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NEW ARCHAEOMETRIC CHARACTERISTICS FOR ANCIENT POTTERY IDENTIFICATION

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Abstract:

The paper presents new achaeometric characteristics determined through electron microscopy scanning coupled with energy dispersive X-Ray Spectroscopy and microFTIR. We studied, by comparing average chemical composition and Si/Al and Ca/Mg ratio, a number of 47 genuine antique pottery items (Liniar Pottery, Pre-Cucuteni and Cucuteni culture pottery) and 20 new items made of clay extracted from the neighbourhood of the archaeological sites, with properties and chemical contents similar to those used by antique potters, but considered fakes. Those characteristics were used to differentiate between a fake and an original artifact, to establish authenticity attributes in the patrimonial revaluation and to select the pottery fragments during coherent structural reintegration and restoration processes.

***Keywords**: Neolithic and Chalcolithic pottery, archaeometric characteristics, SEM-EDX, microFTIR, authentication*

Introduction

Generally, the study of pottery items obtained via archaeological excavations focused mainly on the analysis of their shapes and decorations. Pottery products were considered crucial elements in the establishment of the relative chronology and of the technological and cultural level of a given period of time. During the last decades, old pottery artifacts have become the subject of a different kind of approach, that is, a systematical study of the raw materials and the dough from which they were manufactured, in order to determine certain technological, economic and archaeometric traits [1].

Usually in archaeology, according to scientific data [2-7], it is the theoretical and methodological foundations that dictate the manner of an approach, but in most cases one resorts to classical analytical systems which do not allow for relevant evaluations predominantly referring to archaeometrical characteristics [8]. The resistance of pottery to various damaging agents was tested through the simulation of daily handling, then other physical-structural and mechanical attributes, such as the resistance to thermal shock, porosity, color variation, wall thickness, dough type, and grease removers were determined [9-12].

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Currently, the modern methods which operate with instrumental techniques in co-assisted systems, such as, in our case, electron microscopy scanning coupled with dispersed energy spectrometry (EDX) and with FTIR or the corroboration of data obtained by two or more methods, led to high resolution and good reproducibility data.

Experimental archaeology was increasingly involved [9-11] to elucidate the processes which took place during the pottery manufacture, by simulating ancient techniques on doughs with various mixtures.

Concerning this aspect, this paper presents the determination of certain archeological characteristics, obtained by two modern methods: SEM-EDX and microFTIR, by analysis of genuine ancient pottery and modern forgeries.

Experiment data

a. Samples

We studied more than 200 samples, from which we selected 58, the most representative ones, in a good conservation state. We could easily determine on them the degradation and deterioration effects from surface to the inside. The samples were indexed according to the Arheopolice Project database. The analyzed pottery items were Liniar Pottery, Pre-Cucuteni and Cucuteni culture items from The Eastern Carpathian Region (Moldavia).

b. The SEM-EDX Electron-scan Microscopy

The investigation was performed by means of a SEM VEGA II LSH scanning electronic microscope made by TESCAN for the Czech Republic, coupled with an EDX QUANTAX QX2 detector made by ROENTEC Germany. Quantax QX2 is an EDX detector used for qualitative and quantitative micro-analysis in industry, research and education, which performs quantitative measurements without using specific calibration standards. Quantax QX2 uses a 3rd generation X-flash detector, which does not require liquid nitrogen cooling and is about 10 times faster than the traditional Si(Li) detectors.

c. Micro-FT-IR analyses

The spectra were recorded with a FT-IR spectrophotometer coupled to a HYPERION 1000 microscope, both equipments from Bruker Optic, Germany. The FT-IR spectrometer is a TENSOR 27, which is an advanced flexible desktop instrument suitable for routine applications as well as for laboratory research. The TENSOR 27 is designed for measurements mainly in the mid – infrared region.

The standard detector is a DLaTGS detector, which covers a spectral range from 4000 to 600cm^{-1} and operates at room temperature. The resolution is normally 4 cm^{-1} but it can go up to more than 1cm^{-1} (apodized).

Results and discussions

Relying on the weight percent for 39 antique genuine pottery items (table 1) and 19 new ones (table 2), considered counterfeits, determined through EDX data, we studied some new archaeometric characteristics in order to differentiate between an original and a counterfeit artifact.

