Archaeometric and Chemometric Studies Involved in the Authentication of Old Heritage Artefacts

I. Contributions of the Iasi school of Conservation Science

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The paper presents the results obtained in the study of new archaeometric and chemometric characteristics involved in the authentication of old artefacts made of metal, ceramics and polychrome wood, by the Iasi school of Conservation Science of cultural heritage goods. Most of the metal and ceramic artefacts considered for this study originate from archaeological sites from the region of Moldavia and Dobrudja, and those of polychrome wood from state or private collections and monastic establishments. The data obtained, using modern experimental methods and techniques, has allowed us to set the fundaments of phenomenological aspects related to the conception/execution, acquired patina, evolutive effects of deterioration and degradation, historical contexts, etc. These discoveries have been acknowledged as interdisciplinary effects, as evidenced by the citations listed in scientometric databases.

Keywords: authentication, cultural heritage goods, arhaeometrics study, chemometrics study, scientometrics databases, scientific impact, multidisciplinary aproach

Currently, the research activity in the field of *environmental science and engineering* comprises a series of very attractive and important directions, concerning the integrated and sustainable conservation of historical-cultural and natural heritage goods, the protection and conservation of biodiversity, subordinated to the *Science of Conservation* [1-9].

These directions have allowed for a better conjoint approach by, on the one hand, scientific–theoretical/ fundamental research, and, on the other, the technological-applicative one, creating a modern system for integrating the research–development–innovation activities.

In this sense, we present the results obtained by the research-development-innovation activities in the field of *Conservation Science*, as reflected in the specialized literature, tackling the following issues: the *authentication* of works of art that are less known or have been recently acquired/discovered in archaeological sites, *heritage classing* and *assessing* (auction-house or catalogue price), determining the *state of conservation* (establishing the evolutive deterioration and degradation effects, by elucidating the mechanisms of the destruction and alteration processes), elaborating the *preservation* and *restoration* strategy (*elaborating and carrying out studies for making compatible the preservation and restorations interventions, monitoring the behavior of the interventions for a set time period, and the permanent monitoring of the evolution of the conservation state*), identifying and *using the optimal systems for valorizing* (in museums, tourism, virtual display)/*treasuring* [1-9].

The *authentication* of ancient and historical (from after 1700 AD) artefacts, besides dating and ascertaining the author/school, is concerned with a series of attributes related to the uniqueness, conception, patina, price, owner/ custodian, historical contexts, etc. [10].

Nowadays, this is achieved using modern methods that emply systems of co-assistance and, respectively, corroboration of interdisciplinary techniques. Most of the instrumental methods are assisted by specialized software programs, for processing the images and the experimental data, as well as by complex methods designed from coupled or tandem techniques, for instance Scanning Electron Microscopy (SEM), coupled by X-ray Spectrometry (EDX), or Pyrolytic Gas-chromatography with Silylation, coupled by Mass Spectrometry, etc. [10].

Specific methods that allow obtaining data with manifold implications, for accomplishing various expertization goals, are selected in the experimental protocol.

In general, according to the ethical principles of conservation, the non-destructive methods are the first to be resorted to, which are employed directly on the artefact (that is to say, *without collecting samples*). In the case in which the expertization requires investigating the structures from the volume phase of the artefact, para-destructive, non-invasive techniques will be used, which involve collecting micro-samples from the artefact, with the provision that they should not affect the appearance of the artefact (from the socle, edges, moving craquelures, etc.) [8-10].

From the group of non-destructive methods, we mention: digital photofixation; direct observation using magnification tools; UV, Vis and IR reflectography; reflexive colorimetry (CIE L*a*b*); laser IR thermography; 3D profilometry; X-ray fluorescence; histochemistry on micro-surfaces; microendoscopy in fissures, crevasses, cavities, craquelures (particularly those *raised in a roof*); X-ray and γ-ray radiography; other methods of nondestructive defectoscopy; etc. [1-10].

From among the methods involving sampling, often used for determining the inner structures, we mention the para-

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destructive or micro-analytical ones: optical and electronic microscopy of thin slices (by transmission) and in stratigraphic sections (by reflection); X-ray roentgenography (on mono-crystals or on powders); chemical analyses in solutions, melting, gaseous and solid state (which requires disaggregation and dilutions in water-based or organic solutions); separatology in thin layers (paper or plate chromatography), in columns, or liquid chromatography and gas-chromatography; UV, ViS and IR spectroscopy; Raman and Mössbauer spectroscopy; electron spin spectroscopy; nuclear magnetic resonance; relative or absolute dating methods (dendrochronology, thermoluminescence, obsidian hydration, C14, isotopic dilution, isotopic exchange/potassium-argon 40, rare element/microelement determination, and then evaluating the chemometric markers with archaeometric value, then the stratigraphic, typological, comparative, cartographic, valve, palynological, archaeometric, etc. methods) [10].

