New Trends in Science and Mathematics

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Prof. Hüsniye SAĞLIKER, Ph.D.

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Fundamental Analytical
Methods and Case Studies Used in Archaeological Studies:
The Basis of Archaeometry

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INTRODUCTION

Archaeology is an important science that enlights on the history of humanity when, especially, it is studied together with many sciences such as chemistry, physics, biology, geology, geochronology, geography, geomorphology.

Geochemical analytical methods are necessary in order to determine what archeological artifacts, finds, and cultural heritage items are made of, identify their origins, and the time period they belong to. As a result of the rapid scientific and technological advances precipitated by the Second World War, several analytical techniques were deployed in archaeological studies, including X-ray analysis and electron microscopy, neutron activation analysis, and mass spectrometry (Pollard, Batt, Stern, Young, & Young, 2007). In order to characterize the material of the ceramics from which the pottery is made, to identify the rocks used in the restoration works to identify the rocks used in the construction of the artifact, and to determine their origins, the most important thing is petrographical analysis under a polarizing microscope. With a petrographical analysis, the binder-aggregate ratios of mortars and plasters, the types and properties of the aggregates they contain can be determined, and suggestions can be developed on possible source areas. Moreover the properties of the rock, mineralogicalcompositions, textural properties and alteration and weatheringtypes could be determined, and suggestions can be made for the type of stone to be used in the building. After that, the analytical method can be determined based on the mineralogy-petrography of the material, the scope, and the aim of the study.

There are several comprehensive publications regarding archaeometric studies in the literature. In this study, the most common geochemical techniques such as XRF, XRD, ICP-MS, LIBS, FT-IR and RAMAN which were utilized in archaeological studies and some case studies will be presented (Table 1).

Table 1 Some of the analytical techniques commonly utilized in archaelogical studies modified from Ricci (2017).

Abbreviation	Name	Principle	Sample Preparation	Portable - Labratory
XRF	X-ray Fluorescence	Elemental quantitative and qualitative analysis can be performed. It uses high energy X-ray beam to excite the sample and measures the wavelength and energy of the characteristic radiations emitted from the atoms when they are coming back to the fundamental state. It is used for chemical analysis for the surface of ceramic glaze and body	In the labratory you can make pellet samples (<65 micron sized powder samples) In-situ analyses dont require any sample preparation process	Portable for in-situ measurements Labratory and in-situ
XRD	X-ray Diffraction	Qualitative and semi-quantitative analysis of the mineralogical composition. It is based constructive interference of monochromatic Xrays and a crystalline sample. One of the most useful technique to study the crystal stru cture and atomic spacing of the clay minerals.	10-100 mg of homogenous powdered samples	Labratory
FT-IR	Fourier-Transform Infrared Spectroscopy	It is an absorption technique which can be used to identify chemical compounds and it provides information about the chemical bonding in both inorganic and organic materials.	A few mg samples adequate	Labratory
Raman	Raman Spectroscopy	It is an absorption technique, as FT-IR, for molecular identification based on inelastic scattering of monochromatic light. It allows to identify organic and inorganic materials and in the ceramic material studies it is used to detect minerals.	There is no sample preparation process. It is not a destructive technique.	Labratory and in-situ

LIBS	**Laser-Induced Breakdown Spectroscopy**	It is a practical atomic emission spectroscopy method which is used highly energetic laser pulses to provoke optical sample excitation, offers an appealing means of discriminating different materials, their sources, volcanic ant archaelogical centers in real-time.	There is no sample preparation process. It is not a destructive technique. If you want to make more detail study and get more trace elemnts (even light elements) you can prepare pellet samples	Labratory , in-situ also remote
LA-ICP-MS	**Laser Ablation-Inductively Coupled Plasma Mass Spectrometer**	A type of mass spectrometer that uses induction coupled plasma to ionize the sample. It atomizes the sample and creates atomic and small polyatomic ions that are then detected.	You can make the analyses on the carbon coated thin sections	Labratory

XRD & XRF (XRF, micro-XRF, P-XRF)

One of the most commonly used techniques on the archaeological studies has been XRF. With the advancement of technology, micro-XRF and portable XRF devices have been developed. Portable X-Ray spectroscopy (p-XRF) is a technique utilized to detect and measure the chemical contents of the objects in excavation sites, museums and private collections, as it allows on-site analysis, is non-destructive and could make quick measurements (Şimşek et al., 2014).

