

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

II. Old linden and poplar wood put into work

PETRONELA SPIRIDON¹, IRINA CRINA ANCA SANDU², LILIANA NICA¹, COSMIN TUDOR IURCOVSCI¹, DUMITRU EUGEN COLBU¹, IOAN CRISTINEL NEGRU¹, VIORICA VASILACHE^{3*}, RALUCA ANAMARIA CRISTACHE³, ION SANDU^{4,5*}

¹Alexandru Ioan Cuza University of Iasi, Faculty of Geography and Geology, 22 Carol I Blvd., 700506 Iasi, Romania

²Munch Museum/Munch Museet, Department of Conservation, Toyengata, 53 0578 Oslo, Norway

³Alexandru Ioan Cuza University of Iasi, Interdisciplinary Research Department - Field Science, 54 Lascar Catargi Str., 700107 Iasi, Romania

⁴Romanian Inventors Forum, 3 Sf. Petru Movila St., Bloc L11, III/3, 700089 Iasi, Romania

⁵Alexandru Ioan Cuza University of Iasi - ARHEOINVEST Interdisciplinary Platform, 22 Carol I Blvd., 700506 Iasi, Romania

*The paper presents the results obtained in the study of new archaeometric and chemometric characteristics for old linden wood (*Tilia cordata* Mill.) and poplar wood (*Populus robusta*), put into work. Therefore, based on elemental chemical composition data, humidity, pH, ash and those related to wood extraction components, which are characteristics with low archaeometric potential, the chemometric characteristics with an archaeometric value were evaluated. Samples used in this study, both linden and poplar wood, the following near-linear ratios were observed in the next reports: C/O, (C+H)/O, (C+H)/N_{organic}, pH/Humidity, N_{mineral}/Ash, extract in NaOH/extract in hot water and extract in ethyl alcohol/extract in ether, which can be used in establishing an archaeometric reference.*

Keywords: authentication, polychrome wood artefacts, archaeometrics study, chemometrics study, multidisciplinary approach

Authenticity is the essential aspect of an object of cultural, artistic and historical heritage, which in general requires the establishment of *uniqueness* and refers to the quality of being *genuine* and *original*, as it was put into the work and marked by the time that passed from the moment of conception, as a unique, a *copy* or an *alternative/replacement*. Copies and alternatives may belong to the author, the school (apprentices or disciples), the age, but also to the later periods, and may be executed either in the same artistic technique as the original or in a more modern technique but close to plastic virtues [1-5].

It should be emphasized that authenticity depends on how the object is defined and can be understood differently, depending on the context. To be *authentic*, an object must be *intact*, as it was created, or as a result of the passage of time [6-9].

There are several attributes that compete to establish authenticity as a *heritage element* that is considered in investigating/researching, preserving, restoring and displaying a work of art, namely: *conception/author/dating, material, artistic technique, implementation technology, the environment of creation, use (wear) and the area of use, owner, custodian, museum, transfer and/or itinerary* [4, 5].

The first ones, along with the originality of the material and the technology of putting it into practice, are references to other *types of authenticity*, which, together with *paternity, value share, ranking, and age* form the *group of patrimonial elements* or *patrimonial features*, that characterize and differentiate an object. Also, the traces of time passage, historical wear and use, are part of the *historical stratification*, translated by the *patina* and fix the object in time and space, making it *unique* [4-9].

Authentication expertise through their implications, either in the field of *valuation of cultural-artistic heritage assets* (artometry or artefactometry) and *technical-*

scientific (technometry and scientometry), either as a means of proof or evidence in *forensic* (graphometry, bibliometry, traceometry, emprento- or dactilometry, somatometry, biometry/anthropometry and others) require interdisciplinary knowledge [8, 9].

In solving them, there are used different assisting or corroboration systems between methods and techniques belonging to the related disciplines, which allow to open new directions in authentication, especially under the present conditions of liberalization of the market with heritage assets and the unprecedented development of forgeries, made with vintage or obsolete materials by well-established, hard-to-find processes and techniques.

Authentication methods are grouped into direct examination, *non-destructive*, and *sampling and processing of microsamples* (semi- or pardestructive sampling). In this way, it is possible to use surface analysis methods such as: *textures, irises, bladders, erosions, deposits, finishes, polishes, grinding, inlays, engravings, watermarks, inscriptions or graphs, emblems, legends, various decoration, colour process* etc., when direct observation is used, by naked eye or with optical magnification tools [10-19]. Also, for determination by direct measurement of *size, weight, color, temperature, humidity* etc., used static/dynamic Analog or Digital Photography (Photogrammetry, Stereophotogrammetry and Videogrammetry), UV, Vis and IR Reflectography, X-ray Fluorescence, Reflectance Colorimetry/CIE L*a*b*, and others. Then to methods of direct analysis of internal structures (such as: *the nature, structure and layout of the materials in the substrates, in the preparation layers and in the colour layer, then the strength and bonding elements, defects, hidden deteriorations and degradations* etc.) often used: Thermogravimetry (TG, DTA, DTG and DSC) [20-22], UV, Vis and IR Spectroscopy [23-26], Radiography, IR Thermography, Emission Spectroscopy, Thermoluminescence and Microendoscopy [27-32].