Considering the average values of the gravimetric concentration for each element, of the ancient pottery composition, the following elements are archaeometrically relevant: Fe, Ca, Na and Ti. The elements O, Si, Al, Mg, K and P do not have an archaeometric evolution.

Among the new archaeometric characteristics, we focused on the Si/Al ratio (caustic modulus), the Ca/Mg ratio (alcalinoterreos modulus) and the K/Na ratio (alcalin modulus); average values of concentration for O, Fe, Ti, P and C.

Certain archaeometric characteristic, as the average concentration values for C, depending on the chemical charge of the soil, with nonchronological variation, or the average concentration value for Fe, whose value depends on the clay content used in pottery, which proved unconvincing as archaeometric data.

As reference, we took gravimetric compositions, as well as some new features based on their assessed value for a range of new pottery items, considered fakes, made of clay from nearby archaeological sites, with properties and compositions similar to those used by ancient potters.

Table 1. Elemental composition of the genuine pottery

Sample	Elemental composition - weight percents (%)										
	O	Si	Al	Fe	Ca	Mg	K	Na	P	Ti	C
01A-L	49,26	24,30	8,22	4,50	7,57	1,51	2,50	0,29	0,14	0,55	1,12
02A-L	51,48	24,61	9,19	4,24	4,25	1,84	2,48	0,61	0,22	0,65	0,39
03A-L	48,03	25,98	9,41	4,32	6,18	1,92	2,61	0,43	0,41	0,67	-
AVERAGE	**49,59**	**24,96**	**8,94**	**4,35**	**6,00**	**1,76**	**2,53**	**0,44**	**0,26**	**0,62**	**0,76**
04A-P	49,36	28,27	9,95	3,86	2,09	1,76	3,03	0,77	0,19	0,71	-
05A-P	35,13	29,63	20,94	5,11	1,97	1,52	2,79	0,75	0,28	0,83	1,01
06A-P	47,86	27,77	9,91	4,96	3,36	1,56	3,14	0,70	0,16	0,54	-
07A-P	53,76	23,90	7,26	3,19	3,86	2,32	2,40	1,56	0,26	-	1,45
08A-P	50,40	20,93	7,13	3,27	1,92	1,27	2,28	1,23	0,17	0,38	10,55
09A-P	52,55	19,76	8,80	5,11	5,90	1,92	2,49	0,43	1,21	0,81	0,99
10A-P	51,79	22,33	10,24	5,37	3,81	1,84	1,68	0,51	0,87	0,70	0,83
11A-P	51,29	21,75	10,70	6,01	3,82	1,61	2,17	0,38	1,56	0,64	0,02
12A-P	53,28	22,31	10,39	5,94	3,08	1,26	2,04	0,58	1,05	-	0,04
13A-P	61,55	14,47	7,98	3,97	5,68	1,79	1,12	0,96	1,41	-	-
14A-P	53,92	14,83	9,96	3,84	6,97	1,77	1,58	0,70	4,20	1,08	1,12
15A-P	60,70	12,15	7,35	4,02	8,91	1,38	1,25	0,10	3,92	-	-
16A-P	50,02	27,64	9,69	5,04	1,54	1,37	3,12	0,50	0,29	0,75	-
17A-P	51,18	23,59	9,33	4,37	5,03	1,80	2,45	0,75	0,26	0,56	0,64
18A-P	57,46	19,11	7,32	4,70	6,15	0,91	2,18	0,23	-	0,44	1,46
19A-P	56,98	19,69	7,09	7,09	4,33	0,79	2,90	0,10	0,34	-	-
AVERAGE	**52,33**	**21,76**	**9,63**	**4,74**	**4,28**	**1,55**	**2,29**	**0,68**	**1,08**	**0,68**	**1,81**
20A-C	46,86	24,84	9,06	4,31	9,11	2,18	2,56	1,06	-	-	-
21A-C	45,85	25,53	8,91	4,22	8,02	2,56	2,85	1,04	0,36	0,61	-
22A-C	48,49	25,86	9,13	5,15	4,61	2,22	2,67	1,17	-	0,67	-
23A-C	50,51	23,48	10,25	5,97	2,11	2,50	2,58	2,15	0,28	0,45	-
24A-C	58,23	19,32	9,26	6,31	1,76	1,34	1,82	1,01	0,13	0,44	0,21
25A-C	50,58	22,97	8,08	4,29	7,77	2,15	2,47	0,87	-	0,70	0,09
26A-C	49,87	23,57	8,50	5,13	5,38	1,98	3,22	1,02	-	0,66	0,65
27A-C	49,90	26,26	9,40	6,01	1,46	1,50	2,82	1,45	0,37	0,80	-
28A-C	51,10	23,26	8,66	4,51	5,39	1,62	3,34	1,09	-	1,00	-
29A-C	50,78	22,83	7,76	5,13	6,07	1,71	3,16	1,13	-	0,72	0,69
30A-C	49,31	25,58	9,66	5,15	3,68	2,31	2,16	0,42	1,06	0,62	-
31A-C	50,41	20,47	9,47	6,51	3,99	0,74	2,05	0,40	4,39	0,65	0,89
32A-C	56,48	19,61	9,35	11,19	0,29	0,47	1,23	0,03	0,01	1,32	-
AVERAGE	**50,6**	**23,35**	**9,04**	**5,68**	**4,59**	**1,79**	**2,53**	**0,99**	**0,94**	**0,72**	**0,51**
33A-H	55,09	25,68	9,16	3,49	1,60	1,74	1,99	0,45	0,28	0,48	0,001
34A-C1	56,30	11,80	7,69	4,67	5,13	0,88	1,41	0,37	4,64	0,60	6,45
35A-C1	55,36	23,63	7,73	3,85	1,66	1,09	1,81	0,69	1,07	0,54	2,54
36A-M	57,30	15,20	10,08	4,02	4,00	0,77	1,66	0,54	5,74	0,66	-
37A-C2	51,11	27,94	9,72	4,72	1,44	1,26	2,71	2,71	0,19	0,52	-
38A-S	50,60	26,82	9,17	3,71	4,10	1,52	2,57	0,57	0,27	0,62	-
39A-S	49,66	27,95	8,93	4,52	2,28	1,68	2,68	0,95	0,84	-	-
AVERAGE	**53,63**	**22,72**	**8,93**	**4,14**	**2,89**	**1,28**	**2,12**	**0,90**	**1,86**	**0,57**	**3,00**