Tolun and Ay (2017) found ancient granite quarries in the Troas and Mysia Regions and Western Anatolia in their archeometry studies. Major and trace element chemistry was determined by XRF method and mineralogical petrographic examinations were examined under optical microscope (OM). As a result of this study, they determined that at least two different granite types were used in the Agora of Smyrna and the Ancient City of Tlos.

The lack of detailed petrographic and mineralogical analysis and description of the natural geological building materials used in archaeological structures, the failure to use the correct rock or material during the restoration of such important structures, and the restoration of the building in different rocks instead of the rocks used in ancient times may cause the archaeological structure to turn into a building image outside of its primitive nature.

In order to restore historical buildings correctly, documenting and recording the current state of the building and defining the building materials by making archeometric analyzes during the pre-restoration project preparation phase are the substantial parameters. Recently, archaeometric studies regarding building materials are widely used to solve different problems on archaeological artifacts of different sizes and types (metal, glass, ceramics, stone, mortar, plaster) and building materials. Aydın, Tetiker, and Tanrıkulu (2019) conducted a comprehensive study in the Şanlıurfa-Hacı Yadigâr Mosque. In this study, analyzes were made on mortar, plaster, and stone samples taken from the mosque using optical petrographic, XRD, and P-XRF Methods. In the non-destructive sampling areas, only P-XRF analyzes were made.

Optical microscope, XRD and micro-XRF methods were used in archaeometric studies for Early Roman ceramics in the Tarsus region (Akyol, Tekkök, Kadıoğlu, & Demirci, 2007). Thin sections of the obtained ceramic pieces were taken to show all layers from the outside to the inside, and petrographic examinations were made under an optical microscope. Thin sections were also prepared and studied for clay, (soil) sediment and stone samples. XRD analysis was performed to determine the mineral phases in soil and clay samples. Point Micro-XRF analysis, which is one of the non-destructive methods, was applied to determine the coloring matter in the glaze. Consequently, material characterization was made and sources were tried to be determined, the content and source of paint materials were determined, and the presence of local or non-local ateliers was determined with geochemical methods.

Ceramics composed of variety of clay minerals have a long history and are found in almost all societies. They do not deteriorate easily, and are often found in large quantities in archeological excavations. Clay minerals' structures present in could be detected by XRD (Fig.1) and have frequently been used as a reference for the thermal treatment of ceramic objects since the distinct crystallographic phases of minerals be exposed to variations in clay bounding up with the firing temperature (Mangueira, Toledo, Teixeira, & Franco, 2011)

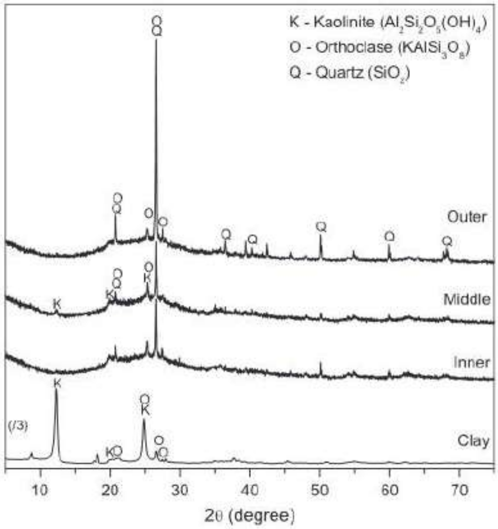

Fig. 1. X-Ray diffractograms of the outer, middle, and inner layers of the ceramic sample and the clay (Mangueira et al., 2011).

In their study, Dirican, Atakuman, Biler, and Erdoğu present the first data of archaeometric studies carried out to determine the raw material sources and production techniques of pottery (ceramic), stone ax and chisel, marble, obsidian and some other stone findings unearthed in Gökçeada Uğurlu Höyük. For this purpose, after petrographic studies to determine the mineralogical and chemical properties of the raw material used in the production of the findings, XRD and P-XRF methods are applied.