* email: viorica_18v@yahoo.com; ion.sandu@uaic.ro

Among the modern semi- or pardestructive methods, it is used: Microstratigraphy, assisted or not by Histochemistry, Optical and Electron Microscopy, UV, Vis and IR Spectroscopy (last in the micro-FT-IR version), Roentgenography or X-ray Diffraction, Nuclear Magnetic Resonance, Thermal Derivation or Differential Calorimetry, normal or Pyrolytic Gas Chromatography, with or without Silylating, assisted by Mass Spectrometry, Isotopic Dilution and Radiometry with radioactive tracers, as well as an entire arsenal of specific methods such as: Dendrocronology, Leukometry, Glycolysis rate, Amino Acid Racemization, Metallography and others. These, besides making use of sampling, are laborious, and equipment and devices very expensive, which is why they are rarely used, many times being required in examination for reasons of uncertainty, unclear or inaccuracy of the data obtained by direct methods or when they are doubtful [30-38].

It should be noted that using different instrumental methods for authentication of works of art is imposed due to the fact that the aesthetic and the plastic expertise, based on stylistic considerations and complemented by the revealing of the characteristic detail, are not sufficient, and therefore, for accuracy of authenticity and identification, it is used corroboration system with them. This is the reason why lately they have witnessed a wide expansion and refinement of the technical — scientific methods of establishing authenticity, going to the level of expert systems, specialized on certain objects, materials, structural — functional elements, effects and temporal phenomena, etc.

For most methods, the most important issue is the reference system, comparison or reporting system as well as the calibration system (curves, diagrams, levels, groups, quotas, grids, etc.). The reference, comparison or reporting systems may be in the form of: catalogs; binders; collections; pinacoteque; warehouses; archives; which contain: objects, scientific replicas or documents, photographed or filmed evidence and drawn documents, requiring complex and laborious fundamental research.

Following the investigation of a heritage object, either by determining the nature and age of the materials, or by establishing specific characteristics of the artistic technique and the technology of putting into work from a certain period (time), geographical area (space) and author (school, current) or through the analysis of patina, polychromy, graphs, deteriorations and degradations etc., it is also possible to establish details about one or more attributes of authentication.

The role of authentication is undeniable, especially because in addition to information about originality, uniqueness and attribution it can be obtain a series of data regarding the conservation status or the nature of the materials, the artistic technique and the technology of putting into work, which allow for compatibility studies and selection of intervention systems in active preservation (treatment) or restoration (consolidation, stabilization, reintegration).

Nowadays, a new interdisciplinary system-archeometry, which identifies and evaluates both structural and compositional evolutionary (archeometric) and chemometric values with archeometric value, has become more and more necessary in authentication [8, 9].

For authentication of old wood artifacts, in the specialty literature there are many reference works that have studied either a certain artifact or a structural component [30-38] in which using chemical and physico-structural analysis data from wood samples with different sessions from time

of putting it into work or cutting, it has been evaluated a series of archaeometric or chemometric features with archeometric value. For example, elemental composition, hygroscopic moisture, ash and extractable components [39], along with the morphology and distribution of anatomical components, porosity, specific weight, etc. are archeometric features, which by corroboration allow the dating of the old wood.

The chemometric reports of wood contraction in the three directions: L (longitudinal), R (radial) and T (tangential); have been proven to be very important archeometric features, because their variation depends on the essence, the age of the wood, the age of the tree, the area of the cutting, the period and the geographical harvesting area, the treatment and processing process at putting into work etc. [39].

These examples have inspired the use of elemental chemical composition data and those of extractive wood components of various essences and antiquities, which are features with low archeometric potential, when evaluating some chemometric features with archeometric value.

This paperwork presents the chemometric characteristics evaluated on the basis of elemental chemical composition and extraction components of linden and poplar wood using samples of four levels of age and with close conservation statuses.

Experimental part

Most of the old paintings with preparation layers (with or without decorative frames) from Romania and Central Italy (Toscana) are made of linden, poplar or fir wood, and rarely from wood of cherry, oak, spruce, beech, maple, maple, elm, cypress etc.

