



Quantitative chemical profiling of coal using core-scanning X-ray fluorescence techniques



Sarah J. Kelloway^a, Colin R. Ward^{b,*}, Christopher E. Marjo^a, Irene E. Wainwright^a, David R. Cohen^b

^a Mark Wainwright Analytical Centre, University of New South Wales, Sydney, NSW 2052, Australia

^b School of Biological Earth and Environmental Sciences, University of New South Wales, Sydney, NSW 2052, Australia

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ABSTRACT

A study has been carried out to test the possibility of measuring detailed quantitative profiles of the abundance of different inorganic elements through exploration cores of coal seams using automated energy-dispersive X-ray fluorescence (ED-XRF) core scanning instrumentation. Such an approach has potential application for rapidly determining the distribution of mineral matter in a coal seam, identifying horizons at which particular elements (such as phosphorus) may be concentrated, evaluating in detail the relationships between different elements in the coal, and maximising the data available to guide broader-scale sampling for conventional analysis programmes.

Profiling was carried out on a series of segments from a 60 mm diameter core of the Goonyella Middle seam in the northern Bowen Basin of Queensland, using an Itrax core scanner (Cox Analytical, Sweden). The scanning process included high resolution optical imaging, X-radiography, and ED-XRF spectroscopy covering the principal major elements, with XRF spectra being obtained at intervals of 200 μm along the axis of each core. A series of calibration curves derived from separately-scanned pressed pellets of reference coals was used to determine the concentration of each element for each step in each sample, allowing a set of quantitative element profiles to be created for each core segment. These were evaluated in conjunction with the relevant X-radiographs and optical images to provide an integrated basis for assessing the variations in inorganic element characteristics through the core sections. The results were also compared to conventionally-determined chemical and mineralogical data for a representative core, to confirm the validity of the quantifications developed.

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1. Introduction

Although the organic matter is a key component for many purposes, the mineral matter in coal, represented by admixed mineral phases and non-mineral inorganic elements (Ward, 2002), is also a significant factor in understanding the processes of coal formation, and in assessing the suitability of individual coal deposits for different industrial purposes. Elements such as Na, Ca, K and Fe, for example, may lower the fusion temperatures of coal ash, leading to build-up of slags and other deposits in combustion applications (Byers, 1996; Creelman et al., 2013; Suárez-Ruiz and Ward, 2008), and sulphur, which may occur in the organic matter as well as in sulphide and other mineral particles, represents a potential source of atmospheric pollution from coal utilisation processes. Sulphur and phosphorus in coal may also be incorporated as contaminants of iron and other coal-derived metallurgical products.

The inorganic chemistry of geological cores taken from coal seams has traditionally been determined from bulk sampling operations

(Standards Australia, 1993; Thomas, 2001), with length-based portions of the core being ashed and the ash analysed using wavelength-dispersive X-ray fluorescence (WD-XRF) spectrometry or inductively coupled plasma (ICP) techniques. These techniques provide low limits of detection and high levels of precision, and, if appropriate procedures are followed for sampling and analysis, the results provide useful information to characterise the large masses of coal that are mined and used from individual seams and deposits.

The distribution of mineral matter within a coal seam is not uniform. Coal beds are typically composites of separate and often quite different layers built up during the precursor process of peat formation, and may also be affected by a range of post-depositional mineralisation processes (Ward, 2002). There is thus an inherent trade-off between the length of the core segments sampled for analysis and the spatial resolution required to evaluate the distribution of the different inorganic components within the seam. Sampling for quality assessment in resource studies is usually based on relatively thick intervals of coal seams, such as macroscopically distinct coal plies or seam subsections that might be extracted separately during mining operations. Sampling of smaller intervals may be undertaken for research purposes (e.g. Dai et al., 2012b), but even then the segments studied are typically centimetres to tens of centimetres in length. As the intervals sampled

* Corresponding author. Tel.: +61 2 9385 8718.

E-mail address: c.ward@unsw.edu.au (C.R. Ward).

become smaller, the overall cost of analysis per metre of coal also inherently increases.

Recent developments in instrumentation mean that the analysis of drill cores can be performed rapidly, at millimetre to sub-millimetre scales, using one of several automated, laboratory-based, core-scanning energy-dispersive X-ray fluorescence (ED-XRF) spectrometer systems. Such units are able to evaluate the variation along a core in the concentration of multiple elements, with detection limits in the ppm range depending on the element concerned. Laboratory core-scanning systems can process cores up to ~1.8 m in length; they may also allow ED-XRF analysis to be supplemented by photographic and radiographic imaging, to relate the analysis profile to other geological features of the drill core. The volume of data generated by such systems is significantly higher than that from traditional bulk sampling. For example, the evaluation of a core 1.8 m in length at intervals of 100 µm can potentially result in 18,000 separate analyses evenly spaced along the core, each covering a large number of major and/or trace element components.

