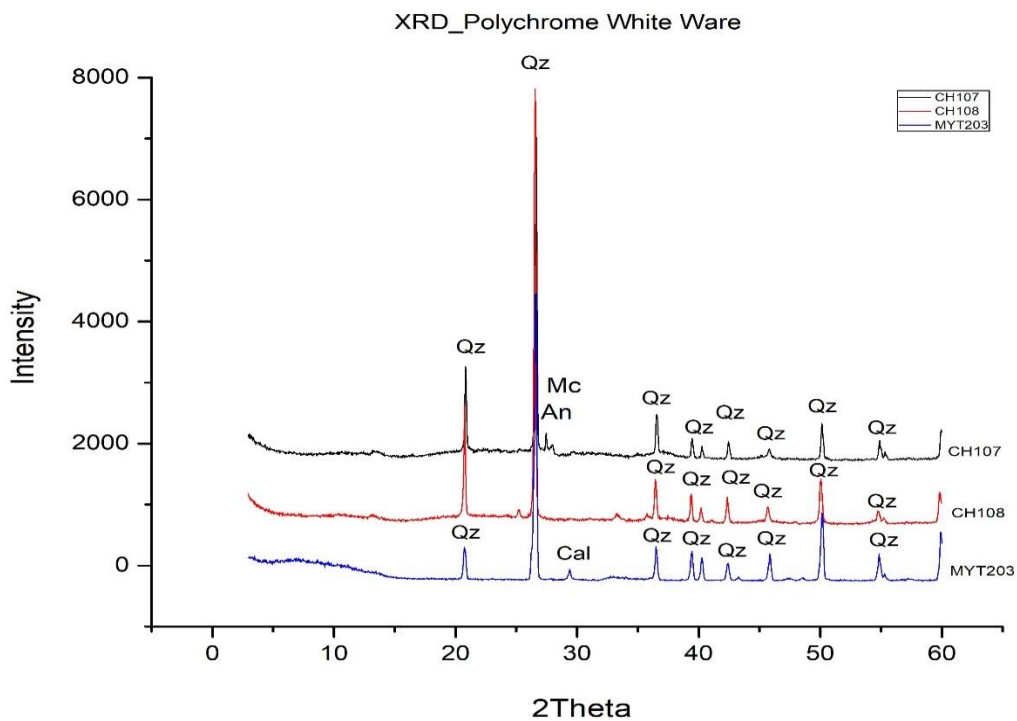


Figure 151 X-Ray Diffraction (XRD) diagrams of Glazed White Ware II (CH107) fragments, Polychrome White Ware (CH108) fragments and Glazed White Ware IV (MYT203) fragments.