Whatever the type of wood supports is used, due to the inhomogeneity of the structure in the volume phase, it presents specific domains, with variable and complex expansions, in terms of chemical composition and structure [33-41], which are dependent on a number of exogenous factors (external agents or conservation environment) and various preservation and restoration interventions, subsequent to the commissioning.

The specificity and complexity of these areas are determined by the endogenous factors (species, wood structure / defects, tree age, wood age, shelf life, cutting period, place and method of cutting, technology and processing and treatment conditions, technology for putting into work and others).

In investigation of heritage objects containing wood elements, the primarily elements are the species, the age of the tree, the age of the wood and its state of preservation.

Species are microscopically determined by morphological examination using identification keys, using tree age annual growth rings, and the age of wood through dendrocronological examination. These data allow to highlight the evolution of structural and chemical characteristics during use/storage (from commissioning).

In this respect, it is very important to correlate the main chemical composition and extraction components, as well as other chemical characteristics, with the age of some samples of wood essences often used as support for icons or other old paintings with preparatory layers. Various samples were taken from different areas with a preserved state of conservation. As standard were used new wood samples of the same indigenous essences, stabilized by known hydro-thermal processes.

Sampling and sample processing

For examination, were taken samples of old linden (*Tillia cordata* Mill.) and poplar wood (*Populus robusta*), taken from the supports of floral icons or decorations, found in various monasteries and private collections from the Romanian region of Moldavia. In this respect, different supports were selected which did not show biotic attack, there were in good conservation status and untreated insect phungic after putting it into work. Two samples of new wood from these two essences, water stabilized, were taken as a standard or witness. First examination was dendrocronological one (tables 1 and 2), after which they were chemically analyzed, for each sample were determined the elemental chemical composition, humidity, pH, ash and composition in extraction components.

Linden wood samples came from old racks or frames: 99 years, 145 years and 200 years old. The poplar wood was also taken from old racks or frames 100 years, 154 years and 208 years old. Linden wood standard test is 6 years old and the poplar one is 7 years old.

Chemical and technical examination

Establishing elemental composition

The C and H composition of wood samples was determined using elemental microanalysis [42-45], by combustion of a 3-7 mg sample, which was thermally decomposed into a quartz tube under the action of an oxygen stream, at a flow rate of 20-25 cm³/min at atmospheric pressure when at 850-950°C resulted CO₂ and water, which were captured in anhydride and ascarite absorption vessels, which were weighed, of the mass to which the C and H content was determined.

Nitrogen was determined by the Kjeldahl method [45, 46].

For the determination of mineral substances, there were used methods described by Simionescu [47].

Ash establishing

A sample of 5-10 g of wood, previously dried at 105°C (up to constant mass), was placed in a crucible which was initially calcined at 700°C and then weighed. Initially, burning in the crucible was performed with flame of a gas lamp. Upon completion of the combustion, the crucible was placed in the calcined at 700°C for 20 min, then the ash crucible was cooled in the desiccators' for 30 min and then weighed. If traces of uncalled substance remained, the ash was treated with 5 cm³ of 1 M sulfuric acid, then evaporated on a water bath and calcined again to a constant mass. The ash content relative to the dry matter was calculated using the formula:

$$\% \text{ ash} = 100 \times m_{\text{residue}} / m_{\text{sample}}$$

Since ash was attributed to mineral substances, oxygen was determined by difference [48-50].

Establishing extractable (soluble) components

It was used the method of extracting soluble substances in cold water (10°C), hot water (85-90°C), aqueous solutions of 1% NaOH, absolute ethyl ether and 96% ethyl alcohol using a Soxhlet [51, 52].

In all cases, after extraction, the solutions obtained were filtered through a quantitative paper (blue band), then a 100 cm³ pipette was taken and introduced into a crystallizer. The extracts were evaporated in a self-adjusting oven, at 105°C, to a weight, when a mass of syrupy consistency was obtained. For each sample, the extract content was calculated using the formula:

$$\% \text{ extract} = 100 \times m_{\text{residue}} / m_{\text{sample}}$$

determining the percentage of substances extracted from the absolutely dry sample.

Establishing pH sample

In order to determine the pH of the 14 wood samples (10 linden and 4 poplar), both the expedient method (with

Table 1

ORIGINE, CONSERVATION STATUS AND DENDROCRONOLOGICAL CHARACTERISTICS OF LINDEN WOOD SAMPLE (*Tillia cordata* Mill.)