Using these methods, it is possible to determine the elemental or phasal composition, the morphology of the microcrystalites and their distribution in the volume phase, at the interfaces or on the surface, the nanostructure of the molecules, the microstructure of the crystallites and the phasal systems, the stratigraphic microstructure, the profile of the structures and of the surface and stratigraphic structural components, etc. Likewise, it allows identifying certain archaeometric (evolving) characteristics, or assessing certain chemometric characteristics with archaeometric function.

The authentication is made on the basis of the *archaeometric characteristics* (chemical composition and structure/distribution of chemical components, the physical-structural morphology of the granules/crystallites/phases and their arrangement in the volume phase of the sample, macrostructural characteristics/specific dimension, density or specific weight, viscosity, etc.), but also of *chemometric* ones, with archaeometric function.

To this purpose, the paper presents the archaeometric and chemometric characteristics established for various types of old artefacts, grouped along the base materials into (1) metals and alloys, (2) ceramics and (3) polychrome wood, which allow authentication, establishing the conservation state, and assessing certain attributes related to the *conception*, *acquired patina*, *historical context*, etc. The work was carried out in the Laboratory of Scientific Investigation and Conservation of Cultural Heritage Goods of the *ARHEOINVEST* Interdisciplinary Research and Training Platform from the Alexandru Ioan Cuza University of Iasi.

Bronze archaeological artefacts

In the structure of the ***archaeological patina*** of the ancient bronze items, there were differentiated *three groups of corrosion end-products*, revealed by microscopic and mineralogical analysis on the surface and in the stratigraphy of the crusts with metallic cores or of the coreless bulks of newly-discovered artefacts, and which are used in *authentication* for determining certain attributes related to the historical contexts they witnessed.

The scientific publications of the Iasi school of Conservation Science [11-31] present different casuistics that highlight, distinctly, the compounds originating from the *primary patina* — the so-called *noble patina*, formed in most cases during the artefact's manufacture and uselife phases through *redox processes (oxides, sulphides, etc.)* -, followed by the *secondary patina* - the so-called *vile patina*, resulting from *acidic-basic processes*, *of complexation, ionic exchange*, and *hydrolysis* (oxyhydroxides, halogens, carbonates, sulfates, phosphates, etc.), formed during the concluding stage of the uselife and the first stage after discardment —, and, lastly, the *tertiary patina* - the contamination patina, formed in the archaeological site, under the influence of *pedological processes* (segregation, diffusion, osmosis, electro-osmosis, hydration/dehydration, fouling, mineralization, monolithization, etc.). The corrosion end-products of the three patinas were identified in items originating from both disturbed and undisturbed sites.

The works in which the three types of patinas are discussed, have been cited by a vast number of authors [32-58].

Figures 1 and 2 present the congruent superstructure (the Liesegang effect) in a bronze coin discovered in the archaeological site from Nufarul (Tulcea County, Romania), in which the three distinct patinas are conspicuous.

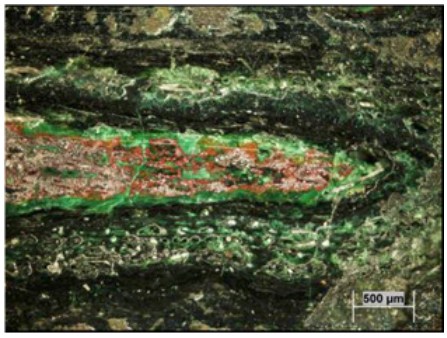

Fig. 1. Cross-section micro-photography (50×) of a bronze coin discovered in the archaeological site from Nufarul, Tulcea County [30, 31].