L: LBK (Linear Band Keramik) – Late Neolithic (6.000-5.500 BC)
P: Pre-Cucuteni – Early Chalcolithic (5.500-5000 BC)
C: Cucuteni – Middle Chalcolithic (5.000/4.700-3.500/3.000 BC)
H: Horodistea – Late Chalcolithic (3.000-2.500 BC)
C1: Costisa – Middle Bronze Age (1.750-1.400 BC)
M: Monteoru – Middle Bronze Age (1.750-1.400 BC)
C2: Cozia – Hallstatt A (1.100-800 BC)
S: Santana de Mures – IVth Century AD

Table 2. Elemental composition of the fake pottery

Sample	Elemental composition - weight percents (%)										
	O	Si	Al	Fe	Ca	Mg	K	Na	P	Ti	C
01F-P	46,38	26,37	7,88	4,32	8,51	1,97	2,99	0,38	0,11	0,79	0,24
02F-P	60,16	17,81	8,05	2,79	3,37	2,79	1,82	1,59	0,15	0,31	1,13
03F-P	57,80	20,04	7,52	3,16	3,26	2,67	1,57	1,42	0,24	0,61	1,67
04F-P	56,92	20,41	9,57	2,35	2,48	3,19	1,72	2,29	0,42	0,39	0,24
05F-P	49,43	29,73	7,27	4,27	3,85	2,02	2,83	0,59	-	-	-
AVERAGE	**54,14**	**22,87**	**8,06**	**3,38**	**4,29**	**2,53**	**2,19**	**1,25**	**0,23**	**0,53**	**0,82**
06F-C	51,75	24,86	8,55	3,98	4,87	2,14	2,36	0,72	0,19	0,54	-
07F-C	52,41	24,85	7,87	3,87	5,61	1,68	2,60	0,48	-	0,60	-
08F-C	50,61	25,82	7,54	4,33	5,34	1,73	2,85	0,45	-	0,89	0,41
09F-C	45,89	25,73	9,53	4,97	6,14	2,99	3,04	1,03	-	0,65	-
10F-C	46,95	22,83	7,28	5,36	9,66	3,14	3,29	0,57	0,04	0,84	-
11F-C	43,09	29,17	8,71	4,97	6,97	2,74	3,06	0,59	0,13	0,54	-
12F-C	44,59	28,97	8,98	4,74	5,72	2,77	2,74	0,65	0,10	0,53	-