Sample	Characteristic			
	Origin	State of conservation *	Age of the tree (years)	Age of the wood (years)
1. New linden (standard)	Water stabilized linden leaf	Excellent	26	6
2. Old linden	The support of a royal door (1903)	Very good (without biotic attack)	62	99
3. Old linden	Icon support (1857)	Very good (without biotic attack)	84	145
4. Old linden	Supporting a mole (1802)	Very good (without biotic attack)	69	200

*) Reported to sampling area

Table 2

ORIGINE, CONSERVATION STATUS AND DENDROCRONOLOGICAL CHARACTERISTICS OF POPLAR WOOD SAMPLE (*Populus robusta*)

Sample	Characteristic			
	Origin	State of conservation *	Age of the tree (years)	Age of the wood (years)
1. New poplar (standard)	Water stabilized poplar leaf	Excellent	21	7
2. Old poplar	Frame a floral frame (1902)	Very good (without biotic attack)	38	100
3. Old poplar	Icon support (1848)	Very good (without biotic attack)	92	154
4. Old poplar	Crucifix support (1794)	Very good (without biotic attack)	71	208

*) Reported to sampling area

chromatic indicators) it was used indicator paper soaked with specific reagents, and on the base of colour was evaluated the pH using the chromatic scale of that set of indicators and the pH meter method. According to the electrochemical method of determining the pH of heterogeneous solids objects by dispersing the finely divided samples into the water, first step was to polish and crushing 2.5-5.0 g of each wood sample, then the powder was passed through the 40-mesh sieve (TAPPI standard), after which the screened material was dispersed into 20 cm³ of bidistilled water (pH = 7.00), thus obtaining a concentrated suspension in which the pH-meter electrodes were submerged. After 30 minutes of maceration under a slight stirring, the pH of the sample was noticed, which was compared with data obtained through expedition method.

The moisture content of the wood samples was established using electrical methods and oven-drying method (with thermostats), at 105°C until constant weight.

For the first group of methods, determinations were performed directly on supports, and for the samples were used parallelepiped test tubes with the size of 60 × 30 × 15 mm. In heating method by using the oven, was used 1 g of sawdust, weighed at the analytical balance. Moisture was expressed as a percentage of the sample taken at work [2, 39].

Results and discussions

Chemical composition and technical analysis

The quantitative determination of the chemical elements in the native wood composition and after processing/treatment or putting into work is very important; on the one hand, information about species and its origin can be obtained, and on the other hand, some characteristics can be assessed regarding the nature of the treatment applied on the object (when old wood was examined) or to establish to degree of aging.

Any kind of wood contains in its elemental chemical composition: C, O, H, N and a number of mineral substances.

Tables 3 and 4 present data about the elemental chemical composition of studied wood, experimentally determined on siccated samples. Moreover, show data's about percentage of ash resulting from combustion, the pH and the humidity of the sample.

Data from table 3 and 4 shows that after the treatments from putting into work, subsequent conservation interventions and due to preservation (aging), the composition in C, H and mineral salts increases sensibly, while the concentration in O and N decreases in about the same ratio. Moreover, there is an increase in pH and a decrease in moisture. It is noted that with the increase of the degree of division and the porosity resulting from the xylophag attack, the humidity of the supports decreases. At collapse, the humidity decreases near the irreversible value (2.5%). Following insecticide treatment with petroleum products, both elemental composition and other chemical characteristics is changing: ash, pH and sample humidity.

Sample	C (%)	H (%)	O (%)	N and mineral substances * (%)	N (%)	Ash (%)	pH of the sample	Humidity (%)
1. New linden (4 years)	49.17	6.41	43.27	1.15	0.75	0.40	5.08	10.00
2. Old linden (97 years)	49.49	6.63	42.80	1.08	0.67	0.41	5.22	9.00
3. Old linden (143 years)	49.84	6.86	42.21	1.09	0.64	0.45	5.46	8.00
4. Old linden (198 years)	49.89	6.97	42.06	1.08	0.61	0.47	5.51	7.50

*) Mineral substances are dosed as ash (SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO etc.)

Table 3
THE ELEMENTAL
CHEMICAL
COMPOSITION OF
LINDEN WOOD
SAMPLES

Sample	C (%)	H (%)	O (%)	N and mineral substances * (%)	N (%)	Ash (%)	pH of the sample	Humidity (%)
1. New poplar (5 years)	50.75	6.64	41.50	1.11	0.80	0.31	4.85	14.00
2. Old poplar (98 years)	50.82	6.93	41.18	1.07	0.74	0.33	4.98	12.00
3. Old poplar (152 years)	51.01	7.14	40.82	1.03	0.68	0.35	5.14	10.00
4. Old poplar (206 years)	51.16	7.21	40.65	0.98	0.61	0.37	5.40	9.00

*) Mineral substances are dosed as ash (SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO etc.)