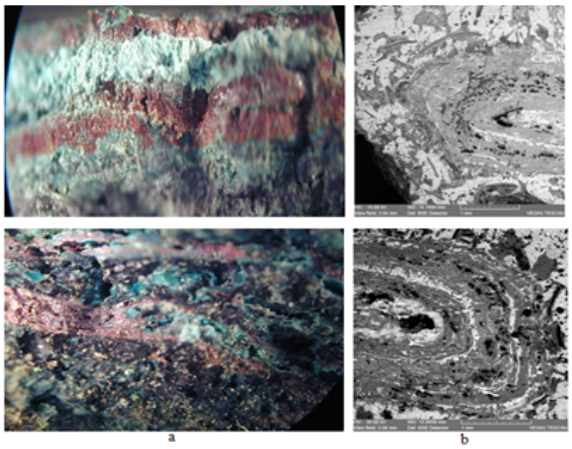

Fig. 2. Details in the stratigraphic section of the bronze coin discovered in the archaeological site from Nufarul, Tulcea County, Romania (fig.1) [30, 31]:a. images from the optical microscope (50×); b – SEM imagery (100×)

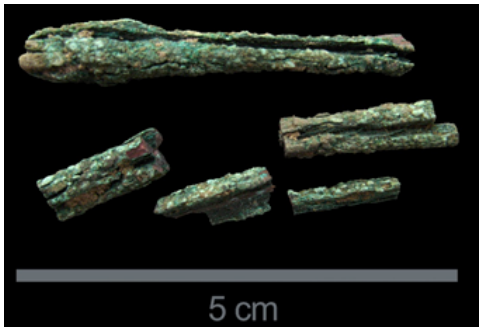

Fig. 3. Fragments from the fibula pinhead discovered at Ibida (Tulcea County, Romania) [11, 12]

Another novel aspect, discovered in the case of an ancient fibula pinhead made from a copper-based alloy, which presents *deep longitudinal craquelures* (fig. 3) resulting from the *contraction from siccativation* (loss of

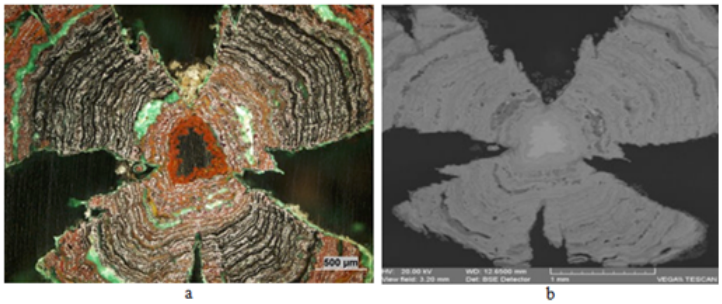

Fig. 4. Details in stratigraphic section of the bronze fibula pinhead discovered at Ibida [11-12]: a. images from the optical microspoce (300×); b – SEM imagery (300×)

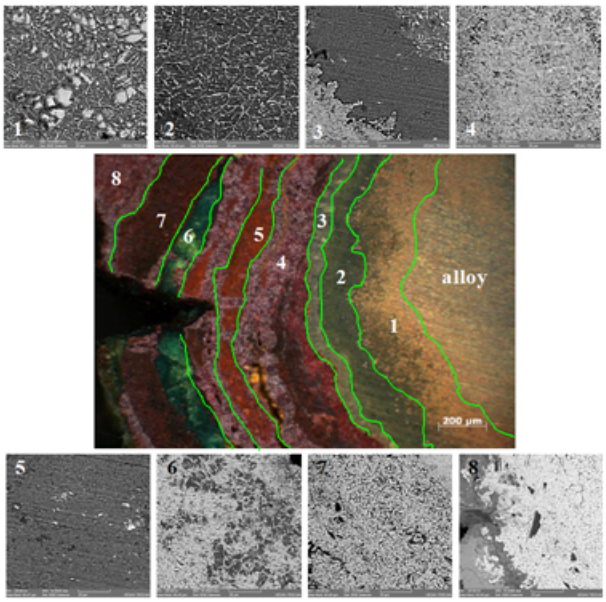

Fig. 5. The SEM stratigraphic microphotogram, with the delineation of the eight Liesegang rings analyzed by EDX [11, 12]

No. of the Liesegang ring	0	1	2	3	4	5	6	8
Ratio of Cu/Sn atomic percentages	10.00	0.65	2.63	9.09	5.26	100.00	4.17	33.33

Table 1
VARIATION IN THE RATIO BETWEEN THE Cu/Sn CONCENTRATION IN THE EIGHT LIESEGANG RINGS FROM FIGURE 2 [11, 12]

crystallization water and from aqua-complexes), is likewise linked to the *Liesegang effect*, which has continuity and preserves its concentric rings of corrosion congruents in the four craquelated caps (fig. 4).

The SEM-EDX analysis determined the variation of the nature of the congruents from each Liesegang ring, in relation to the metallic core (fig. 5 and table 1).