In this study, although the source of ilmenite and smectites, which are the materials of pottery ceramics, was found on Gökçeada, the central source of the serpentinites determined in the structure of some materials could not be found around Gökçeada. However, the fact that the source of the obsidians was determined as Göllüdag and Nenezi Dağ in Central Anatolia makes this study interesting and clearly reveals the importance of geochemical analyzes in archaeological studies.

LIBS & LA-ICP-MS

LIBS is a very fast, sensitive and successful technique for qualitatively and quantitatively determining the elements in a given sample, and it was first applied in this field in 1963, right after the discovery of lasers (Miziolek, Palleschi, & Schechter, 2006). By using LIBS method, major, trace and even some rare earth elements (Na, Li) within the sample can be detected easily and fastly. Along with that, more detailed trace element contents can be determined with micro-LIBS device. LIBS has recently been used frequently in geology studies because it does not require any sample preparation, can be applied on solid samples, and has advantages such as fast analysis and fast results (i.e. (Gençoğlu Korkmaz, Gündoğdu, Kılıç, & Kurt, 2021). LIBS can also be used with XRF or RAMAN to give more effective results. Recently, the Raman method is frequently preferred with LIBS in studies such as mineral determination, identification and classification of minerals, since no damage is caused to the samples to be analyzed and analyzes are carried out in an easy and practical way without sample preparation (Akçe & Kadıoğlu, 2020; Bazalgette Courrèges-Lacoste, Ahlers, & Pérez, 2007; Koralay & Kadioglu, 2015; Lu et al., 2015; Sharma, Misra, Lucey, Wiens, & Clegg, 2007; Yunfeng, Ying, Jingwen, Zhongchen, & Ying, 2015).

LIBS is a widespread (Awasthi, Kumar, Rai, & Rai, 2016; Harmon et al., 2018; Muhammed Shameem et al., 2020; Remus et al., 2010; Remus et al., 2012) and easy technique for analysis and characterization of the composition of a broad variety of objects of cultural heritage containing sculpture, painted artworks, polychromes, icons, pottery, and glass, metal, and stone artifacts. Remus et al. (2012) tested whether the localities of obsidians taken from two different obsidian centers in California could be distinguished and to what extent sub-sources could be identified in each of these centers in their study on obsidians using the LIBS method (Fig. 2). Classification of samples was performed using PLSDA, a common chemometric technique to perform statistical regression on high-dimensional data. Separation of samples from Coso Volcanic Field, Bodie Hills, and other large obsidian fields in north-central California has been demonstrated to be possible with greater than 90% accuracy using both spectral bands. Since obsidian is a high silica rhyolitic volcanic glass and almost every obsidian has a similar composition, it was preferred to determine the detection limits of trace elements with the LIBS method in order to be geochemically distinctive, thus determining the obsidian sources and sub-sources.

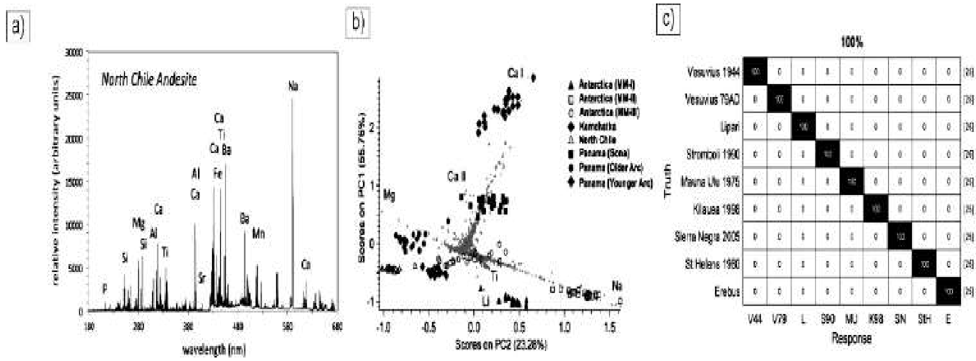

Fig. 2. a) Al, Mg, Si, Ba, Ca, Ti, Fe, P spectra obtained as a result of LIBS analysis on obsidians, b) PCA distributions and discrimination diagram and c) PLSDA diagram Remus et al. (2012) modified.