Table 4
ELEMENTARY
CHEMICAL
COMPOSITION OF
POPULAR WOOD
SAMPLES

Table 5
THE COMPOSITION OF LINDEN WOOD SAMPLES IN EXTRACTION COMPONENTS

Sample	Extract in cold water 10°C (%)	Extract in hot water 80°C (%)	Extract in aqueous solutions of cold NaOH 1% (%)	Extract in anhydrous ethyl ether (%)	Extract in 96% ethyl alcohol (%)
1. New linden (4 years)	0.87	3.36	18.75	1.27	4.25
2. Old linden (97 years)	0.84	2.76	18.44	0.96	3.98
3. Old linden (143 years)	0.82	2.19	17.96	0.55	3.86
4. Old linden (198 years)	0.79	1.86	17.58	0.41	3.78

Table 6
THE COMPOSITION OF POPLAR WOOD SAMPLES IN EXTRACTION COMPONENTS

Sample	Extract in cold water 10°C (%)	Extract in hot water 80°C (%)	Extract in aqueous solutions of cold NaOH 1% (%)	Extract in anhydrous ethyl ether (%)	Extract in 96% ethyl alcohol (%)
1. New poplar (5 years)	0.74	3.28	23.34	1.66	2.28
2. Old poplar (98 years)	0.68	2.79	20.96	0.91	2.49
3. Old poplar (152 years)	0.65	2.25	18.54	0.63	2.65
4. Old poplar (206 years)	0.61	1.81	17.14	0.49	2.81

Tables 5 and 6 show the composition of wood samples in soluble components, separated by extraction. There is a clear difference between the concentrations of extractable components in organic solvents of the new samples compared to the old samples. In contrast, extracts in aqueous systems have much less differentiation.

It is known that, over time, wood loses some of the fat and etheric components. By aging, much of them degrades, turning into volatile components [53-55].

Subsequently based on elemental chemical composition data (C%, H%, O%, N_{organic}%), the ash% by combustion, of the humidity content % (UR) and the pH of

the wood sample taken into study [42-45] seven sets of chemometric characteristics were evaluated based on the age of the wood, which are shown in figure 1.

All seven graphs have gradual curves, but there are only four reports of almost linear, namely: C/N_{mineral}, (C+H)/N_{organic}, (C+H)/O and N_{mineral}/Ash, which can be used in archeometric determinations, while the others do not have linear evolutions, being without archeometric function.

Similarly, for poplar wood, based on primary data of elemental chemical composition, pH sample, humidity and ash the charts of evolution have been drawn for seven

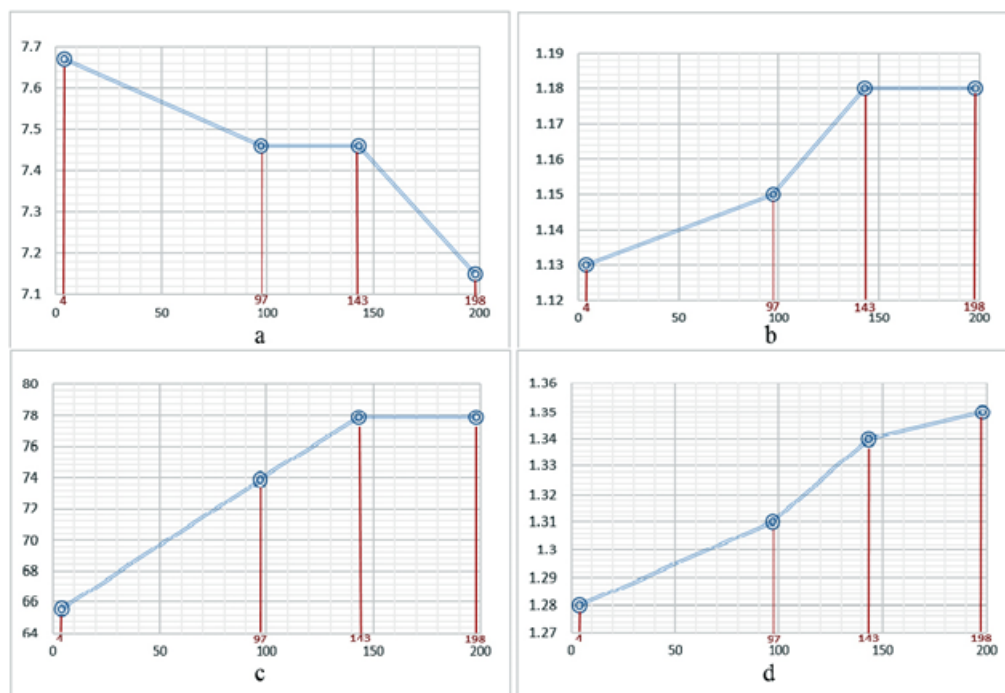


Fig. 1. Variation of chemometric characteristics depending on the length of linden wood:
a - C/H, b - C/O, c - C/N_{organic},
d - (C+H)/O,

