

COMPARATIVE ANALYSIS OF MICROSCOPIC IMAGES AND XRF AND EDS RESULTS OF BRICKS FROM ARHEOLOGICAL SITES MEDIANA AND NAISUS

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Abstract. *This paper describes the archaeological sites Mediana and Naisus during Late Antiquity. Microscopic images of bricks, and the results and analysis of XRF bricks from these archaeological sites are shown. Based on the results, it can be concluded that a similar brick exterior, and approximately the same chemical composition. One reason is, most likely, a similar chemical composition of the soil, because the archaeological sites are geographically close to each other. Another reason could be the same way bricks were produced, and that the same fuel was used in the kilns.*

Key words: *Mediana, Naisus, brick, microscopic images, XRF and EDS results, late antiquity*

1. INTRODUCTION

The archaeological site of Mediana is a suburb of ancient Nais and is located in the eastern part of today's Niš, on the road to Niška Banja (Figure 1). The residence of the Roman emperors was built in the early fourth century, in the time of Constantine the Great (*Flavius Valerius Aurelius Constantinus*, 274 - 337, Roman Emperor 306 - 337). During the Roman Empire it was a luxury suburb Roman town and military camp Nais. Magnificent imperial summer residence, with additional villas and other buildings were built next to the river Nišava and hot springs. The central space is a villa with peristyle of 6 000m², with elaborate mosaics and beautiful frescoes, ornate pillars, with spa and barns on the west side, the system of water supply (aqueduct and the water tower) on the south side.

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Fig. 1 Archeological Site Mediana, 41°8'9"N 81° 51'51" W

Many Roman objects were found in Mediana, ceramics and metals, as well as decorative marbles. In one room, a group of damaged statues was discovered in 1972. They represent Askelepie (*Ἀσκληπιός*, *Asklepios*, *Aesculapius*, the Greek mythology, the god of health), Hygeia (*Ἑγεία*, *Hygia*, in Greek mythology, patron of pharmacists), Dionysus (*Διώνυσος*, *Dionysos*, *Dionysos*, god of wine), Hercules - Hercules (*Ἡρακλῆς*, *Heracles*, he was a demigod) and Satyrs (*σάτυρος*, *Satyros* - *Σάτυροι*, *Sátyroi*, in Greek mythology, semi-human and half-animals, but they are not immortal demigods) [1].

Mediana was completely destroyed during the Hun invasion of 441 year. At the beginning of the VIII century, during the raid of Slavs and Avars, the summer residence of the imperial city was desolated forever.



Fig. 2 Archaeological Site Naissus, 43 ° 19'09 "N 21 ° 53'46"E

Naissus (*Naissus*) is an ancient Roman city. It was first settled by Dardanians in the III century BC (Figure 2). Roman emperor Constantine the Great (*Flavius Valerius Aurelius*

Constantinus, 274 - 337, Roman Emperor 306 - 337), and Constantius II (*Flavius Constantius*, ? - 421) were born in Naisus. During the V and VI century BC it was alternately raided by the Huns, Ostrogoths, Gepids, Avars and Byzantines, but the Slavic tribes conquered it in 612-614. year. At the site of the ancient Naisus today is the city of Niš. Naisus was developed in the western part of the valley of Niš, which is connected by the river basins with neighboring central Balkan region [2], on the bank of the right. Throughout the city passed important roads: *Via Militaris*, which led from Singidunum and Viminacium to Serdica and Constantinople; military road connecting Raciariu the Danube (*Archar in Bulgaria*) and Lissus (*Lissus, Lezha in Albania*). During the reign of Diocletian (*Gaius Valerius Auerlius Diocletianus*, 236 or 237 - 316), the city belonged to Upper Moesia. Later it belonged to Mediterranean Dacia (*Dacia Mediterranea*). In the I century Naisus had no significance for the Roman Empire. Over time, it was colonized and received the status of a municipality (*municipium*). Usually municipalities were not founded by the Romans, but were created by dividing the existing settled unroman places. Their citizens, unlike the colonies did not have Roman citizenship, but the Latin law (*ius Latii*), but they had a duty of Roman citizens, they payed taxes, they were subject to military service, but did not take part in political life. In the executive government there were four magistrates elected for one year, while authorities performed an advisory like the Senate. Municipia lost their meaning when, Emperor Caracalla (*Lucius Septimius Basijana, Lucius Septimius Bassianus, from the age of seven, Marcus Aurelius Antoninus, Marcus Aurelius Antoninus*, 186 - 217) gave all subjects Roman citizenship with the edict *Constitutio Antoniniana* in 212.. The name *Caracalla* comes from the Gaul tunic, which he wore and introduced to fashion. He was the son and successor of Septimius Severus (*Lucius Septimius Sever, Leptis Magna*, 146 - 211) [3]. From the expression of *municipium* originated the term for the municipality.