Restoration of archaeological metal objects includes a complex and multistep process that contains the object’s analysis, strengthening its surface and internal structure, corrosion stabilization, preservation, reconstruction of components, and replenishment of losses. Cleaning of abrasive layers has a critical role due to the possibility of removing contaminations, suggesting the shape of the object being restored and its technological and decorative features. Recent studies have recommended several methods for cleaning metal objects (Craddock, 2009; Scott, 1994). Laser cleaning is a promising approach, which can solve some complicated restoration tasks as the cleaning of objects with inlays. Fs lasers present pioneering cleaning facilities to reveal the information about the investigation of some historical materials such as coins (Abdel-Kareem, Al-Zahrani, Khedr, & Harith, 2016), parchment papers (Walczak & Heald, 2008), wood or soil based cultural heritage objects (Kaminska et al., 2005). It has been presented that copper-zinc alloy coin determines optimal conditions for removal of acorroded layer. Korkmaz et al. (2022) utilized a historical coin, which was made of copper-zinc alloys was used for ablation process to investigate the effectiveness of 800nm wavelength for cleaning surface corroded layers (Fig. 3). Hence, a series of experiments were performed using laser power values ranging from 10-500 mW per pulse and several repetitive applications were executed to reach desired results. Experimental studies reveal that the best method applied on archaeological artifacts is mechanical cleaning without using chemicals. At the same time, it has the advantage of minimizing the risk of any layer or corrosion formation on the work because it is a recyclable work. For this reason, it is one of the most successful techniques among conservation methods, as it offers the opportunity to analyze again in the future.

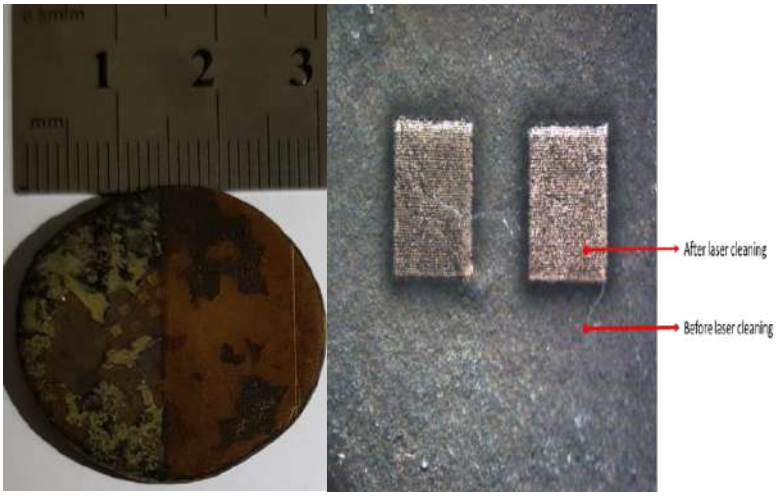

Fig. 3. Before and after photos of cleaning the surface of a historical coin collected in Konya region with Fs-LIBS (Korkmaz et al., 2022)

It is commonly known that obsidian (volcanic glass) was used in the construction of many tools in archaeological periods. Erturac et al. (2012) performed some geochemical analysis (for 8 major oxides and 30 minor elements) using the LA-ICP-MS method of 200 samples taken from various levels of each obsidian outcrop in their study from the Göllüdağ Volcanic Complex (Central Anatolia).

Obsidians, which can be found in 10-50 cm dimensions and do not contain devitrification products such as lithophytic and sphelurite (McPhie, 1993), and therefore observed as outcrops suitable for chipping, are defined as obsidian sources (OBS). Prehistoric obsidian workshops (chipping centers) are observed in many OBS environments around Göllüdağ (Balkan-Atlı et al., 2010). In this study, obsidians were classified as a result of spatial geochemical analyzes, their sources were determined and their relationships were revealed. This study clearly reveals that trade routes in the past could be determined by identifying and correlating material sources with detailed spatial analyzes of the materials and minerals with LA-ICP-MS.