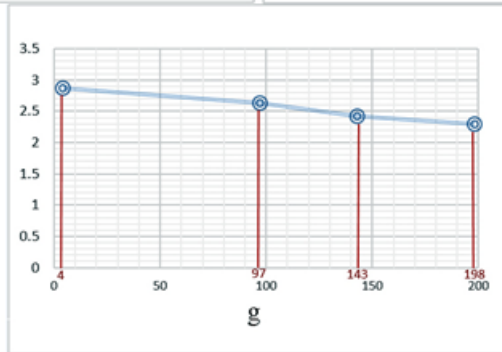
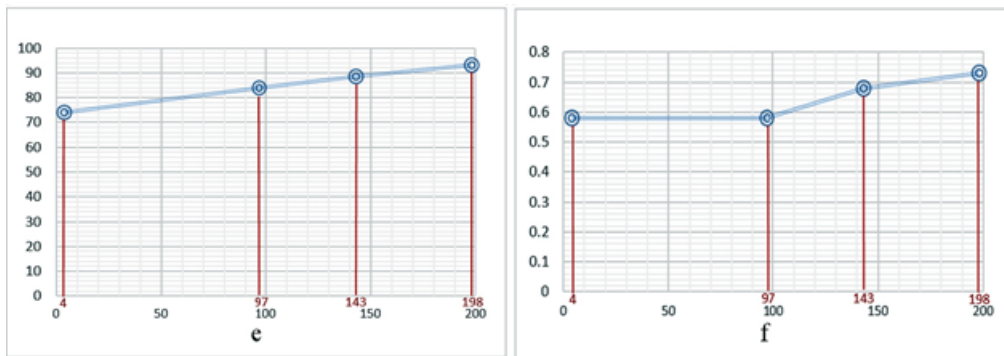


Fig. 1. Variation of chemometric characteristics depending on the length of linden wood:
 e - (C+H)/N_{organic}, f - pH/Humidity, g - N_{mineral}/Ash

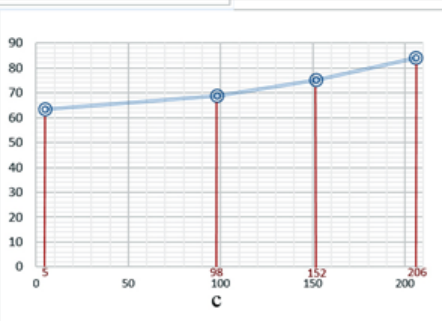
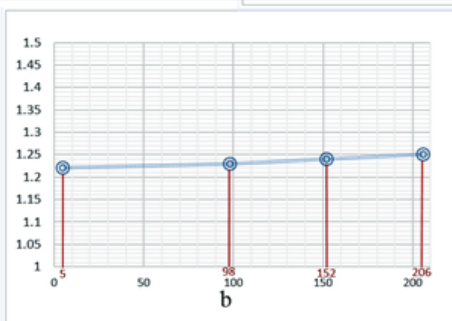
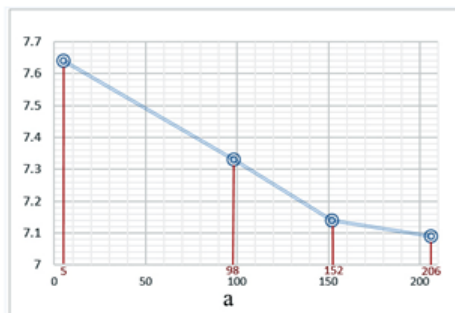
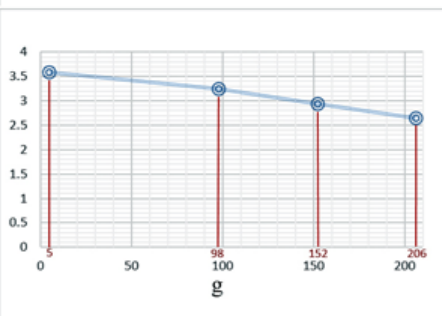
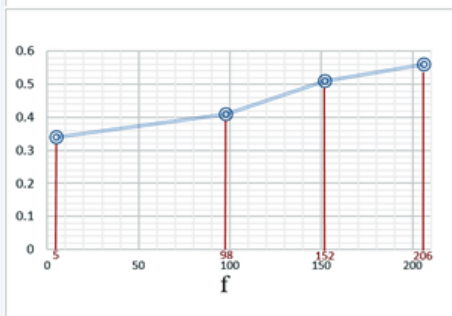
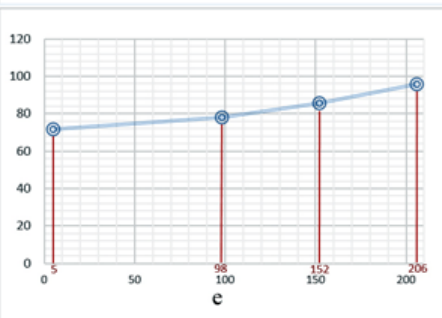
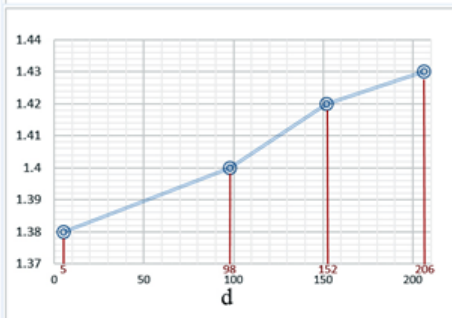