At the time of Diocletian (*dating was performed on the coins*), just north of the city, outside the walls (*extra muros*), monumental baths with hypocaust were built. Naisus was ruled by Licinius (*Flavius Galerius Valerius Licinius Licinianus*, 250 - died in 325), after the defeat at Vinkovci 316, led against Constantine. The inscriptions on the altar indicates that they mainly represented the cults of the Roman gods: Jupiter, Juno, Fortune, Liber and Libera, Hercules, and Mitre as well as the Thracian and Illyrian deities. Since there is no established function of the luxurious object under the arches, with decorations and on walls and the floor, it is assumed that it was a temple dedicated to Jupiter [4]. In the mortar in the octagonal room were found bronze coins Maximinus Daia (*Gaius Valerius Galerius Maximinus, Maximinus Daia, Daza, Maximinus II*, 270 - 313), and it is assumed that the building is from the fourth century. Around 4km north of Villa Naisus was a villa on Vinik, which is not fully explored, but in it is found greater quantities of silver coins, along with molds for casting.

In a late antique record is reported that in the Naisus factory have existed: a workshop for the production of military equipment, because of the proximity of an area rich in iron ore and workshops for the development of precious metals, especially silver [5] [6].

2. RESULTS AND DISCUSSION

Bricks from the archaeological site of Mediana are slightly lighter brick-red. By the looks of the sample can be noted the presence of iron and silica material (Figures 3 and 4). Iron ions, which are components of the bricks, give the characteristic red color, while the quartz material gives an inhomogeneous structure, and building materials more resistant and suitable for constructing buildings and walls. Fragments of the rest of the residence, with

no visible evidence of a possible seal manufacturer, so the statement is that the bricks were built and manufactured on the spot.