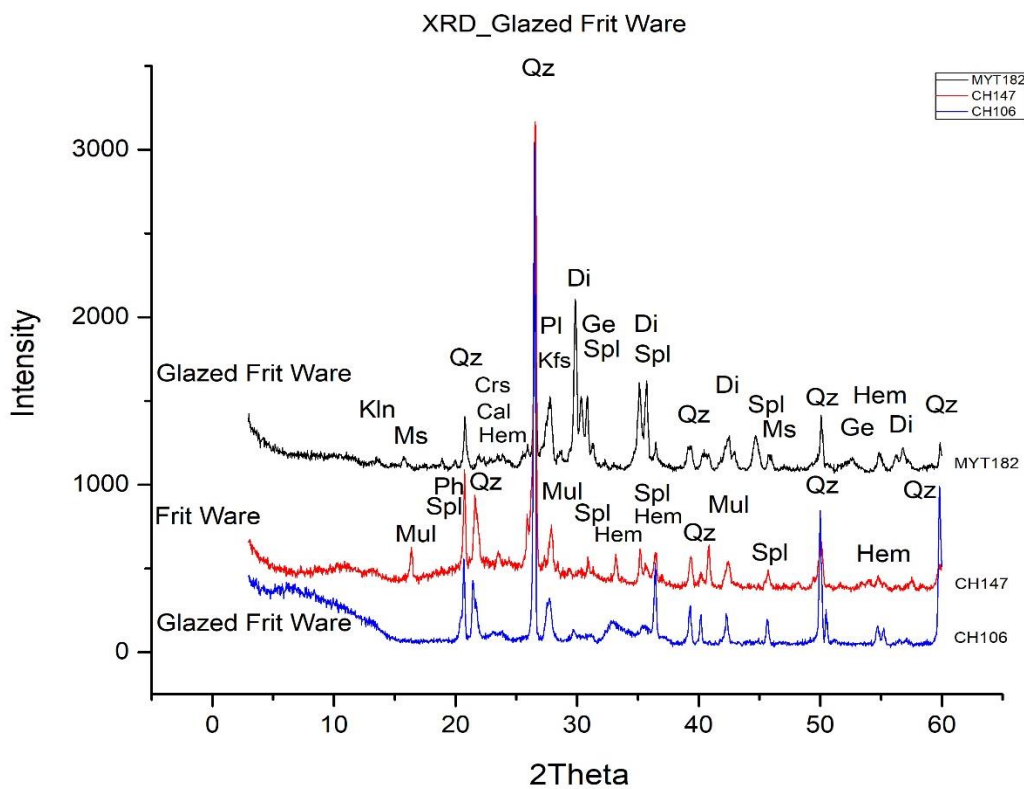


Figure 152 X-Ray Diffraction (XRD) diagrams of fragments of Glazed Frit Ware (MYT182, CH106) fragments and Frit Ware (CH147) fragments.

X-RAY DIFFRACTION AT SAMPLES FROM CHALCIS IN EUBOEA																
		Qz	Pl	Kfs	Hem	Ms/Ilt	Di	Crs	Ge	Mul	Cal	Sa	Hl	Anl	Do	
Champlevé Ware	CH84	+	+	+	+	+	+									
Champlevé Ware	CH92	+	+	+	+	+	+									
Champlevé Ware	CH124	+	+	+	+	+	+									
Champlevé Ware	CH141	+	+	+	+		+									
Champlevé Ware	CH148	+	+	+			+									
Champlevé Ware	CH151	+	+	+	+		+									
Incised Sgraffito Ware	CH83	+	+	+	+	+										
Incised Sgraffito Ware	CH113	+	+	+	+	+										
Incised Sgraffito Ware	CH133	+	+	+	+			+								
Incised Sgraffito Ware	CH150	+	+	+		+										
Incised Sgraffito Ware	CH153	+	+	+	+		+									
A8A amphora	CH155	+	+	+	+	+										
Günsein 3 Amphora	CH158	+	+	+	+											
Günsein 3 Amphora	CH159	+	+	+	+	+		+								
Günsein 3 Amphora	CH160	+	+	+	+		+									
Günsein 2 Amphora	CH161	+	+	+	+	+										
Günsein 2 Amphora	CH162	+	+	+	+	+										
Günsein 2 Amphora	CH163	+	+	+	+	+	+									
Slip-Painted Ware	CH94	+	+	+	+	+										
Slip-Painted Ware	CH121	+	+	+	+		+	+								
Slip-Painted Ware	CH138	+	+	+		+										
Splashed Ware	CH89	+	+			+					+					
Splashed Ware	CH145	+	+	+				+								
Plain Glazed Ware	CH88	+	+		+	+										
Plain Glazed Ware	CH125	+	+		+		+	+								
Maiolica	CH154	+	+				+		+							
Polychrome Marbled Ware	CH102	+	+	+	+		+									
Roulette/Veneto Ware	CH82	+	+	+	+	+					+					
Glazed Frit Ware	CH106	+	+	+				+								
Frit Ware	CH147	+	+	+				+		+						
Polychrome White Ware	CH108	+														
Glazed White Ware II	CH107	+	+	+												
Zeuxippus Ware	CH101	+	+		+		+	+								
Lustre Ware	CH110	+	+	+	+		+		+							

Table 29 X-Ray Diffraction (XRD) in the sampled pottery collection from Chalcis in Euboea.

