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Journal of Archaeological Science 33 (2006) 1095–1104

Journal of Archaeological SCIENCE

http://www.elsevier.com/locate/jas

Pigment analysis in Bronze Age Aegean and Eastern Mediterranean painted plaster by laser-induced breakdown spectroscopy (LIBS)

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Received 21 February 2005; received in revised form 5 October 2005; accepted 26 November 2005

Abstract

Laser-Induced Breakdown Spectroscopy (LIBS) was used in the examination of Bronze Age painted plaster samples from several sites in the Aegean and Eastern Mediterranean. The elemental content of paint materials was determined in most cases leading to the identification of the pigment used in agreement with data from analyses of the same samples with other established techniques. The analyses demonstrate that a virtually non-destructive technique such as LIBS provides sufficient data for the elemental characterisation of painting materials while in parallel has the capability for routine, rapid analysis of archaeological objects enabling the quick characterisation or screening of different types of artefacts. This certainly shows an important way forward in technological studies of fragile and scarce archaeological material.
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Keywords: Pigment analysis; Plaster; Bronze Age Aegean and Eastern Mediterranean; Laser; LIBS

1. Introduction

From the constant growing literature on Aegean and Eastern Mediterranean painted plaster, a clear tendency for its importance and its value became apparent [36,29–31,68]. This is certainly the case for iconographical studies, especially after the excavations at Akrotiri (Thera – Greece), Tell el-Dab'a (Egypt), Miletus (Turkey), Tel Kabri (Israel) and now Qatna (Syria) which have brought to light very interesting decorated plaster with 'Aegean-style' painting schemes [7,57–59,28,36,38,42,43,68]. These iconographic studies, based on the wall paintings' themes and motifs, investigate a large variety of aspects related to the past society of which they were the products. These include, first of all, directly deduced aspects such as nature's faunal and floral presence (Figs. 1 and 2), clothing, jewellery and fashion, and architecture. Furthermore, indirect evidence for religious and ritual practices, social rank, status and prestige, and power symbolism has been revealed. Moreover, the investigation of the chronological framework of these fragmentary wall paintings adds to our knowledge about contacts between Bronze Age societies within the Aegean and beyond via trade, exchange and other forms of communication between them.

One of the major drawbacks of the study of Bronze Age Aegean painted plaster is the fact that the material is very fragmentary, most of which was not found in situ but mostly in secondary contexts [10,12] apart from Akrotiri on Santorini [32]. Furthermore, no iconographic or textual documentation of the craft in action has survived for the Aegean and Eastern Mediterranean world (in contrast to the situation in Egypt [4]). This makes wall painting scholars very reliant only on the information that can be extracted directly from the fragments themselves.

Since the first discoveries of Minoan and Mycenaean painted plaster around the end of the 19th and early 20th centuries at Tiryns, Orchomenos, Thebes and Myceanae and later on at Pylos on the Greek Mainland, at Knossos on Crete and at Phylakopi on Milos, iconographic and technological studies

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0305-4403/$ - see front matter © 2006 Elsevier Ltd. All rights reserved.
doi:10.1016/j.jas.2005.11.016

Fig. 1. Fragmentary wall painting from Palaikastro, Crete, representing blue and green plant motifs.

have gone hand in hand in order to understand what and how these prehistoric societies were able to produce some of the earliest and most significant works of art in Bronze Age Europe. These wall and floor paintings are claimed to be the first ones to have been executed in the *buon fresco* technique and this fact has now been clearly demonstrated in a recent PhD project to be published soon by A. Brysbaert (see also ref. [12]). This has serious implications, first, for the materials used and the techniques applied, second, for our general understanding and appreciation of very early developed technologies of these prehistoric societies and, most of all, for our understanding of the choices and limitations people had in executing their material culture [47].

Since the 1970s, this fragmentary material has been approached by a variety of angles in order to provide more information, more specifically by means of controlled and systematic analytical studies [18,66,67]. Mineralogical, chemical and elemental analyses of both pigments and plaster from a range of Bronze Age sites in the broader Mediterranean has been conducted in the past, even before the 1970s (summarized in fig. 1 in ref. [15]). However, most of these techniques require sampling or sample preparation. Nowadays, sampling becomes, rightly so, increasingly restricted so alternative methods need to be explored and evaluated in order to continue this important type of research (on all archaeological material) and to continue to produce unambiguous and valuable data to work with.

Fig. 2. Detail of a plant motif, viewed by raking light, from Tell el-Dab'a, Egypt.

The current paper is part of a larger research project, which sets out to analyse, in an interdisciplinary and comparative way, the evidence for an apparent but partial common technological background and knowledge between the Aegean and the East Mediterranean societies concerning the execution and applied materials involved in painting on plaster surfaces [12]. An essential component of this study was the investigation of samples and objects with a variety of analytical techniques. The information that is gained from analytical studies can be of great value in uncovering and understanding technological aspects of materials and manufacture used in production processes (les chaînes opératoires) of specific objects and artefacts [55,65]. In answer to such research questions, both qualitative and quantitative data aid towards a better understanding of the materials, their function, and the role they play on how human (inter)action is constructed with their environment and each other (technological transfer via craftspeople in time and space). Such data form the key to how trade, contacts and exchange on a regional and an interregional level can be detected as important aspects of the overall social dynamics of the Late Bronze Age societies in the Eastern Mediterranean.

The focus herein is on the use of laser-induced breakdown spectroscopy (LIBS) [2,3,26,52] to determine the elemental content of painted plaster from Eastern Mediterranean Bronze Age in an effort to identify the types of pigment and material used and to assess the potential of the technique in comparison with other analytical techniques such as Scanning Electron Microscopy coupled to X-ray analysis and micro-Raman microscopy which have been used in the broader study on technology of Bronze Age painted plaster (see above). Other non-destructive techniques such as X-Ray Fluorescence or (micro-)XRF [61,41], which can provide non-destructive in situ (micro)analysis of painted plaster and other materials, and alpha-PIXE [62], are being tested for their use in analysing art and archaeological materials [22–24,27], but are not discussed in detail in this paper since they were not part of the overall project conducted by one of its authors.

LIBS is a versatile technique that provides information on the elemental content of most materials since it covers, in theory, all elements of the periodic table. It appears as a highly promising analytical tool in a broad spectrum of applications because of its simplicity and modularity in instrumentation, speed of analysis and easy automation, features very important in industrial process control or screening applications which require handling of large numbers of samples in short times. Furthermore, portable versions of LIBS technology have been introduced recently with emphasis on field expeditions related to environmental and security applications [72,21,60]. Moreover, the LIBS technique is of extreme value in archaeological applications because it is virtually non-destructive [52]. This is a very positive asset to studying

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