Fig. 3 Sample bricks from the archaeological site of Mediana

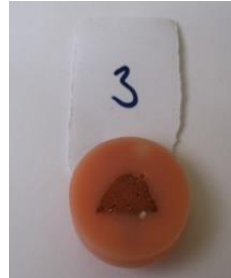


Fig. 4 Brick pattern (mold) from the site of Mediana

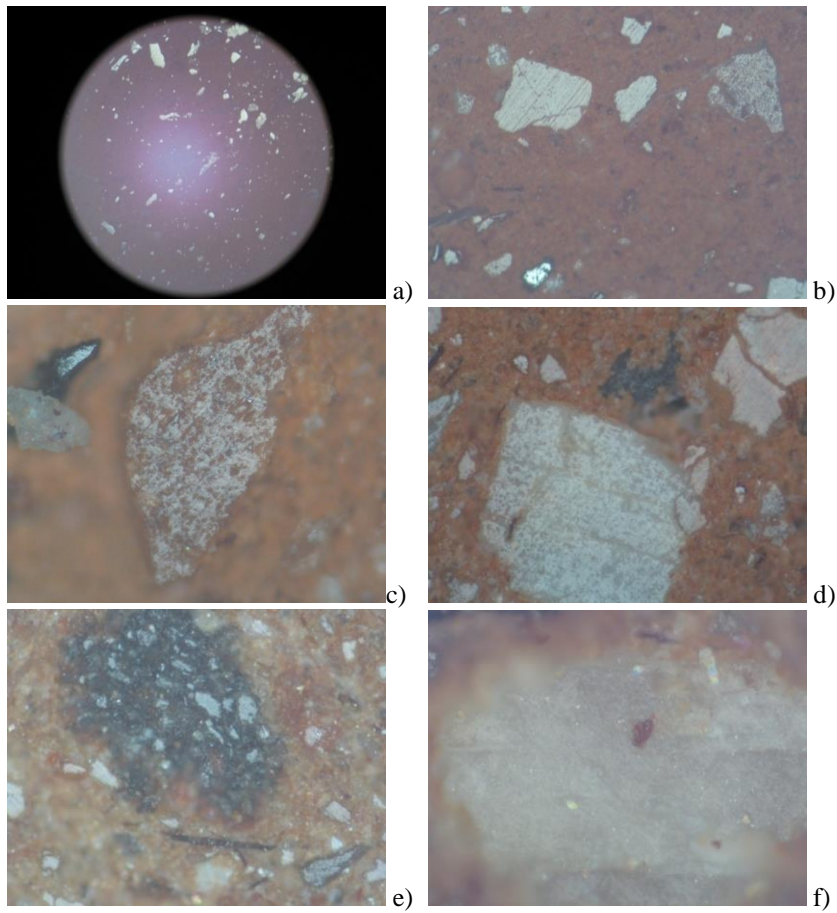


Fig. 5. Microscopic images of the sample from the site of Mediana

Micrographs show that the brick structure is inhomogeneous and that the basic raw material for making bricks were: clay, red sand and quartz. According to Figure 5 (a, b, c, d, e, f), the sample is grain structure, considerable inhomogeneity, but there is no rule in the geometric arrangement of particles. Notable are coarse quartz and other silicate minerals composition, with no carbonate. The sample contains a lower percentage of iron and nearly twice as much aluminum, while the percentage of magnesium, titanium and alkali metals is negligible (the components of clay Substances Directive). Analysis of the structure of bricks from the archaeological site Mediana was performed with results and microscopic images of archaeological sites in other parts of the world, in a similar period of time [7] [8] [9] [10].

In confirmation, on the convenience of building materials for the construction of residences in the reign of Constantine the Great, the recording was made by electron microscopy, shows a compact structure with small inhomogeneities. Practically, this is a confirmation of the conclusion given by the optical microscopic analysis of this sample (Figures 6 and 7, and Tables 1 and 2).

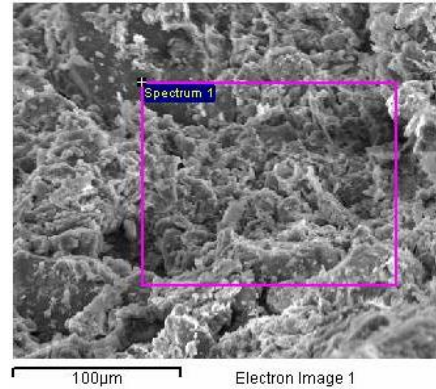


Fig. 6. SEM Median brick

Table 1. EDS analysis bricks Median, elements in the sample (%)

Spectrum	In stats.	O	Na	Mg	Al	Si	K	Ca	Ti	Fe	Total
Spectrum 1	Yes	54.70	1.29	1.41	8.32	25.81	1.80	1.27	0.63	4.76	100.00
Mean		54.70	1.29	1.41	8.32	25.81	1.80	1.27	0.63	4.76	100.00

