Forensic Science International 245 (2014) 195–201

Contents lists available at ScienceDirect

Forensic Science International

journal homepage: www.elsevier.com/locate/forsciint

Soil examination for a forensic trace evidence laboratory – Part 2: Elemental analysis

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ARTICLE INFO

Article history:
Available online 14 October 2014

Keywords:
Laser induced breakdown spectroscopy
LIBS
X-ray fluorescence spectroscopy
XRF
Scanning electron microscopy/energy dispersive X-ray spectroscopy
SEM/EDX

ABSTRACT

Laser-induced breakdown spectroscopy (LIBS), X-ray fluorescence spectroscopy (XRF) and scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDX) are compared in terms of their discrimination power when applied to Australian soil specimens. SEM/EDX and XRF are frequently used in forensic laboratories for the elemental analysis of paint and glass, and for miscellaneous examinations. LIBS is an emerging technique for forensic applications, with a number of researchers promoting its use for the elemental profiling of glass fragments. In this study, 29 soil specimens were analysed, with 12 specimens coming from the Canberra area and the remaining 17 specimens from other sites around Australia. As very good discrimination results were obtained for each of the analytical methods, any of these elemental analysis techniques, available in a trace evidence laboratory, could be used as part of a wider examination protocol to differentiate soil specimens.

1. Introduction

Soil may be encountered in many different situations in forensic science, for example: clothing and shoes from a suspect alleged to have stepped in a garden bed prior to entering the victim's house; a dirty shovel recovered from a suspect's house alleged to have been used to bury materials; and soil from a suspect's vehicle that may have been at a burial site [1–3]. Ultimately, soil can be used as evidence to indicate or preclude an association between a suspect, a victim or an object with a particular scene, assist with locating and identifying the scene of a crime, or contribute to forensic intelligence.

The forensic analysis of soils developed into a trace evidence sub-discipline that was commonly carried out in forensic science laboratories; however, in Australia and elsewhere, interest in the examination of traditional trace evidence waned from the late 1980s and especially through the 1990s. In contrast with the general decline in soil examinations across operational forensic laboratories, there has been an increased interest in the forensic applications of soil examination and, more generally, in forensic geosciences in organisations whose core interest is geology and soil science.

In our previous paper [4], we proposed a model for how a partnership, involving roles for trace evidence examiners and forensic soil specialists, might work in practice. The ultimate aim of this partnership would be to increase the use of soil examinations as a forensic science discipline. The role of the trace evidence examiner in this model would be to screen and triage soil samples using techniques routinely used in forensic laboratories. In our first paper in this series, we discussed the analysis of colour. In this paper we turn our attention to the use of elemental composition.

The use of scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDX) is widespread in most trace evidence laboratories for the examination of gunshot residue (GSR) particles and for the elemental analysis of microscopic materials [5]. A closely related technique, X-ray fluorescence spectroscopy (XRF), is also frequently used in trace evidence laboratories for the elemental analysis of paints, liquids and miscellaneous substances [6]. Laser induced breakdown spectroscopy (LIBS) is an emerging technique for forensic applications, with a number of researchers promoting its use for the elemental profiling of glass fragments [7].

The purpose of this paper is to determine whether it is feasible to apply typical elemental profiling techniques used in the analysis of other forms of trace evidence for the screening of soil prior to being forwarded for more specialised examinations. In this paper, we report on an investigation into the application of LIBS, XRF and SEM/EDX for the discrimination of soils using a selection of Australian soils as a test sample set.

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http://dx.doi.org/10.1016/j.forsciint.2014.10.018
0379-0738/

2. Materials and methods

The soil specimens used in this study consisted of samples collected from six sites around the Canberra area and samples previously collected by the Commonwealth Scientific and Industrial Research Organisation (CSIRO) and stored as part of the CSIRO National Soil Archive. At each Canberra area site, a five-metre by five-metre square grid was established with 9 locations at regular intervals within the grid determined as collection positions. At each position, the surface debris and leaf litter were removed then samples of surface soil (0–5 cm depth) and sub-surface soil (5–10 cm depth) collected. As the scope of this research did not include an assessment of the homogeneity of soil across a domain typical of a crime scene (for example, the few square metres applicable to a clandestine grave site) specimens were combined for each collection position within the grid, resulting in 2 soil specimens per Canberra area site (one combined surface sample and one combined sub-surface sample). The Canberra area soils were rudosols and chromosols from volcanic mountains, alluvial fans, granitic material and metasediments [8].