X-RAY DIFFRACTION AT SAMPLES FROM THE ATHENIAN AGORA IN ATTICA															
		Qz	Pl	Kfs	Hem	Ms/Ilt	Di	CrS	Ge	Mul	Cal	Sa	HI	Anl	Do
Incised Sgraffito Ware	AGBZY841	+	+	+	+		+		+						
Incised Sgraffito Ware	AGBZY846	+	+	+	+			+							
Green and Brown Painted Ware	AGBZY853	+	+		+		+	+							
Green and Brown Painted Ware	AAG61	+	+		+		+	+							
Green and Brown Painted Ware	AAG69	+	+		+		+	+							
Zeuxippus Ware Subtype	AAG43	+	+	+	+	+									
Zeuxippus Ware Subtype	AAG47	+	+		+						+				
Zeuxippus Ware Subtype	AAG50	+	+	+	+	+					+		+		
Slip-Painted Ware	AAG59	+	+	+	+	+									
Polychrome Painted Ware/Maiolica	AAG29	+	+		+		+		+						
Polychrome Painted Ware/Maiolica	AAG30	+	+				+		+						
Polychrome Painted Ware/Maiolica	AAG31	+	+				+		+						
Polychrome Painted Ware/Maiolica	AAG32	+	+				+		+						
Polychrome Painted Ware/Maiolica	AAG66	+	+		+	+	+		+						
Polychrome Painted Ware/Maiolica	AAG67	+	+				+		+						
Maiolica	AAG70	+	+		+		+		+						
Polychrome Sgraffito Ware	AAG37	+	+				+	+						+	
Polychrome Sgraffito Ware	AAG62	+	+		+		+								
Polychrome Sgraffito Ware	AAG60	+	+	+			+	+	+						
Monochrome Glazed Ware	AAG20	+	+		+	+	+		+						
Monochrome Glazed Ware	AAG26	+		+	+		+		+						
Monochrome Glazed Ware	AAG33	+	+				+		+						
Monochrome Glazed Ware	AAG36	+	+		+		+								
Monochrome Glazed Ware	AAG53	+	+				+								
Monochrome Glazed Ware	AAG54	+	+	+		+									
Monochrome Glazed Ware	AAG55	+	+		+		+								
Monochrome Glazed Ware	AAG71	+	+				+	+							
Spanish Lustre Ware	AAG68	+	+		+		+		+						

Table 30 X-Ray Diffraction (XRD) in the sampled pottery collection from the Athenian Agora in Attica.

X-RAY DIFFRACTION AT SAMPLES FROM THE CASTLE OF MYTILENE IN LESVOS															
		Qz	Pl	Kfs	Hem	Ms/Ilt	Di	Crs	Ge	Mul	Cal	Sa	Hl	Anl	Do
Polychrome Painted Ware	MYT228	+						+							
Unglazed Painted Ware	MYT207	+	+	+		+					+				
Maiolica	MYT227	+	+				+		+						
Polychrome Sgraffito Ware	MYT194	+	+	+	+							+			
Polychrome Sgraffito Ware	MYT209	+	+		+		+		+						
Polychrome Sgraffito Ware	MYT217	+	+	+			+	+	+						
Polychrome Sgraffito Ware	MYT229	+	+	+	+										
Monochrome Glazed Ware	MYT199	+	+	+	+										
Monochrome Glazed Ware	MYT206	+	+							+					
Monochrome Glazed Ware	MYT213	+	+		+	+									
Monochrome Glazed Ware	MYT231	+													
Polychrome Marbled Ware	MYT165	+	+	+							+				
Polychrome Marbled Ware	MYT197	+	+	+	+		+	+							
Polychrome Marbled Ware	MYT208	+	+	+			+	+							
Polychrome Marbled Ware	MYT211	+	+				+	+	+						
Roulette/Veneto Ware	MYT178	+	+		+	+									
Glazed Frit Ware	MYT182	+	+	+			+								
Glazed White Ware IV	MYT203	+									+				
Painted Ware	MYT187	+	+				+	+	+						
Painted Ware	MYT191	+	+	+	+					+					+
Miletus Ware	MYT167	+	+	+		+									
Miletus Ware	MYT204	+	+	+											
Miletus Ware	MYT226	+	+			+					+				
Iznik Ware	MYT168	+						+							
Iznik Ware	MYT170	+						+							
Kütahya Ware	MYT179	+					+	+							
Kütahya Ware	MYT184	+						+							

Table 31 X-Ray Diffraction (XRD) in the sampled pottery collection from the Castle of Mytilene in Lesvos.