Particular attention was given to researches related to the elucidation of the mechanism by which the *Liesegang effect* occurs during the underground lying period in archaeological sites. They have shown that this effect is due to the formation in certain environmental conditions (humidity, temperature, oxygen concentration, *p*H, etc.) of membrane systems at the surface of the primary (*noble*) patina, which in the presence of the anion chloride and oxygen from the ground produces a congruent super-structuring of the compounds from the secondary (*vile*) patina.

Thus, two types of membrane systems were confirmed microscopically in cross-section:

- continuous and uniform membranes from continuous and uniform membranes from *hydrogels* of Sn(IV), Pb(IV) and Zn(II), which allow the concentric stratification of the congruents based on Cu(II), more or less unpurified with Sn(II) chlorides, followed by layers of malachite, nantokite, atacamite/paratacamite, brochantite, etc. (fig. 6);

-diffusive porous membranes from chloro- or hydroxyapatite (in saline mediums, weakly alkaline, and in the presence of the ion phosphate), which initially forms similar, though discontinuous superstructures, while in certain situations through inverted osmosis occurs the destructuring through dissolution in the fluidic soil water, preserving the membrane system in the form of a stratified microporous honeycomb (fig. 7,8).

Elucidating the mechanism by which the Liesegang effect forms in the lying period was previously presented in two monograph works [11, 12] and in a series of papers published in established journals [16-31].

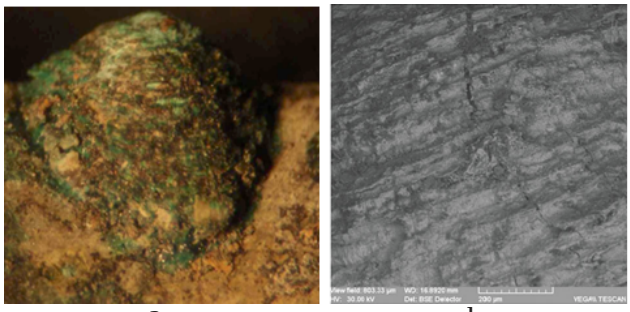

Fig. 6. The structural discontinuity on the surface of a bulk with Liesegang effect, with concentric distribution formed in the presence of hydrogels of Sn(IV) [22, 23]: a – stereomicroscopic image (50×), b – SEM image (300×)

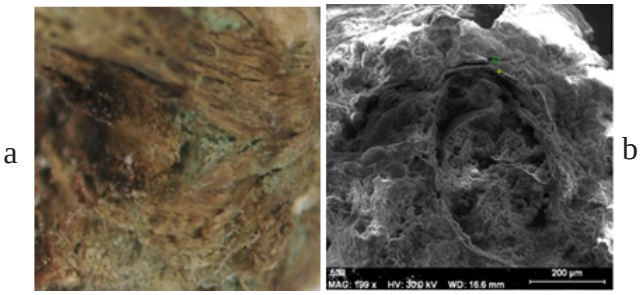

Fig. 7. Discontinuity of the chloro- and hydroxyapatite superstructures in stratigraphic section of the Liesegang effect, with distribution perturbed by the aggressiveness of the lying soil [22, 23]: a – membrane system in the form of a honeycomb, free of congruents after the dissolution with underground water; b - the SEM image of the Liesegang stratifying perturbed in the lying period (200×)

The majority of the published works referring to the elucidation of the mechanism by which the Liesegang effect forms have been cited in many ISI-ranked scientific journals, presented at the end of the reference list [32-58], many citing two to up to four of the papers by our team.

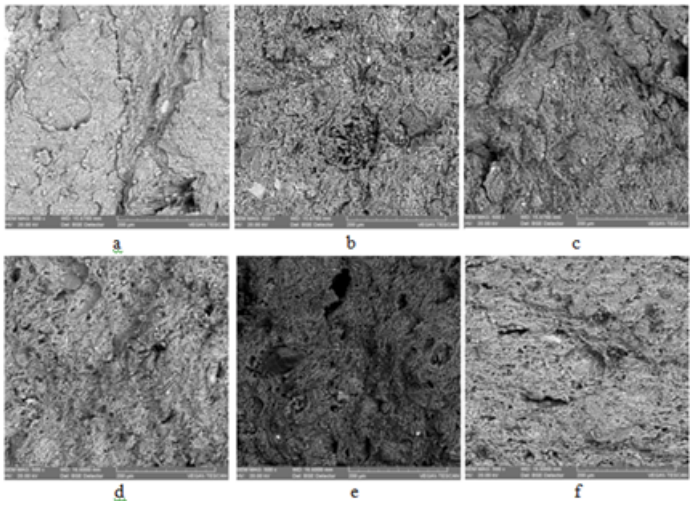

Fig. 8. SEM stratigraphic images of Cucutenian ceramics [61]: a – Cucuteni A, b – Cucuteni B, c – Cucuteni AB. d – Cucuteni; e – Linear Cucuteni; f – Precucuteni