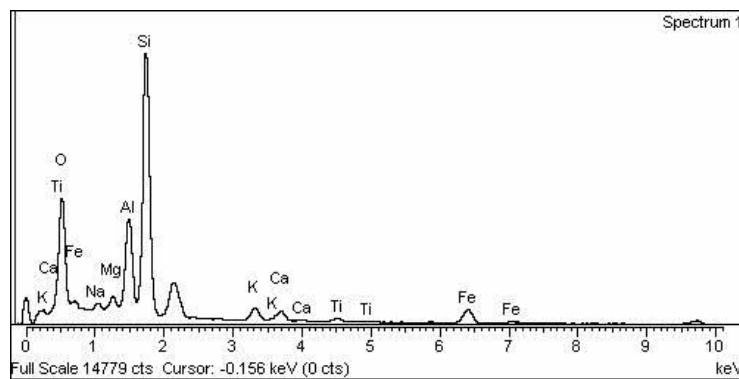
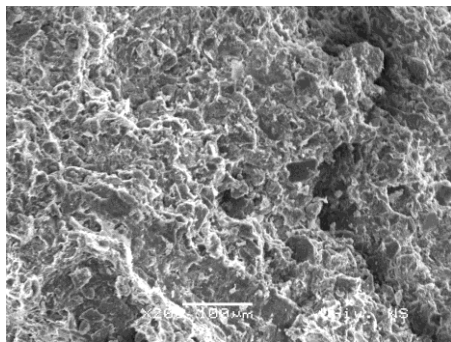


Fig. 7 EDS Median brick

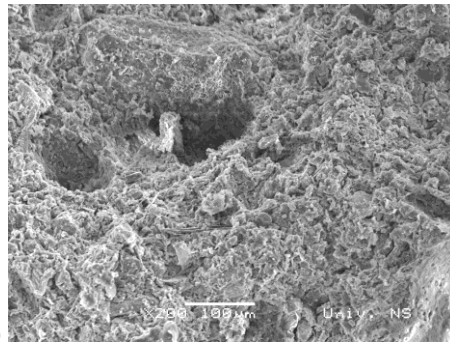
Table 2. EDS analysis of brick Median%

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
O K	39.49	0.5499	54.70	0.35	69.26
Na K	1.05	0.6172	1.29	0.11	1.14
Mg K	1.14	0.6174	1.41	0.08	1.18
Al K	8.02	0.7340	8.32	0.11	6.25
Si K	25.42	0.7501	25.81	0.22	18.61
K K	2.13	0.9026	1.80	0.05	0.93
Ca K	1.46	0.8797	1.27	0.05	0.64
Ti K	0.64	0.7751	0.63	0.05	0.27
Fe K	5.06	0.8102	4.76	0.10	1.73
Totals			100.00		

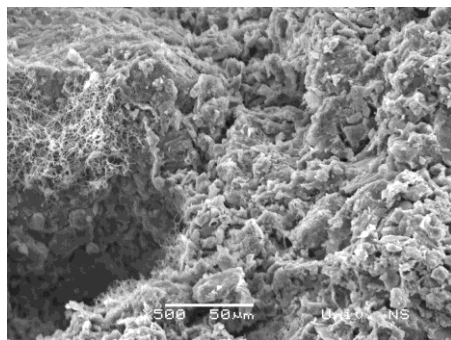
All the analysis and commentary Figure 8 (a, b, c, d, e, f, g) is made based on the published works of foreign researchers and at 200 magnification (Figure 8.a., b.) and at 500 magnification (Figure 8.c.) and the 1000 magnification (Figure 8.d., e.) and with magnification 2 000 (Figure 8.f., g.) shows the flaky structure of quartz [11] [12].



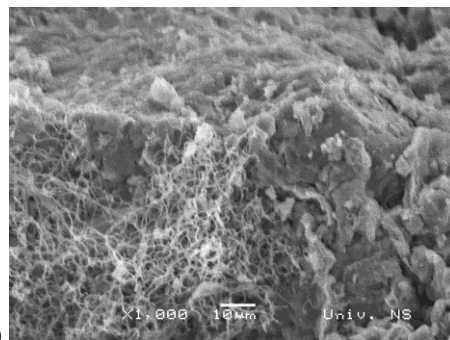
a)



b)



c)



d)