Galiová et al. (2010) have analyzed the dentin of a prehistoric bear (Ursus arctos) tooth by both LIBS and LA-ICP-MS (Fig. 4). From an archaeological point of view, it has been possible to reconstruct the etiology of the brown bear fossil, ie nutrition, health and migration, based on the measurements made. The Sr=Ca and Sr=Ba profiles in the sample showed seasonal fluctuations, proving that this bear migrated between the location of the hibernaculum and the location of the fossils. Combined with results from other techniques (i.e., study of cementation increases), it was concluded that this bear specimen most likely hunted before hibernating while foraging and migrating near a human settlement (where fossils were found). This study demonstrated that LIBS and LA-ICP-MS can be successfully applied as direct or complementary techniques in spatially resolved microchemical analysis of fossil specimens.

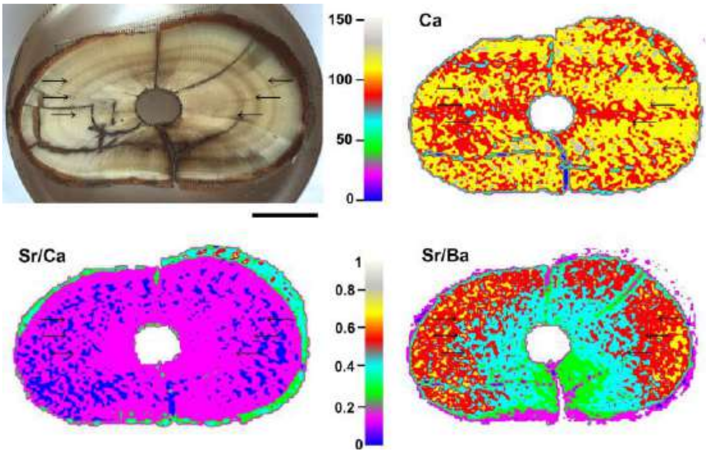

Fig. 4. Element mappings by using LA-ICP-MS and LIBS modified from Galiová et al. (2010)

FT-IR & RAMAN

FT-IR is an absorption technique which can be used to identify chemical compounds and it provides information about the chemical bonding in both inorganic and organic materials. Raman is an absorption technique, as FT-IR, for molecular identification based on inelastic scattering of monochromatic light. It allows to identify organic and inorganic materials and in the ceramic material studies it is used to detect minerals (Ricci, 2017).

Point analysis, line length analysis, and mapping could be done with Confocal Raman spectroscopy (CRS). Generally, point analysis is applied in mineralogical determinations. Eroğlu, Bilgen, Yetiş, Kadıoğlu, and Deniz (2021) conducted a coprehensive study on Determination of Construction Materials of Karabuk Ovacik Çukur Mosque and in their study, the pigment structures used in the wall paintings of the building were determined. In addition, the FT-IR analysis technique was used to determine the binder that allows the material used in the paint samples to adhere to the surface. The analyzes of the pigment and possible binders in the paint samples taken from the south and west walls were generated. Moreover, FT-IR and CRS analyzes were applied to the red, yellow, blue, green, brown, and black paint layer samples taken. In this study, data on aggregate binder ratios in mortar-plaster and gypsum, lime and cement contents in binders were obtained in detail. Rock types and building materials were characterized. In paints, pigments of natural and artificial origin and different types of organic binders (protein, oil and resin) are used; It is determined that modern paints are preferred in the decorations on the cement-based walls of the recent period.

CONCLUSIONS

Analytical techniques applied in archaeometric studies can be divided different subgroups in accordance with sample preparation process (the sampling strategy/non-destructive and destructive techniques), characterization and identification of the materials, conservation and preservation of the cultural assests, technology and techniques used.

Considering the economic costs, an analytical method suitable for the purpose should be chosen, and a significant light should be enlighted on the history, together with developing techniques and technologies, by conducting studies in collaboration with other important sciences such as biology, chemistry and geology.

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