Ceramics	Si/Al	Ca/Mg	K/Na	Ceramics	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
Media	**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	**Media**	**2.58**	**2.56**	**256**
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
Media	**2.26**	**2.76**	**3.37**	**39A-S**	3.13	1.36	2.82
				Media	**2.54**	**2.26**	**2.36**

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

Table 2
THE Si/Al, Ca/Mg AND K/Na CHEMOMETRIC RATIOS OF ARCHAEOMETRIC ASSESSMENT OF ANCIENT CUCUTENIAN CERAMICS [61]

Chemometric characteristics with archaeometric value for ceramic artefacts

With respect to the identification of the archaeometric characteristics, highlighted in ancient ceramics and used for authentication [59-65], our team has investigated the *evolutive modules* Si/Al, Ca/Mg, and K/Na, which were shown to be excellent *chemometric relations* (fig. 8 and tables 2 and 3), then the *rate of ceram solubilisation* (aluminosilicates) under the influence of alkaline carbonates and phosphates (figs. 9–11); changes in the *granulometric distribution* across time; the presence of *manufacturing inclusions*; the type of *temper*, *slip* or *glazing*; the *temperature*, *time and type of firing* at manufacture; the *nature and structure of the superficial crust* formed during underground lying; etc.).

The results concerning the involvement of archaeometry and chemometry in authenticating ancient ceramics published by our team have been cited in a series of specialized scientific works [66-74].

Chemometric characteristics with archaeometric function, for artefacts of polychrome wood

In the case of old easel paintings on wooden mediums, a series of archaeometric characteristics were ascertained, according to the type of component materials [10, 75-103].

Thus, for the wooden mediums (panels, chassis, frames, and casings), on the basis of the *normal domain of variation of the hydric equilibrium*, two archaeometric characteristics have been ascertained: the *duration and critical point of correlation of the hydric equilibrium* (the intersection of the adsorption-desorption curves of the hygroscopic water, respectively the RMC = f(t) curves, with the limits of the domain of variation between the maximum value RMC = ΔEMC and the minimum hypothetical one RMC = 0 - figure 12 and tables 4 and 5) [76, 77]; the *chemometric reports of wood contraction* (tables 6–8) along the three directions: L (longitudinal), R (radial) and T (tangential); the *remanent concentration in crystalline cellulose*; the *remanent concentration in volatile compounds*; the *concentration in ash*; etc. [10, 77-84].

Table 3

THE Si/Al, Ca/Mg AND K/Na CHEMOMETRIC RATIOS OF ARCHAEOMETRIC ASSESSMENT OF COUNTERFEIT CUCUTENI CERAMICS [61]

Ceramics	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
Media	**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: Counterfeit Precucuteni ceramics
C: Counterfeit Cucuteni ceramics

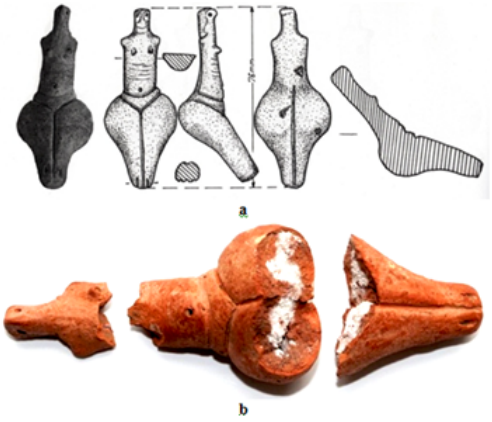

Fig. 9. Anthropomorphic statue no. 6 from the Cucutenian cultic complex found in Isaiia (Iasi County, Romania): a – original photograph and drawings by the authors of the discovery; b – the broken statuette, with deposits of structurally reformed carbonates and inner retracement of the aluminosilicates [65]