The application of these techniques to the analysis of coal cores is not without challenges. The problem of rough surfaces, for example, may be significant; air between the sample surface and the X-ray detector attenuates the fluorescing X-rays, and changing distances between the detector and sample may lead to the false impression that the elemental concentration is changing along the core. Some automated core scanners have the capacity to maintain a constant distance between the detector and the sample surface during measurement, which can reduce this effect. Such problems may be minimised if it is possible to provide a completely flat surface for exposure to the scanning system, i.e. to carefully cut the core down its length in a way that avoids fracturing and crumbling. However, this may be difficult with coal, where weak and brittle organic material is interlayered with hard mineral-rich phases in the cored seam section. To allow for subsequent core logging and to keep open options for sampling and analysis programmes, coal cores should ideally be presented to the instrument without splitting. In such cases the XRF spectrum from the curved external surface would need to be measured, and suitable data processing applied, if necessary, to reduce any artefacts arising from the rough surface of the as-drilled core.

The inorganic elements of interest in coal are usually present at low overall concentrations; they can thus be difficult to quantify using an energy-dispersive detector, due to a relatively poor energy resolution, and to background interference from the source X-rays and other scattering processes. Although some variation may exist in the chemistry of the individual macerals (e.g. Li et al., 2010; Ward et al., 2005), the organic matrix in coals of a given rank is relatively uniform in composition; attenuation due to X-ray absorption by the surrounding elements in the organic matter may therefore not vary greatly within individual seams, and if so this will aid the creation of matrix-matched calibration standards and allow quantification of element concentrations from the spectral data (Wilhelms-Dick et al., 2012).

The purpose of the present study was to evaluate the capability of core-scanning ED-XRF instrumentation for use in coal quality assessment, and to determine whether rapid, quantitative chemical profiling is possible on an unmodified coal core. As part of the process the project aimed to develop and test a series of calibration curves, reported in a companion study (Kelloway et al., 2014), as a basis for quantitative determination of element concentrations in small core intervals, and to evaluate the results in the light of complementary lithological, chemical and mineralogical data.

A number of different coal samples were cross-analysed in the companion study using a variety of techniques (Kelloway et al., 2014), and the results integrated to develop a calibration system for conversion of the raw X-ray count data from the core scanner to element concentration profiles. As an extension of that process the present study was intended to validate use of the core scanner for non-destructive analysis of as-recovered coal seam cores, and to determine whether fine-scale

element distributions within the core can be quantified at useful detection limits. Such scanning has potential application for rapid assessment of elemental profiles in coal seam cores, identifying horizons at which particular elements (such as phosphorus) may be concentrated, evaluating in detail the relationships between different elements in the coal, maximising the data available to guide broader-scale sampling for conventional analysis programmes, and identifying factors affecting the analytical results. In conjunction with X-radiography, carried out as part of the scanning process, the results may also assist in the design of crushing and preparation processes to provide optimum beneficiation of the coal seam.

2. Materials and methods

2.1. Samples studied

2.1.1. Coal core samples

A coal seam core from the Goonyella Middle seam, an economically-significant coal of Permian age in Moranbah Coal Measures of the northern Bowen Basin, Australia (Mallett et al., 1995), was used as a basis for testing the applicability of the core-scanning XRF technique. This coal is of high volatile bituminous rank, with a mean maximum vitrinite reflectance of 1.1 to 1.2%, and had previously been subjected to a series of core-scanning studies using a hyperspectral infra-red imaging system (Fraser et al., 2006). Low-temperature oxygen-plasma ashing and X-ray diffraction analysis conducted to support that study (Henwood, 2004) indicate that the coals of the seam typically contain 13–23% mineral matter, and that the mineral matter is made up mainly of quartz (15–60%), kaolinite (20–50%) and interstratified illite/smectite (15–30%), with minor proportions of siderite (3–6%) and traces of ankerite in some parts of the seam.

Although the analyses for the previous work programme required relevant core segments to be broken and crushed, making them unsuitable for further scanning, a number of intervals of otherwise unbroken coal core, located between the sections sampled, were used to test the capability of the core scanner in the present study. Seven such segments of coal, each 100 to 150 mm in length (Table 1), were subjected to analysis using ED-XRF core scanning techniques. These segments were made up of interbedded dull (inertinite-rich) to bright (vitrinite-rich) coal lithotypes, with varying proportions of admixed mineral matter. The cores were analysed whole, approximately 60 mm in diameter; with one exception (segment 9; see below), no additional preparation or splitting was undertaken prior to the scanning process.

2.1.2. Calibration standards

In addition to the core segments, a number of powdered reference coals were used to provide a basis for calibration of the data obtained from the core scanning process (Table 2). These and the associated calibration process for the core scanner are discussed separately by Kelloway et al. (2014). The samples used for calibration included an

Table 1
Depth intervals represented by core segments, Grosvenor GD-104.

Segment number	Core depths (m)		Segment length (m)	Segment lithology
	From	To		
1	291.64	291.74	0.10	Coal, dull and bright, silty bands
2	292.44	292.59	0.15	Coal, dull and bright, silty bands
3	292.84	292.94	0.10	Coal, dull, minor bright bands, stony
4	293.37	293.51	0.14	Coal, dull, minor bright bands, stony
6	294.19	294.34	0.15	Coal, dull and bright, interbedded
7	290.82	290.95	0.13	Coal, bright, minor dull bands, stony at base
8	289.02	289.14	0.12	Coal, mainly dull, stony
9	293.03	293.06	0.03	Coal, mainly dull

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