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Application of PIXE using Al funny filter for cluster analysis of Byzantine amphorae from Beirut

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Abstract

Particle induced X-ray emission (PIXE) has been employed to analyze Byzantine amphorae found during an extensive campaign of excavations of the historical center of Beirut. Cluster analysis was applied to the PIXE results to verify the origin of 39 selected amphorae, based on their elemental composition. Archeologists have previously identified these amphorae to be Byzantine and have distinguished two different types. The cluster analysis of the studied samples revealed that they had the same origin and can be both attributed to the Beirut production. In this paper we demonstrate that the use of an appropriate pinhole filter, so-called "funny filter", as X-ray absorber, can offer considerable analytical advantages for the determination of elemental composition of thick target samples such as ancient ceramic materials. In fact, major, minor and trace elements can be obtained simultaneously in one run, instead of the two-run protocol employed in our previous work [M. Roumié et al., Nucl. Instr. and Meth. B 215 (2004) 196]. This can significantly reduce the acquisition time as well as the spectra treatment, especially when a large number of samples are to be studied.

Keywords: PIXE; Funny filter; Gupix; Cluster analysis; Archeometry

1. Introduction

The archeological excavations in the historical center of Beirut [1–4] have uncovered a considerable number of Byzantine amphorae (4th–7th century A.D.). From the typology and the visual aspect of the clay, archeologists have attributed these amphorae to Beirut production. Two main types were distinguished by their morphology. Unlike the Roman and medieval amphorae of Beirut, no kilns were found to characterize the Beirut production at the Byzantine period. Hence,

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determination of the chemical compositions may resolve the uncertainty on the possible origin of these objects. PIXE cluster analysis has already shown to be sensitive enough to attribute certain "carrot amphorae" found in southern France to Beirut production of the Roman period [5]. Other studies on Italian, Syrian, Mexican, African and American archeological materials [6–8] have revealed as well the capacity of PIXE to answer different archeological questions.

In a previous work, the typical two-run PIXE measurement was employed to measure: (1) the major elements using 1 MeV proton beam and without any X-ray absorber and (2) the trace elements using 3 MeV with Al foil as X-ray absorber [5]. Instead of this protocol, one-run PIXE protocol, using 3 MeV energy of proton beam and using Al funny filter, was applied this time for the determination of the elemental composition of thick target samples such as ceramic amphorae. This procedure allowed both the lighter ($10 < Z < 27$) and heavier elements ($Z > 26$) to be analyzed simultaneously. Indeed, the so-called funny filter (a filter with a tiny hole drilled at its center) is an X-ray absorber foil that is placed in front of the Si (Li) detector. This absorber has only a small effect on the less intense higher energy X-rays, but it stops most or all the intense low energy X-rays except for those that pass through the pinhole [9]. The result is a spectrum where both low and high energies X-rays are accumulated with an acceptable count rate (<1000 counts/s).

Two databases already existing and containing characteristic elemental compositions of Beirut productions of the Roman and medieval [4,5] periods will be employed in the present study. In fact, these two productions were certified by the existence of kiln materials. The Roman database was determined by the typical two-run PIXE at the accelerator laboratory of Beirut. The medieval one was established using "wavelength dispersive X-ray fluorescence" (WD-XRF) of the Lyon Laboratory. The two databases helped the attribution of the 39 chosen samples to a Beirut production when a cluster analysis based on 12 elements sufficient for a classification study, was performed. These 12 elements showed a good correlation between the data obtained by both PIXE and XRF techniques.

2. Archeological context and samples

The archeological excavations conducted in Beirut's city center, which were started in 1993 by a French team, allowed the unearthing of remains dating from the Persian era to the XIXth century. Among these remains, kilns from the Roman and medieval period were studied and attest to the production of pottery during these periods in Beirut. Concerning the Byzantine period (IV–VII centuries A.D.), a large number of amphorae were found and examined. Morphological and clay studies of some of these amphorae categorize them as local production, but no kilns were found that would define a Beirut production. Moreover, two main groups were identified: (i) type BEY 1 dated from the IV c. A.D. with some characteristics pertinent to Roman Beirut productions and (ii) type BEY 2 (V–VII c. A.D.) showing morphological difference with BEY 1. In addition, the type BEY 2 was also divided into three subgroups (A, B and C) that were also distinguished typologically. The amphorae selected for the study (39 samples) were chosen to represent as much as possible the pre-groups of the Byzantine period, already established according to typological criteria. Another set of ceramics (12 samples), dated from the Roman period, was also included in the measurements. These 12 objects underwent analysis by two different analytical techniques: XRF at the Lyon laboratory and PIXE, with its two different protocols, at the accelerator laboratory of Beirut. Therefore, any use of the already established databases, Roman and medieval, should be allowed after comparison and normalizing the elemental values to be used for cluster analysis.

3. Experimental

3.1. Sample preparation

The surface layers of the potsherds were removed, since they are likely to present alterations