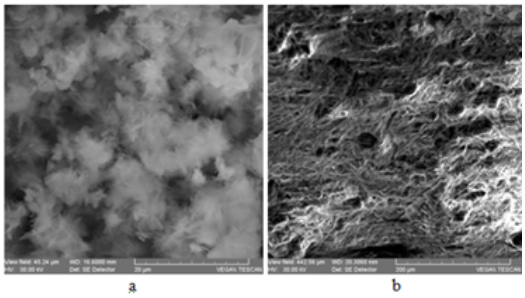

Fig. 10. The SEM microphotography of the acicular Aragonite microcrystalites, structurally reformed from calcite and the alkaline solubilisation of the ceramic matrix of aluminosilicates [65]

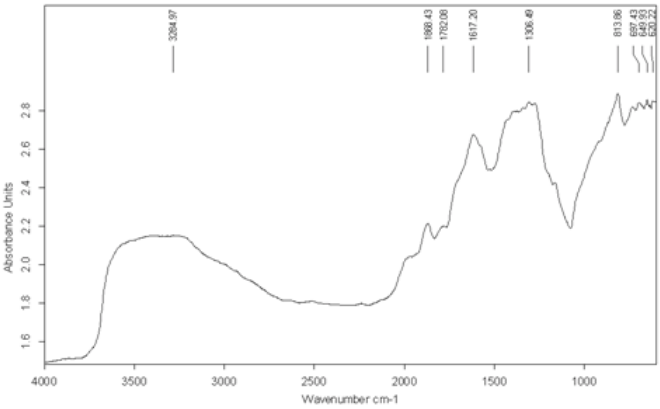

Fig. 11. The FTIR specter, which highlights the presence of aragonite over remanent calcite through the peak from 1306 cm^{-1}, shifted and more pronounced that the peaks of calcite from ca. 1400 cm^{-1}[65]

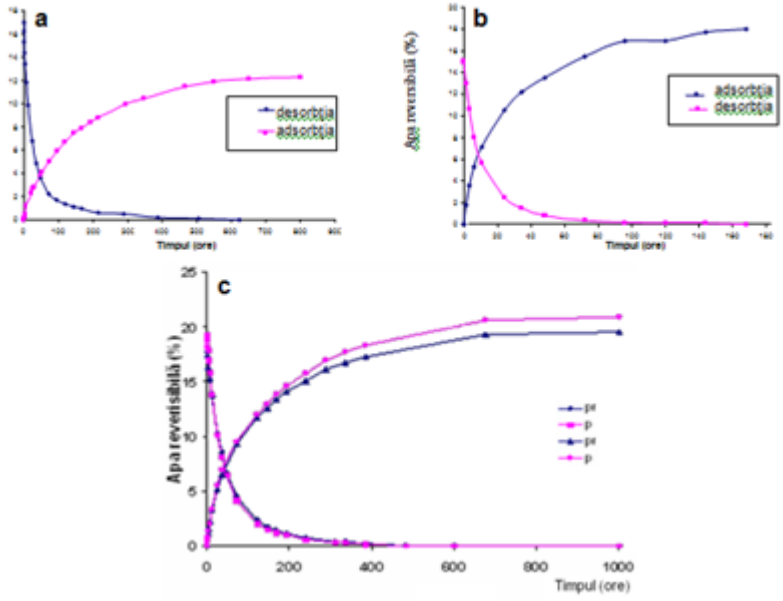

Fig. 12. The hydration-dehydration curves involved in evaluating the normal domain of variation of the hydric equilibrium for linden wood: a. new, b. old, c. treated with red petroleum and propolis [76, 77]

The dendrochronological characteristics of the two wooden samples are presented in Table 5.

The chemometric reports of wood contraction along the three directions: L (longitudinal), R (radial) and T (tangential) were found to be very important archaeometric characteristics, as their variation depends on the wood's species and age, the age of the tree, the chopping area, the period and geographical area of collecting, treatment and processing during manufacture, etc. [77-78].

Tables 6, 7 and 8 present the data on wood contraction (%) for four species (linden, poplar, fir, and oak) along the three directions (L, R and T), and the values of the chemometric characteristics [78].