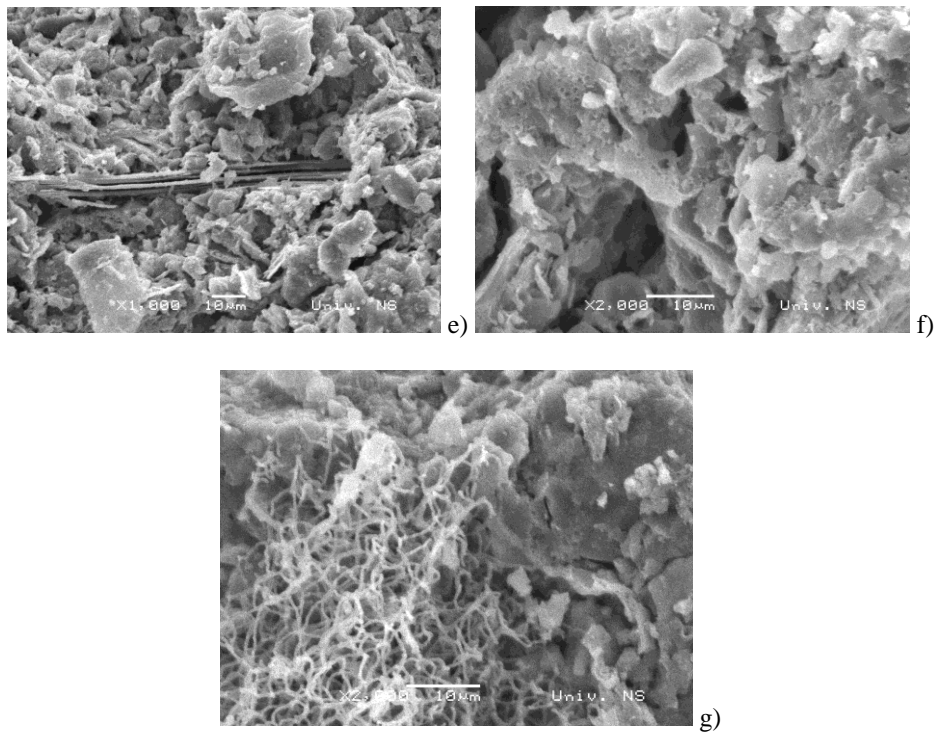


Fig. 8 SEM Median brick

Sample bricks from the archaeological site Naisus are the characteristic red color, due to the presence of iron, and quartz material giving it an inhomogeneous structure (Figures 9 and 10). The fragment of brick is from the walls of the factory, workshop for production of military equipment, which most likely dates from the fourth century. The quality of the brick states that the sample was not part of the temple dedicated to Jupiter.