X-RAY DIFFRACTION DEPENDING ON THE TYPOLOGY 1-3															
		Qz	Pl	Kfs	Hem	Ms/Ilt	Di	Crs	Mul	Cal	Ge	Sa	Hl	Anl	Do
Champlevé Ware	CH84	+	+	+	+	+	+								
Champlevé Ware	CH92	+	+	+	+	+	+								
Champlevé Ware	CH124	+	+	+	+	+	+								
Champlevé Ware	CH141	+	+	+	+		+								
Champlevé Ware	CH148	+	+	+			+								
Champlevé Ware	CH151	+	+	+	+		+								
Incised Sgraffito Ware	AGBZY841	+	+	+	+		+				+				
Incised Sgraffito Ware	AGBZY846	+	+	+	+			+							
Incised Sgraffito Ware	CH83	+	+	+	+	+									
Incised Sgraffito Ware	CH113	+	+	+	+	+									
Incised Sgraffito Ware	CH133	+	+	+	+			+							
Incised Sgraffito Ware	CH150	+	+	+		+									
Incised Sgraffito Ware	CH153	+	+	+	+		+								
Green and Brown Painted Ware	AGBZY853	+	+		+		+	+							
Green and Brown Painted Ware	AAG61	+	+		+		+	+							
Green and Brown Painted Ware	AAG69	+	+		+		+	+							
A8A amphora	CH155	+	+	+	+	+									
Günsenin 3 Amphora	CH158	+	+	+	+										
Günsenin 3 Amphora	CH159	+	+	+	+	+		+							
Günsenin 3 Amphora	CH160	+	+	+	+		+								
Günsenin 2 Amphora	CH161	+	+	+	+	+									
Günsenin 2 Amphora	CH162	+	+	+	+	+									
Günsenin 2 Amphora	CH163	+	+	+	+	+	+								
Zeuxippus Ware Subtype	AAG43	+	+	+	+	+									
Zeuxippus Ware Subtype	AAG47	+	+		+					+					
Zeuxippus Ware Subtype	AAG50	+	+	+	+	+				+			+		
Zeuxippus Ware	CH101	+	+		+		+	+							
Slip-Painted Ware	AAG59	+	+	+	+	+									
Slip-Painted Ware	CH94	+	+	+	+	+									
Slip-Painted Ware	CH121	+	+	+	+		+	+							
Slip-Painted Ware	CH138	+	+	+		+									
Splashed Ware	CH89	+	+			+				+					
Splashed Ware	CH145	+	+	+				+							

Table 32 X-Ray Diffraction (XRD) results that were separated based on the sampled pottery types.

X-RAY DIFFRACTION DEPENDING ON THE TYPOLOGY 2-3															
		Qz	Pl	Kfs	Hem	Ms/Ilt	Di	Crs	Ge	Mul	Cal	Sa	Hl	Anl	Do
Plain Glazed Ware	CH88	+	+		+	+									
Plain Glazed Ware	CH125	+	+		+		+	+							
Polychrome Painted Ware/Maiolica	AAG29	+	+		+		+		+						
Polychrome Painted Ware/Maiolica	AAG30	+	+				+		+						
Polychrome Painted Ware/Maiolica	AAG31	+	+				+		+						
Polychrome Painted Ware/Maiolica	AAG32	+	+				+		+						
Polychrome Painted Ware/Maiolica	AAG66	+	+		+	+	+		+						
Polychrome Painted Ware/Maiolica	AAG67	+	+				+		+						
Polychrome Painted Ware	MYT228	+						+							
Maiolica	AAG70	+	+		+		+		+						
Maiolica	CH154	+	+				+		+						
Maiolica	MYT227	+	+				+		+						
Polychrome Sgraffito Ware	AAG37	+	+				+	+						+	
Polychrome Sgraffito Ware	AAG62	+	+		+		+								
Polychrome Sgraffito Ware	AAG60	+	+	+			+	+	+						
Polychrome Sgraffito Ware	MYT194	+	+	+	+							+			
Polychrome Sgraffito Ware	MYT209	+	+		+		+		+						
Polychrome Sgraffito Ware	MYT217	+	+	+			+	+	+						
Polychrome Sgraffito Ware	MYT229	+	+	+	+										
Monochrome Glazed Ware	AAG20	+	+		+	+	+		+						
Monochrome Glazed Ware	AAG26	+		+	+		+		+						
Monochrome Glazed Ware	AAG33	+	+				+		+						
Monochrome Glazed Ware	AAG36	+	+		+		+								
Monochrome Glazed Ware	AAG53	+	+				+								
Monochrome Glazed Ware	AAG54	+	+	+		+									
Monochrome Glazed Ware	AAG55	+	+		+		+								
Monochrome Glazed Ware	AAG71	+	+				+	+							
Monochrome Glazed Ware	MYT199	+	+	+	+										
Monochrome Glazed Ware	MYT206	+	+							+					
Monochrome Glazed Ware	MYT213	+	+		+	+									
Monochrome Glazed Ware	MYT231	+													

Table 33 X-Ray Diffraction (XRD) results that were separated based on the sampled pottery types.