The results of the chemometric investigations, which showed that wood contraction provides very important

Item no.	Sample	Specific characteristic		Reversible water		Change			
		Time, t_c (h)	U_c (%)	Limit (%)	Time, (h)	t_c	U_c (%)	U_M	t_M (h)
1.	Linden* new, untreated	46.00	3.90	12.20	800	-	-	-	-
2.	Linde* new, treated with red petroleum	46.00	7.25	19.20	800	0	+3.35	+7.00	0
3.	Linden new, treated with propolis	44.50	7.45	21.00	800	-1.50	+3.55	+8.80	0
4.	Linden** old, untreated	8.50	6.40	18.00	200	-	-	-	-
5.	Linden** old, treated with red petroleum	21.50	7.80	14.50	500	+13.00	+1.40	-3.50	+300
6.	Linden** old, treated with propolis	24.50	8.00	17.50	500	+16.00	+1.60	-0.50	+300

Linden* - new wood; Linden**- old wood from the year 1740.

Table 4
THE VALUES OF THE CHEMOMETRIC CHARACTERISTICS INVOLVED IN ARCHAEOMETRY AND IN THE TREATMENT IMPACT STUDY FOR LINDEN WOOD [76]

Sample origin	Characteristics			
	Provenance	State of conservation*	Age of the tree (years)	Age of the wood (years)
New linden (benchmark)	Linden-wood planks, water stabilized	Excellent	68	4
New linden (benchmark)	Linden-wood board, water stabilized	Excellent	76	7
Old linden	Support of an altar-screen console (1835)	Advanced biotic attack, strongly fragilized, few transversal craquelures	72	173
Old linden	Support of a window frame (1740)	All main casuistics (good state, biotic attack, fragilization, deteriorations, etc.)	86	268

*) In relation to the area of collecting

Table 5
THE PROVENANCE, STATE OF CONSERVATION AND DENDROCHRONOLOGICAL CHARACTERISTICS OF THE LINDEN-WOOD SAMPLES (*Tillia cordata Mill)* [76]

Wood species	ΔL (%)			ΔR (%)			ΔT (%)		
	i	ii	iii	i	ii	iii	i	ii	iii
Linden	0.20	0.24	0.18	3.20	4.20	3.00	6.00	5.30	4.70
Poplar	0.14	0.18	0.20	3.00	3.00	3.00	4.80	5.20	5.40
Fir	0.27	0.20	0.27	2.00	2.00	2.00	4.60	4.80	4.30
Oak	0.13	0.26	0.10	4.00	4.40	4.40	6.30	6.40	6.20

* Cases: i – untreated wood (benchmark); ii – wood treated with red petroleum; iii – wood treated with an propolis alcoholic solution in red petroleum

Table 6
THE VARIATION OF THE WOOD* CONTRACTION (%) ALONG THE THREE DIRECTIONS (ΔL, ΔR AND ΔT) WHEN CHANGING THE ATMOSPHERIC HUMIDITY FROM 100% TO 25% (HR) [78]

Wood species	Type of treatment *	Ratio of the contraction difference		
		ΔT/ΔL	ΔR/ΔL	ΔT/ΔR
Linden	i	28.57	15.24	1.88
	ii	22.08	17.50	1.26
	iii	26.67	16.67	1.60
Poplar	i	34.29	21.43	1.60
	ii	28.89	16.67	1.73
	iii	27.00	15.00	1.80
Fir	i	17.04	7.41	2.30
	ii	24.00	10.00	2.40
	iii	21.50	10.50	2.05
Oak	i	48.46	30.77	1.58
	ii	24.62	16.92	1.45
	iii	62.00	44.00	1.41

* Cases: i – untreated wood (benchmark); ii – wood treated with red petroleum; iii – wood treated with an propolis alcoholic solution in red petroleum

Table 7
THE CHEMOMETRIC RATIOS OF THE DIFFERENCES IN CONTRACTION ALONG THE THREE DIRECTIONS, WHEN CHANGING THE ATMOSPHERIC HUMIDITY FROM 100% TO 25% (HR) [78]

Wood species	Type of treatment *	Contraction difference (%) for		
		Volume (ΔV)	Density (Δρ)	Porosity (Δp)
Linden	i	7.50	0.21	2.64
	ii	9.50	0.14	3.34
	iii	6.50	0.26	2.29
Poplar	i	7.10	0.00	1.44
	ii	7.10	0.00	1.44
	iii	7.10	0.00	1.44
Fir	i	6.50	0.06	0.50
	ii	6.70	0.03	3.00
	iii	6.80	0.01	5.00
Oak	i	9.00	0.03	0.40
	ii	10.20	0.01	1.20
	iii	9.50	0.02	0.80

* Cases: i – untreated wood (benchmark); ii – wood treated with red petroleum; iii – wood treated with an propolis alcoholic solution in red petroleum

Table 8
THE CHEMOMETRIC RATIOS OF THE DIFFERENCE IN VOLUME CONTRACTION, DENSITY AND POROSITY OF THE WOOD ALONG THE THREE DIRECTIONS, WHEN CHANGING THE ATMOSPHERIC HUMIDITY FROM 100% TO 25% (HR) [78]

archaeometric data, were published in numerous scientific papers [104-157].