Fig. 9 Sample bricks from the archaeological site Naissus



Fig. 10 Brick pattern (mold) from the site Naissus

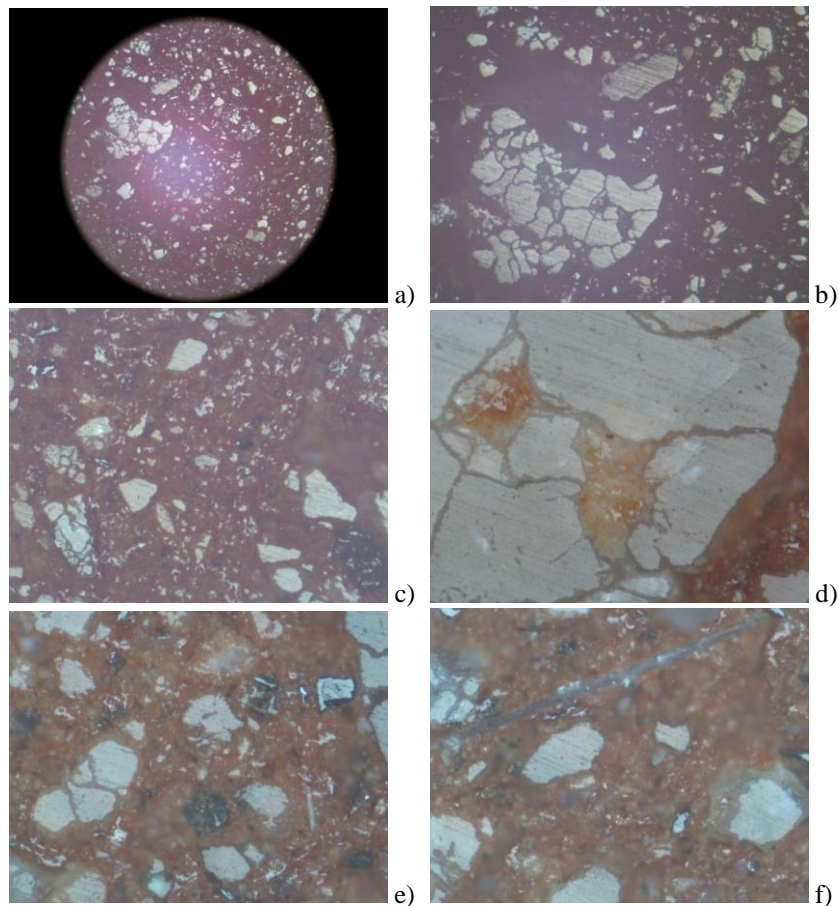


Fig. 11 Microscopic images of the sample from the site naissus

Micrographs show that the brick structure is moderately inhomogeneous, which means that the basic raw material for making are clay, red in color and quartz sand. According to Figure 11 (a, b, c, d, e, f), the sample is grain structured, does not show significant unhomogenic, but there is no rule in the geometric arrangement of particles. At higher magnification can be observed that there are bigger parts of quartz and other silicate mineral composition, the composition of which are not carbonates, except for one minor offenses. In addition to iron and aluminum in tarces can also be found magnesium, titanium and alkali metals as well as clay. Inhomogeneous structure is observed in the micrographs and confirms that the ceramic substance, is a type of bricks and can only be used for the building and is not suitable for making containers or ceramic bowl. Analysis of the structure of bricks from the archaeological site Naisus was performed with results and microscopic images of archaeological sites of other parts of the world, in a similar period of time [12] [13] [14].

Confirmation, on the convenience of building materials for the construction of a factory for the production of military equipment at the archaeological site Naisus, is the recording made by electron microscopy, and it shows a compact structure with small inhomogeneities. Practically, this is a confirmation of the conclusion given by the optical microscopic analysis of this sample (Figures 12 and 14, and Tables 3 to 5).

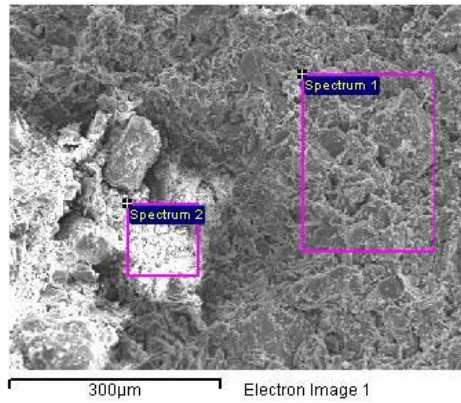


Fig. 12 SEM Naissus brick

Table 3 EDS Aanalysis Naissus bricks, elements in the sample (%)

Spectrum	In stats.	C	O	Na	Mg	Al	Si	Cl	K	Ca	Ti	Fe	Total
Spectrum 1	Yes	0.00	53.74	1.33	1.47	8.56	25.02		2.00	1.67	0.49	5.73	100.00
Spectrum 2	Yes	22.79	48.90	0.67	1.05	4.99	12.98	0.95	0.85	2.32	0.37	4.13	100.00