Fig. 2. Variation of the chemometric characteristics according to the age of poplar wood:

a - C/H, b - C/O, c - C/N_{organic}, d - (C+H)/O, e - (C+H)/N_{organic}, f - pH/Humidity, g - N_{mineral}/Ash



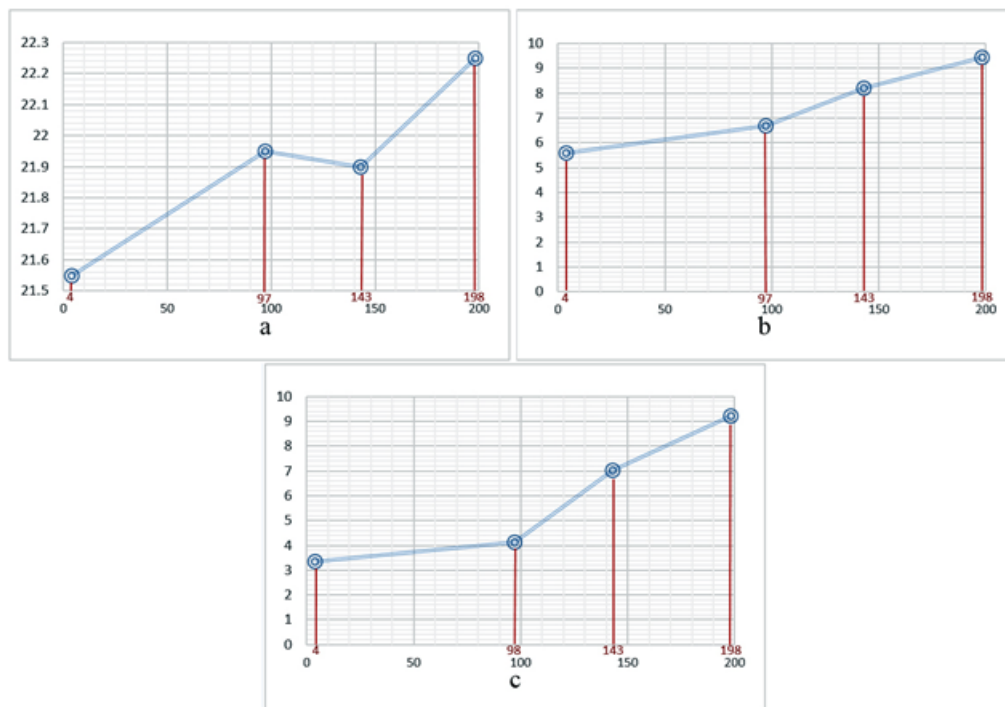


Fig. 3. Graphical representation of component composition ratios extracts from linden wood:
 a. aqueous 1% NaOH/cold water (10°C); b. aqueous 1% NaOH/hot water (80°C) ; c. ethyl alcohol (96%) /ethyl ether anhydrous

series of chemometric characteristics according to the age of the wood, as it can be notice in figure 2.

Compared with linden wood, the poplar wood has all chemometric characteristics with gradual evolution almost linear, which would allow their use in archeometric assessments.