Conclusions

On the basis of the data available in the dedicated literature published in the last years by the Iasi school of Conservation Science, concerning the archaeometric and chemometric characteristic, with archaeometric value involved in authentications, establishing the state of conservation and assessing specific attributes related to the *conception*, *acquired patina*, *historical context*, etc. for metal, ceramic and polychrome wood, the following conclusions can be drawn:

- in the structure of the corrosion end-products/bulk, compounds can be distinguished according to their presence either in the *primary (noble) patina* - formed during the artefact's manufacturing and in uselife through *redox processes* (*oxides, sulfates*, etc.) -, in the *secondary (vile) patina* - resulting from *acidic-basic processes of complexation, ionic exchange* and *hydrolysis* (in the form of oxyhydroxides, halogens, carbonates, sulfates, phosphates, etc.), occurring during the ending stages of the uselife and the early stage after discardment -, or in the *tertiary (contamination) patina* - formed in the archaeological site, under the influence of *pedologic processes* (segregation, diffusion, osmosis, electro-osmosis, hydration/dehydration, fouling, mineralization, monolithization, etc.);

- the corrosion end-products of the three patinas have been identified in items originating from both disturbed and undisturbed sites;

- the congruent suprastructure (the Liesegang effect) is conspicuous in bronze coins, in which the three patinas are well individualized in the stratigraphic section;

- the Liesegang effect has continuity and preserves concentric rings of corrosion congruents in all four craquelated caps of an ancient fibula pinhead made from a copper-based alloy, which displays *deep longitudinal craquelures* resulting from *contraction at siccativation* (the loss of crystallization water and from aqua-complexes);

- the mechanism by which the Liesegang effect forms during underground lying in archaeological sites is due to the forming in certain environmental conditions (humidity, temperature, oxygen concentration, *p*H, etc.) or membrane structures at the surface of the primary patina, which in the presence of the anion chloride and of the oxygen from the soil lead to a congruent suprastructuring of the compounds from the secondary patina;

- two membrane systems have been identified microscopically in the stratigraphic structure (cross section): (i) continuous and uniform, from *hydrogels* of Sn(IV), Pb(IV) and Zn(II), which allow the concentric stratification of the congruents of chalcogens based on Cu(II), more or less unpurified by Sn(II) chlorides, followed by layers of malachite, nantokite, atacamite/paratacamite, brochantite, etc. and (ii) diffusive porous membranes, from *chloro–* or *hydroxyapatite* (in saline-weakly alkaline lying mediums, and in the presence of the ion phosphate), which initially forms similar, but discontinuous suprastructurings, and in certain cases through inverse osmosis occurs destructuring through dissolution in fluidic soil water, keeping the membrane system in the form of a stratified microporous honeycomb;

- the *evolutive modules* Si/Al, Ca/Mg and K/Na were found to constitute excellent *chemometric ratios*, alongside the *rate of ceram (aluminosilicates) solubility* under the influence of alkaline carbonates and phosphates;

- the change in time of the *granulometric distribution*; the presence of *manufacturing inclusions*; the type of *temper*, *slip* or *glazing*; the *temperature, time and type of firing* during manufacturing; the *nature and structure of the superficial crust* formed during underground lying, and others, are all very important archaeometric characteristics;

- for the wooden supports, the *normal domain of variation of the hydric equilibrium* allows establishing two *archaeometric characteristics*: *the time and critical point of correlation of the hydric equilibrium* (the intersection of the hydroscopic water adsorption-desorption curves, respectively the RMC = f(t) curves, with the limits of the domain of variation between the maximum value RMC = ΔEMC and the minimum hypothetical one RMC = 0);

- alongside the two chemometric characteristics, other characteristics were highlighted in the case of old wood, namely: the remanent concentration in crystalline cellulose; the remanent concentration in volatile components; the concentration in ash; etc.;

- the chemometric ratios of the wood contraction along the three directions - L (longitudinal), R (radial) and T (tangential) - represent a group of very important archaeometric characteristics, since their variation depends on the species, age of the wood, age of the tree, area of chopping, period and geographical area of collecting,treatment and processing during manufacturing, etc.

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