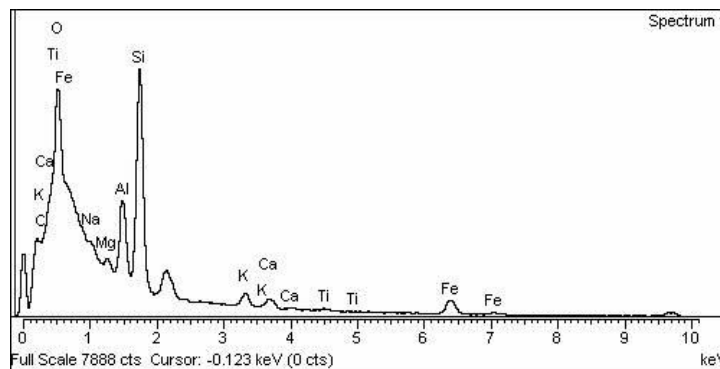
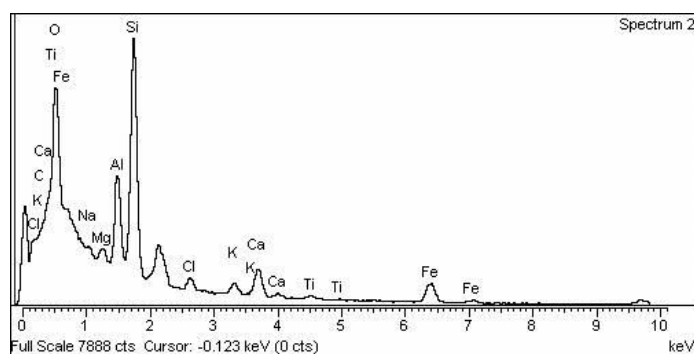


Fig. 13 EDS Naissus brick

Table 4 EDS analysis bricks Naissus%

Element	App Conc.	Intensity Corn.	Weight%	Weight% Sigma	Atomic%
C K	0.00	0.1142	0.00	0.00	0.00
O K	18.44	0.5424	53.74	0.94	68.68
Na K	0.51	0.6101	1.33	0.33	1.18
Mg K	0.57	0.6113	1.47	0.19	1.23
Al K	3.94	0.7279	8.56	0.23	6.49
Si K	11.78	0.7442	25.02	0.52	18.21
K K	1.15	0.9058	2.00	0.11	1.05
Ca K	0.93	0.8815	1.67	0.10	0.85
Ti K	0.24	0.7764	0.49	0.09	0.21
Fe K	2.94	0.8118	5.73	0.20	2.10
Totals			100.00		

**Fig. 14** EDS Naissus brick**Table 5** EDS analysis bricks Naissus%

Element	App Conc.	Intensity Corn.	Weight%	Weight% Sigma	Atomic%
C K	5.18	0.1711	22.79	6.65	32.37
O K	25.81	0.3972	48.90	4.26	52.15
Na K	0.54	0.6015	0.67	0.16	0.50
Mg K	0.85	0.6090	1.05	0.13	0.74
Al K	4.82	0.7261	4.99	0.44	3.16
Si K	13.35	0.7742	12.98	1.13	7.88
Cl K	0.85	0.6707	0.95	0.10	0.46
K K	1.04	0.9284	0.85	0.09	0.37
Ca K	2.76	0.8951	2.32	0.21	0.99
Ti K	0.38	0.7709	0.37	0.06	0.13
Fe K	4.35	0.7932	4.13	0.37	1.26
Totals			100.00		

All the analysis and commentary Figures 15 (a, b, c, d, e) are made based on the published works of foreign researchers and at 200 magnification (Figure 15.a.) and at 500 magnification (Figure 15.b.) and the enlargement of 1000 (Figure 15.c.) and with magnification 2 000 (Figure 15.d., e.) show the flaky structure of quartz [15] [16].