13F-C	48,23	30,44	7,68	3,97	3,56	1,95	2,72	0,42	0,23	0,58	0,18
14F-C	52,10	24,23	7,67	4,19	6,35	1,66	2,43	0,54	0,13	0,57	0,09
15F-C	48,80	25,80	8,12	4,46	6,45	2,03	3,06	0,55	0,07	0,62	-
16F-C	53,80	24,35	7,41	4,19	5,18	1,46	2,17	0,46	0,06	0,61	0,20
17F-C	51,52	23,78	7,87	3,33	5,08	2,42	2,36	0,94	0,14	0,62	1,78
18F-C	49,86	25,33	8,28	4,76	5,95	1,69	2,73	0,50	0,17	0,56	0,12
19F-C	46,84	26,85	8,74	4,42	5,99	2,80	2,87	0,59	0,24	0,61	-
20F-C	49,86	25,33	8,28	4,76	5,95	1,69	2,73	0,50	0,17	0,56	0,12
19F-C	46,84	26,85	8,74	4,42	5,99	2,80	2,87	0,59	0,24	0,61	-
AVERAGE	48,95	25,95	8,20	4,42	5,93	2,23	2,74	0,60	0,15	0,62	0,41

P: Pre-Cucuteni fake pottery; C: Cucuteni fake pottery

In table 3 and table 4 the Si/Al, Ca/Mg and K/Na ratios for ancient and respectively for fake pottery are presented.

Table 3. Si/Al, Ca/Mg and K/Na ratios for ancient pottery

Sample	Si/Al	Ca/Mg	K/Na	Sample	Si/Al	Ca/Mg	K/Na
01A-L	2,96	5,01	8,62	**20A-C**	2,74	4,18	2,42
02A-L	2,68	2,31	4,07	**21A-C**	2,87	3,13	2,74
03A-L	2,76	3,22	6,07	**22A-C**	2,83	2,08	2,28
Average	**2,79**	**3,41**	**5,75**	**23A-C**	2,29	0,84	1,20
04A-P	2,84	1,19	3,94	**24A-C**	2,09	1,31	1,80
05A-P	1,41	1,30	3,72	**25A-C**	2,84	3,61	2,84
06A-P	2,80	2,15	4,49	**26A-C**	2,77	2,72	3,16
07A-P	3,29	1,66	1,54	**27A-C**	2,79	0,97	1,94
08A-P	2,94	1,51	1,85	**28A-C**	2,69	3,33	3,06
09A-P	2,25	3,07	5,79	**29A-C**	2,94	3,55	2,80
10A-P	2,18	2,07	3,29	**30A-C**	2,65	1,59	5,14
11A-P	2,03	2,37	5,71	**31A-C**	2,16	5,39	5,13
12A-P	2,15	2,44	3,52	**32A-C**	2,10	0,62	41,00
13A-P	1,81	3,17	1,17	**Average**	**2,58**	**2,56**	**2,56**
14A-P	1,49	3,94	2,26	**33A-H**	2,80	0,92	4,42
15A-P	1,65	6,46	12,50	**34A-C1**	1,53	5,83	3,81
16A-P	2,85	1,12	6,24	**35A-C1**	3,06	1,52	2,62
17A-P	2,53	2,79	3,27	**36A-M**	1,51	5,19	3,07
18A-P	2,61	6,76	9,48	**37A-C2**	2,87	1,14	1,00
19A-P	2,78	5,48	29,00	**38A-S**	2,92	2,70	4,51
Average	**2,26**	**2,76**	**3,37**	**39A-S**	3,13	1,36	2,82
				Average	**2,54**	**2,26**	**2,36**