Similarly, the chemometric characteristics were determined for the ratios between extractable components in aqueous 1% NaOH/cold water (10°C) and aqueous 1% NaOH/hot water (80°C), as well as those extracted in ethyl alcohol (96%) and in ethyl ether anhydrous in linden wood with different ages, respectively for poplar wood. Figure 3

shows the evolution curves of the three reports for different age of linden wood. From these chemometric features, only the last two reports have archeometric value.

Regarding evolution of the second group of chemometric characteristics (ratios between the composition in extractable components) depending on the age of the poplar wood, it is notice that there are developments of the curves very similar to those for linden wood (fig. 4).

As in previous results, from three chemometric features in this case the last two may have archeometric value.

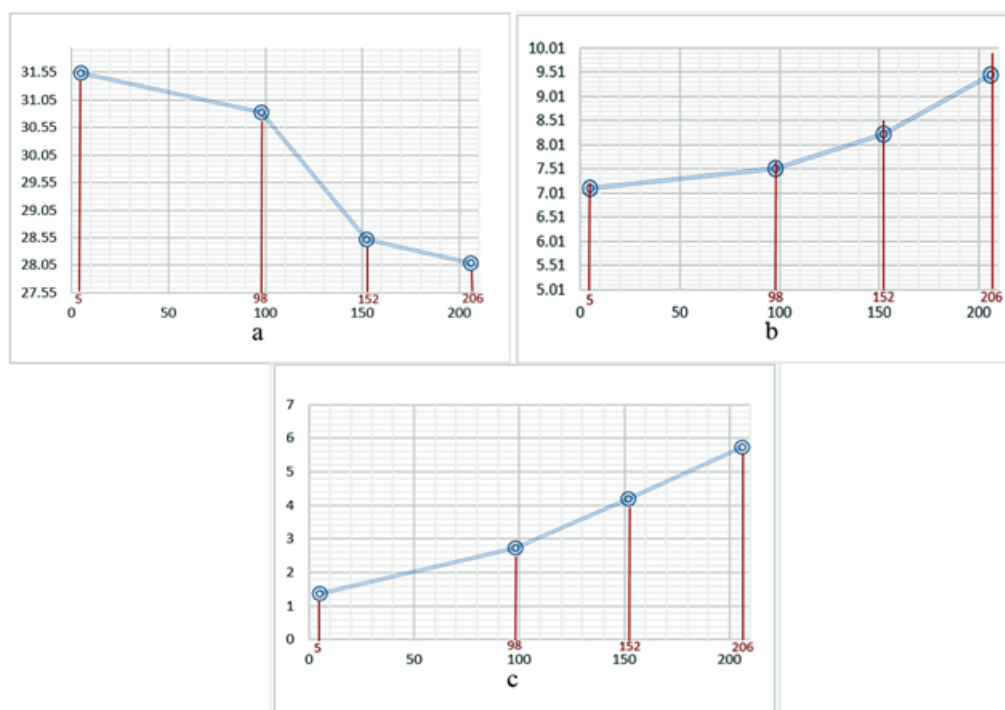


Fig. 4. Graphical representation of component composition ratios extracts from poplar wood:
 a. aqueous 1% NaOH/cold water (10°C); b. aqueous 1% NaOH/hot water (80°C) ; c. ethyl alcohol (96%) /ethyl ether anhydrous

Conclusions

In the research, the data on elemental chemical composition were approached (C%, H%, O%, N_{organic} %), ash%, humidity% and pH of sample, along with those on linden and poplar extraction components, for four levels of age and close conservation status, which were used to determine chemometric characteristics with archeometric value.

Regarding these examination, the following conclusions can be drawn:

- From seven curves of chemometric ratios on elemental composition data, pH of sample, wood humidity and ash (C/H, C/O, (C + H)/O, (C+H)/N_{organic}, pH/Humidity and N_{mineral}/Ash according to age) for linden wood only four reports have archeometric value (gradual evolutions, close to linearity): C/N_{mineral}, (C+H)/N_{organic}, (C+H)/O and N_{mineral}/Ash. In exchange for poplar wood all reports can be used as archeometric value;

- Similarly, graphical representations of the concentration ratios in extractable components (ratios of the extractable component composition in 1% cold NaOH (10°C) and those extracting in cold water (10°C), then those extracting in 1% cold NaOH (10°C) and those extracting in hot water (80°C) and the last ones, those extracting in ethyl alcohol (96%) and those extracting in anhydrous ethyl ether), depending on the age of the linden and separate the age of poplar wood, of which only the last two have an archeometric value (extractable in 1% cold (10°C) NaOH in relation to those extracting in hot water (80°C) and those extractable in ethyl alcohol (96%) relative to those extracting in anhydrous ethyl ether).

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Manuscript received: 01.10.2016