X-RAY DIFFRACTION DEPENDING ON THE TYPOLOGY 3-3															
		Qz	Pl	Kfs	Hem	Ms/Ilt	Di	Crs	Ge	Mul	Cal	Sa	Hl	Anl	Do
Polychrome Marbled Ware	CH102	+	+	+	+		+								
Polychrome Marbled Ware	MYT165	+	+	+							+				
Polychrome Marbled Ware	MYT197	+	+	+	+		+	+							
Polychrome Marbled Ware	MYT208	+	+	+			+	+							
Polychrome Marbled Ware	MYT211	+	+				+	+	+						
Roulette/Veneto Ware	CH82	+	+	+	+	+					+				
Roulette/Veneto Ware	MYT178	+	+		+	+									
Glazed Frit Ware	MYT182	+	+	+			+								
Glazed Frit Ware	CH106	+	+	+				+							
Frit Ware	CH147	+	+	+				+		+					
Glazed White Ware II	CH107	+	+	+											
Glazed White Ware IV	MYT203	+									+				
Polychrome White Ware	CH108	+													
Spanish Lustre Ware	AAG68	+	+		+		+		+						
Lustre Ware	CH110	+	+	+	+		+		+						
Painted Ware	MYT187	+	+				+	+	+						
Painted Ware	MYT191	+	+	+	+					+					+
Unglazed Painted Ware	MYT207	+	+	+		+					+				
Miletus Ware	MYT167	+	+	+		+									
Miletus Ware	MYT204	+	+	+											
Miletus Ware	MYT226	+	+			+					+				
Iznik Ware	MYT168	+						+							
Iznik Ware	MYT170	+						+							
Kütahya Ware	MYT179	+					+	+							
Kütahya Ware	MYT184	+						+							

Table 34 X-Ray Diffraction (XRD) results that were separated based on the sampled pottery types.

POTTERY TYPES	SAMPLES	XRD	TEMPERATURE °C
Champlevé Ware	CH84	Di	>950
Champlevé Ware	CH92	Di	> 950
Champlevé Ware	CH124	Di	> 950
Champlevé Ware	CH141	Di	> 950
Champlevé Ware	CH148	Di	> 950
Champlevé Ware	CH151	Di	> 950
Incised Sgraffito Ware	CH83	Ms/Ilt	800-950
Incised Sgraffito Ware	CH113	Ms/Ilt	800-950
Incised Sgraffito Ware	CH150	Ms/Ilt	800-950
Incised Sgraffito Ware	CH133	Crs	> 1000
Incised Sgraffito Ware	CH153	Di	> 950
A8A amphora	CH155	Ms/Ilt	800-950
Günsenin 3 Amphora	CH158	Qz, Pl, Kfs	> 950
Günsenin 3 Amphora	CH159	Crs	> 1000
Günsenin 3 Amphora	CH160	Di	> 950
Günsenin 2 Amphora	CH161	Ms/Ilt	800-950
Günsenin 2 Amphora	CH162	Ms/Ilt	800-950
Günsenin 2 Amphora	CH163	Di	> 950
Slip-Painted Ware	CH94	Ms/Ilt	800-950
Slip-Painted Ware	CH138	Ms/Ilt	800-950
Slip-Painted Ware	CH121	Di, Crs	> 1000
Splashed Ware	CH89	Ms/Ilt	800-950
Splashed Ware	CH145	Crs	> 1000
Plain Glazed Ware	CH88	Ms/Ilt	800-950
Plain Glazed Ware	CH125	Di, Crs	> 1000
Maiolica	CH154	Di, Ge	> 950
Polychrome Marbled Ware	CH102	Di	> 950
Roulette/Veneto Ware	CH82	Ms/Ilt, Cal	800-850
Glazed Frit Ware	CH106	Di	> 950
Frit Ware	CH147	Di, Mul	> 1000
Polychrome White Ware	CH108	Qz	800-850
Glazed White Ware II	CH107	Qz, Pl	800-850
Zeuxippus Ware	CH101	Di, Crs	> 1000
Lustre Ware	CH110	Di, Ge	> 950

Table 35 Firing Temperature according to X-Ray Diffraction (XRD) analyses in the sampled pottery collection from Chalcis in Euboea.