L: LBK (Linear Band Keramik)
P: Pre-Cucuteni
C: Cucuteni
H: Horodistea
C1: Costisa
M: Monteoru
C2: Cozia
S: Santana de Mures

Table 4. Si/Al, Ca/Mg and K/Na ratios for fake pottery

Sample	Si/Al	Ca/Mg	K/Na
01F-P	3,35	4,32	7,87
02F-P	2,21	1,21	1,14
03F-P	2,66	1,22	1,11
04F-P	2,13	0,78	0,75
05F-P	4,09	1,91	4,80
Average	**2,84**	**1,70**	**1,75**
06F-C	2,91	2,28	3,28
07F-C	3,16	3,34	5,42
08F-C	3,42	3,09	6,33
09F-C	2,70	2,05	2,95
10F-C	3,14	3,08	5,77
11F-C	3,35	2,54	5,19
12F-C	3,23	2,06	4,22
13F-C	3,96	1,83	6,48
14F-C	3,16	3,83	4,50
15F-C	3,18	3,18	5,56
16F-C	3,29	3,55	4,72
17F-C	3,02	2,10	2,51
18F-C	3,06	3,52	5,46
19F-C	3,07	2,14	4,86
20F-C	3,06	3,52	5,46
19F-C	3,07	2,14	4,86
Average	**3,16**	**2,66**	**4,58**

P: Pre-Cucuteni fake pottery
C: Cucuteni fake pottery

Regarding the Si/Al ratio for ancient pottery evaluated from weight percent (gravimetric concentrations), it varies between 1.5 and 3.0, except for some values (min and max), not included in the Gaussian distribution (deviations). It does not have an archaeometric evolution.

As for the counterfeit pottery, the Si/Al ratio determined varies between 3.0 and 4.0, not including the values, which are deviations. The average Si/Al ratio for ancient pottery is 2.54 and for fake pottery is 2,44, the difference being inconclusive.

The Ca/Mg ratio for antique pottery, ranges from 1 to 6, and the average values have an archaeometric evolution, decreasing in time from 3.4 to 2.3. For the fake pottery this ratio varies between 1.5 to 3.5, not including the deviated values. The average value is under 2.3. This ratio can be used in differentiating a fake from an original.

The K/Na ratio for ancient pottery has an archaeometric value that decreases from 5.75 to 2.35, the ratio with the most heterogeneous values. Concerning the fake pottery, this ratio is related to the clay, ranging from 1.75 to 4,58.

In this regard, other variable archaeometric features were searched, allowing a clear distinction between the two types of pottery: authentic and fake. Thus, two groups were evaluated by their average concentrations of chemical elements for all pottery items, making the difference between genuine and forgery. The minimal and maximal values were not included in the Gaussian distribution (deviations).

The average values, clearly differentiating between antiques pottery and forgeries were the average concentration of oxygen, silicon, aluminum, iron, magnesium and phosphor.

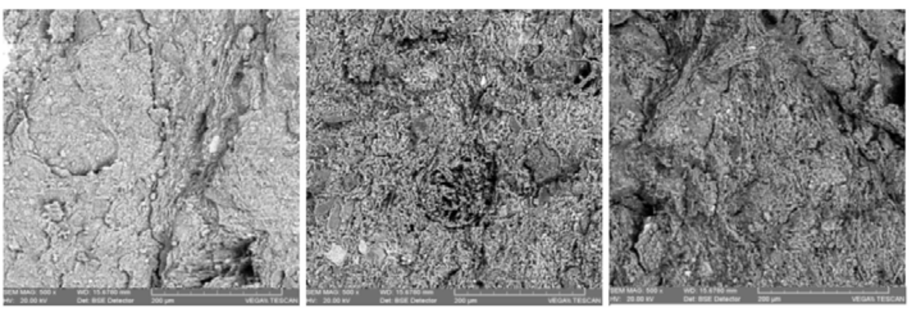
In the original pottery oxygen and silicon have lower values and the other elements (Al, Fe, Mg, P) have higher values than in the fake pottery.