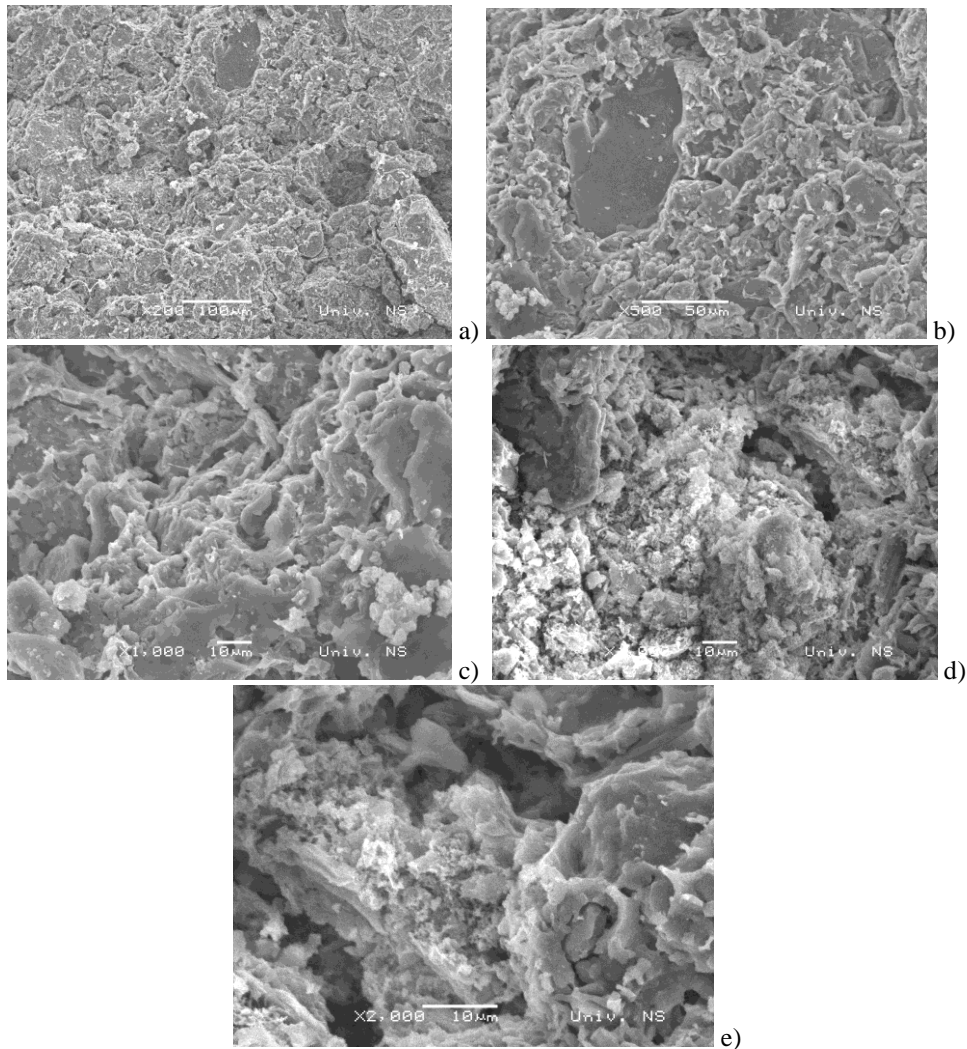


Fig. 15 SEM Naissus brick

3. CONSLUSION

Based on the results, it can be concluded that the bricks have the same exterior, and approximately the same chemical composition. One reason is, most likely, a similar chemical composition of the soil, because the archaeological sites are geographically

close to each other. Another reason could be that the bricks were produced in the same way and that the same fuel in the kilns was used.

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UPOREDNA ANALIZA REZULTATA MIKROSKOPSKIH SNIMAKA I XRF I EDS REZULTATA OPEKA SA ARHEOLOŠKIH LOKALITETA MEDIANA I NAISUS

U ovom radu su opisani arheološki lokaliteti Mediana i Naisus u periodu kasne antike. Prikazani su mikroskopski snimci opeka, kao i rezultati XRF i EDS analiza opeka sa navedenih arheoloških lokaliteta. Opeke su sličnog spoljašnjeg izgleda, ali i približno istog hemijskog sastava. Sam hemijski sastav zemljišta je veoma sličan, ali i način proizvodnje opeke i pretpostavlja se da je korišćeno isto gorivo u pećima za pečenje.

Ključne reči: *Mediana, Naisus, opeka, mikroskopsko snimci, XRF i EDS rezultati, kasna antika*