In addition, seventeen soil specimens were selected from the CSIRO National Soil Archive, covering Queensland (Qld), New South Wales (NSW), South Australia (SA) and the Northern Territory (NT). The collection method for the CSIRO samples varied; however, all soil samples were surface soil (0–5 cm depth). The soil specimens from the CSIRO National Soil Archive included sodosols, tenosols, kandosols, vertosols, dermosols and calcarosols from plateaus, plains, rises, low hills and alluvial plains. A total of 29 soil specimens were examined in this study. Analysis of these soil specimens using infrared spectroscopy and visible microspectrophotometry has been discussed previously [4]. Table 1 provides additional details for the soil collection sites used in this study.

The soil specimens were oven dried for 24–48 h at 47 °C then lightly crushed, using a mortar and pestle to break up any agglomerates, and dry sieved [9]. The <38 μm fraction was collected for analysis. No attempt was made to remove any organic content. Previous work by the authors detail the rationale for this soil preparation procedure [4]. Fig. 1 shows a representation of Australia and the approximate locations of the soil collection sites.

Fig. 1. Map of Australia [10] with the approximate sampling locations for the soil specimens used in this study.

2.1. LIBS method

For analysis using LIBS, approximately 10 mg of soil sample was compressed at approximately 2 tonnes using a 13 mm die set, creating a thin, indurate soil disc. The discs were mounted on carbon tape mounted on a glass slide and presented to the LIBS. A Photon Machines LIBS instrument using Chromium software (version 2012.3.19.1) with a Nd:YAG laser (QI Quantel laser – 54.8 mJ) operating at 266 nm was used for the LIBS data collection. Data collection was optimised using the following acquisition parameters: laser power of 100%; spectral delay of 2 μs; repetition rate of 0.67 Hz; spot size approximately 700 μm; shot count of 5 with 2 cleaning shots; and an argon purge. Line analysis of the soil disc was conducted, resulting in an average of 7 acquisition spots. LIBS analysis was conducted in triplicate for each soil sample. The LIBS spectral data was analysed by determining the

Table 1
Details on the soil collection sites used in this study.

Soil site	State*	Area	Longitude/latitude (°)	Landscape
Site 1	ACT	Mount Ainslie	149.16393/−35.26905	Large, steep slope with native dense bushland vegetation
Site 2	ACT	Coppins crossing	149.02872/−35.30909	Flat, cleared land with grasses and introduced plants
Site 3	ACT	Tharwa	149.04715/−35.47497	Flat, cleared farmland with grasses
Site 4	ACT	Gordon	149.08511/−35.47252	Hill top with small native shrubs and grasses
Site 5	NSW	Burra	149.18990/−35.66950	Cleared hillside with grasses
Site 6	NSW	Cuumbeun Nature Reserve	149.27187/−35.35716	Hillside with native open-forest vegetation
Site 7	Qld	Tara	150.0344/−27.5220	Mid-dense, native vegetation
Site 8	Qld	Tara	150.0317/−27.5262	Dense, native vegetation
Site 9	Qld	Buchanan	145.8511/−21.4651	Very sparse, native vegetation
Site 10	Qld	Aurukun	143.0844/−13.6485	Very sparse, native vegetation
Site 11	Qld	Weipa	142.4678/−11.6485	Sparse, native vegetation
Site 12	Qld	Townsville	146.6511/−19.3151	Very sparse, native vegetation
Site 13	Qld	Townsville	146.6844/−19.2818	Very sparse, native vegetation
Site 14	SA	Cooper Pedy	134.9896/−29.0341	Very sparse, native vegetation
Site 15	SA	Cooper Pedy	134.4680/−28.2080	Very sparse, native vegetation
Site 16	SA	Penola	140.7741/−37.3274	Mid-dense, native vegetation
Site 17	SA	Millicent	140.3900/−37.6058	Cleared farmland
Site 18	SA	Cultana	137.6850/−32.9108	Mid-dense, native vegetation
Site 19	SA	Cultana	137.5766/−32.9355	Mid-dense, native vegetation
Site 20	SA	Oodnadatta	135.2691/−27.9919	Sparse, native vegetation
Site 21	NT	Alice Springs	133.2077/−24.6066	Mid-dense, native vegetation
Site 22	NSW	Temora	147.4743/−34.4684	Cleared land
Site 23	NSW	Rannock	147.2577/−34.6658	Cleared farmland

* ACT - Australian Capital Territory, NSW - New South Wales, Qld - Queensland, SA - South Australia, NT - Northern Territory.