From the average concentrations, from which deviations were excluded, the following values were noted:

- For oxygen, the Pre-Cucuteni pottery the average value is 52.33 and for the fake ones it is 54.14 - a difference of 1.81 percent. For the Cucuteni pottery the average value is 50.60 for the original and 48.95 for the fake ones - a difference of 1.65 percent. Oxygen is not relevant, because in pottery enrichment in O may occur, because of the development in time of stable compounds (hydrogels, cristalohydrates and acvo complexes). On the other side, the different burning of the ancient pottery allowed some metallic cations to be in a superior or inferior oxidation state (ie Ti(II and IV) and Fe(II and III)).
- For silicon and magnesium is obvious that the concentration is higher on fake pottery;
- The aluminum, iron and phosphor concentrations is lower by one percent in fake pottery;

In order to identify the fake pottery from the original ones, next to the composition data, very important is the microscopic data regarding granulometry and the homogeneity.

In Figure 1 SEM images of some representative pottery with incomplete burning from the Pre-Cucuteni, Cucuteni and Linear pottery cultures are presented.

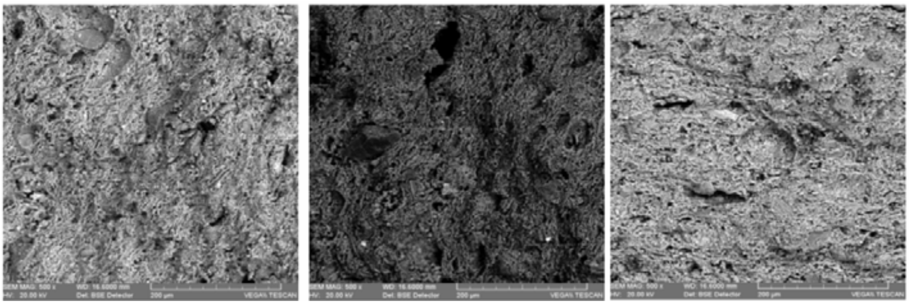


a b c

Fig. 1. SEM image of old pottery with incomplete burning 500X:
a – Cucuteni, index no. 27A-C; b – Liniar pottery, index no. 01A-L;
c – Pre-Cucuteni, index no. 11A-P;

The Cucuteni pottery (Fig. 1.a) pug (clay paste) is not homogenous, with longitudinal late cracks. The Liniar pottery (Fig. 1.b) is considered a medium one, homogenous with a low burning temperature with limited cracks, as a mosaic. The Pre-Cucuteni pottery (Fig. 1c.) is a medium one with grains of SiO_2, non-homogenous with longitudinal late cracks.

In Figure 2, SEM images of some representative pottery with complete burning from the Cucuteni culture are presented.

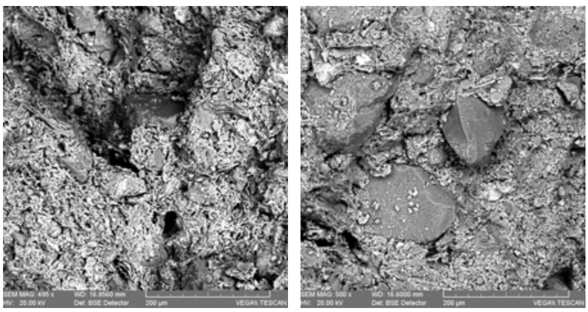


a b c

Fig. 2. SEM image of old pottery with complete burning, 500X:
a – Cucuteni, index no. 25A-C, b – Cucuteni, index no. 21A-C, c – Cucuteni, index no. 26A-C.

The Cucuteni pottery (Fig.2.a.) is a fine one, characterized by a homogenous pug, uniform granulometry and primary burning. The second Cucuteni pottery (Fig.2.b) is thick, non-uniform with small late cracks. The third Cucuteni pottery (Fig.2.c) is a fine one, with homogenous and uniform granulometry.

Figure 3 presents two fake pottery samples. It can be well observed that the pug is not well mixed, as it is non-homogenous and presents large grains of SiO_2.



a **b**

Fig. 3. SEM Image of fake pottery, BSE 500X: a – index no. 12F-C; b – index no. 13F-C

These two are very different in morphology, the ancient pottery being easy to identify because of the presence of some formations with homogenous structure in a non-omogenous matrix.

In the Figure 4 the microFTIR spectrums for the all 8 pottery samples are presented.