POTTERY TYPES	SAMPLES	XRD	TEMPERATURE °C
Polychrome Painted Ware/Maiolica	AAG29	Di, Ge	> 950
Polychrome Painted Ware/Maiolica	AAG30	Di, Ge	> 950
Polychrome Painted Ware/Maiolica	AAG31	Di, Ge	> 950
Polychrome Painted Ware/Maiolica	AAG32	Di, Ge	> 950
Polychrome Painted Ware/Maiolica	AAG66	Di, Ge	> 950
Polychrome Painted Ware/Maiolica	AAG67	Di, Ge	> 950
Maiolica	AAG70	Di, Ge	> 950
Polychrome Sgraffito Ware	AAG37	Di, Crs, Anl	> 1000
Polychrome Sgraffito Ware	AAG60	Di, Ge	> 950
Polychrome Sgraffito Ware	AAG62	Di	> 950
Incised Sgraffito Ware	AGBZY841	Di, Ge	> 950
Incised Sgraffito Ware	AGBZY846	Crs	>1000
Green and Brown Painted Ware	AGBZY853	Di, Crs	>1000
Green and Brown Painted Ware	AAG61	Di, Crs	>1000
Green and Brown Painted Ware	AAG69	Di, Crs	>1000
Zeuxippus Ware Subtype	AAG43	Ms/Ilt	800-950
Zeuxippus Ware Subtype	AAG47	Cal	800-850
Zeuxippus Ware Subtype	AAG50	Ms/Ilt, Cal	800-850
Monochrome Glazed Ware	AAG20	Di, Ge	> 950
Monochrome Glazed Ware	AAG26	Di, Ge	> 950
Monochrome Glazed Ware	AAG36	Di	> 950
Monochrome Glazed Ware	AAG53	Di	> 950
Monochrome Glazed Ware	AAG54	Ms/Ilt	800-950
Monochrome Glazed Ware	AAG55	Di	> 950
Monochrome Glazed Ware	AAG71	Di, Crs	>1000
Slip-Painted Ware	AAG59	Ms/Ilt	800-950
Spanish Lustre Ware	AAG68	Di, Ge	> 950

Table 36 Firing Temperature according to X-Ray Diffraction (XRD) analyses in the sampled pottery collection in Attica from the Athenian Agora pottery.

POTTERY TYPES	SAMPLES	XRD	TEMPERATURE °C
Polychrome Sgraffito Ware	MYT229	Qz, Pl, Kfs	> 1000
Polychrome Sgraffito Ware	MYT194	Qz, Pl, Kfs, Sa	> 1000
Polychrome Sgraffito Ware	MYT217	Di, Crs, Ge	> 1000
Polychrome Sgraffito Ware	MYT209	Di, Ge	> 950
Polychrome Painted Ware	MYT228	Crs	> 1000
Painted Ware	MYT191	Mul, Do	> 1000
Painted Ware	MYT187	Di, Crs, Ge	> 1000
Polychrome Marbled Ware	MYT165	Cal	800-850
Polychrome Marbled Ware	MYT197	Di, Ge	> 950
Polychrome Marbled Ware	MYT208	Di, Ge	> 950
Polychrome Marbled Ware	MYT211	Di, Ge, Crs	> 1000
Monochrome Glazed Ware	MYT213	Ms/Ilt	800-950
Monochrome Glazed Ware	MYT199	Qz, Pl, Kfs	800-850
Monochrome Glazed Ware	MYT206	Mul	> 1000
Monochrome Glazed Ware	MYT231	Qz	> 1000
Unglazed Painted Ware	MYT207	Ms/Ilt, Cal	800-850
Glazed White Ware IV	MYT203	Cal	800-850
Roulette/Veneto Ware	MYT178	Ms/Ilt	800-950
Iznik Ware	MYT168	Crs	> 1000
Iznik Ware	MYT170	Crs	> 1000
Kütahya Ware	MYT179	Di, Crs	> 1000
Kütahya Ware	MYT184	Crs	> 1000
Miletus Ware	MYT167	Ms/Ilt	800-950
Miletus Ware	MYT204	Qz, Pl, Kfs	800-900
Miletus Ware	MYT226	Ms/Ilt, Cal	800-850
Glazed Frit Ware	MYT182	Di	> 950
Maiolica	MYT227	Di, Ge	> 950

Table 37 Firing Temperature according to X-Ray Diffraction (XRD) analyses in the sampled pottery collection from the Castle of Mytilene in Lesvos.