The first three (11A-P, 27A-C and 01A-L) are the ones with incomplete burning, which while they stayed in soil, preserved their water (zeolitic, coordinative and crystallization) from 3200…3800 cm^{-1} and the hydrogels from 1400…1500 cm^{-1}.

The well burned pottery, from the second group (21A-C, 25A-C and 26A-C), the peaks values for water and hydrogels are lower and they have in common a peak value at 2500 cm^{-1}, which can also be found in fake pottery.

The curves for the fake pottery (12F-C, 13F-C) are very different from those of the genuine pottery according to picks from 1200 cm^{-1}, 2500 cm^{-1} and 3700 cm^{-1}.

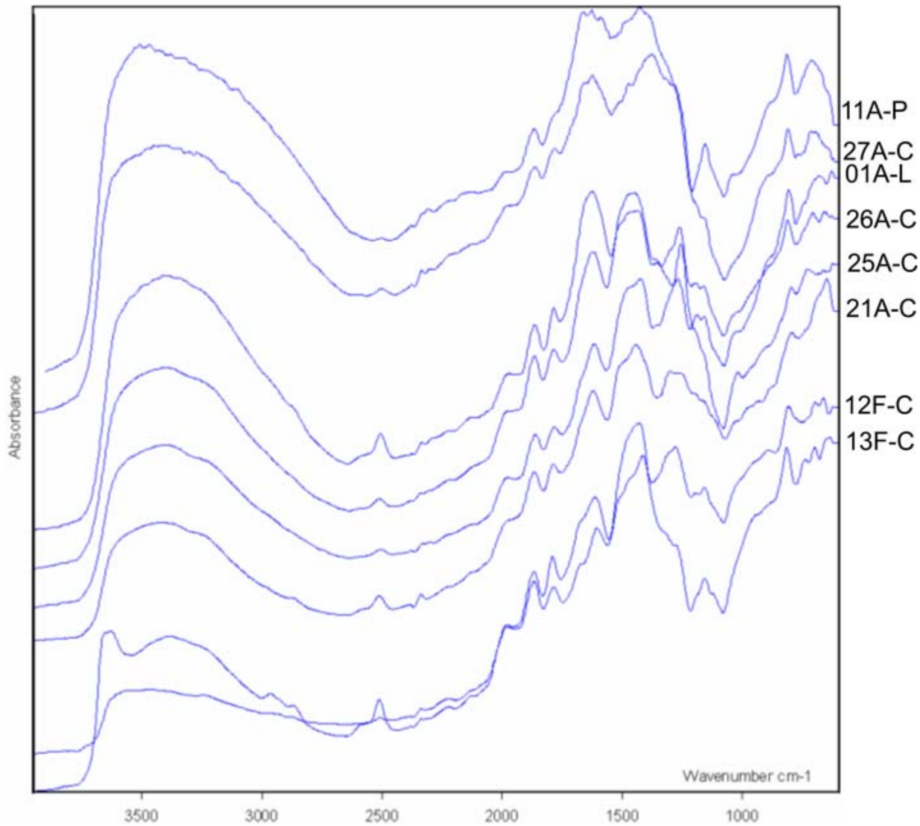


Fig. 4. The micro-FTIR spectra of the pottery samples

Conclusions

In conclusion, methods involving SEM-EDX and micro-FTIR spectroscopy made it possible to evaluate the gravimetric concentration and to determine the potentially relevant archaeometric characteristics. Out of these, the best evidence for the difference between original and counterfeit, the Si/Al (caustic module), the Ca/Mg (alkaline-earth module) and the K/Na (alkaline module) ratios have a positive archaeometric evolution.

Regarding composition, the average values for silicon and magnesium are higher in fake pottery and the values for aluminum, iron and phosphor are lower in the fake pottery.

SEM-EDX and microFTIR analysis uncovered some structural and chemical characteristics which are different in ancient pottery than in fake, artificially aged pottery.

Thus, by SEM-EDX, for identification we used the grain size, homogeneity and the uniform distribution of the structural formations.

In the microFTIR analysis we evaluated the group characteristic vibrations for different types of water, hydrogels and other elements.

The fake pottery presented large size grains and mineral formations non-uniformly distributed and fine sharp edges. Regarding their composition, a lower concentration of oxygen and titanium and an increased percentage of silicon were obvious.

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