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Application of high-resolution X-ray microcomputed tomography for coal washability analysis

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ABSTRACT

Float and sink testing is the standard laboratory method for determining the coal washability characteristics using heavy liquids. In this paper, we describe an alternative technique for determining coal washability using High-Resolution X-ray Microcomputed Tomography (HRXMT). Specifically, the application of HRXMT and specialized software have enabled us to determine the particle characteristics of coal samples with the considerable details needed for building washability analysis. Coal samples taken from the feed streams to the dense medium circuits at two plants in Australia were obtained, characterized and used in the analysis. The coal samples were fractioned into the size and density fractions using sieves and heavy liquids as per the Australian Standard AS 4156.1-1994. The fraction-averaged densities were determined using helium gas pycnometry. The float and sink results were obtained for calibration and validation of the HRXMT results. Our results show that HRXMT can be used to perform coal washability analysis. It provides a new methodology for non-destructive and non-toxic determination of coal washability data. It is safe and fast. Significantly, the successful applications of X-ray computed tomography to perform coal washability analysis would allow for developing an on-line coal washability monitoring system, which would not be achievable with the traditional float-and-sink analysis.

1. Introduction

The washability curve for coal is the basic indicator of the coal quality and its amenability to cleaning. It determines whether the coal can be cleaned to meet the necessary product specifications, or whether it needs further processing (e.g., size reduction and separation). The washability curves are currently obtained by sink-float analysis as specified in many standards, e.g., Australian Standard AS 4156.1-1994. It uses a number of mixtures of organic liquids, which are toxic, environmentally hazardous as determined by the Australian Safety and Compensation Council. They may also interfere with the properties of interest for a coal producer or end user. The safety and technical concerns associated with their use have been recognized by the coal industry for some time. Several studies into replacing the liquids or finding alternative methods have been conducted and funded by the Australian Coal Research Program (ACARP), all indicating advantages and disadvantages. Zirconium dioxide, cesium formate, potassium formate, perchloroethylene free organics and re-engineered organics appear as the most likely options for the replacement.

The principle of sink-float analysis is nothing more than density fractionation. However, it is a very time-consuming method, and cannot be used for on-line real-time analysis and control. On the other hand, the scanned sections produced by X-ray Computed Tomography (XCT) are maps of the density variation in the scanned volume (voxel) which can be determined in a matter of minutes. Reconstruction of 3D particle populations from XCT scans provides sufficient information not only to construct the washability curve for the coal sample but also provides information on the particle size and grain size distribution of mineral matter grains present in the coal particles (Lin et al., 1991).

XCT had its origin in the medical service and has now been applied to a wide variety of non-medical and industrial applications. XCT techniques have an inherent advantage in providing very detailed images of the internal structures of opaque materials in a non-destructive manner and recreating 3D images without destroying the original object. The use of XCT for coal washability analysis has been demonstrated in previous publications using both milli XCT scanners for coarse coal fractions (> 9 mm) and micro XCT scanners for fine coal fractions (< 1 mm) (Lin et al., 1991, 2002; Miller and Lin, 2018). The prefix milli- and micro- are used to indicate that the pixel sizes of the cross-sections are in the millimetre and micrometre ranges, respectively. The micrometre sizes have also resulted in the term "high-resolution X-ray microtomography", abbreviated as HRMXT, and similar

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terms. HRMXT is preferably used in this paper. It is expected that XCT washability analysis can help avoid the major concerns about the traditional float and sink analysis such as the health, safety, and environmental issues associated with the use of heavy liquids. The time required for the XCT analysis is substantially less than traditional sink-float analysis. It would be excellent if XCT washability analysis could become a standard for the coal industry in the near future.

In this paper, we describe and present the latest results of the application of the HRXMT technology to obtain the washability analysis of the Australian coal samples using a new HRXMT scanner, the VersaXRM-500 from Xradia, USA (now Zeiss). The Xradia VersaXRM presents the latest generation of HRXMT microscopy with 3D sub-micron imaging with true spatial resolution and largest working distance with zoom-in and out choices on small and large samples. Together with our specialized software developed in-house, the coal washability characteristics in considerable details are shown to be vital for developing a reliable HRXMT analysis of coal washability.

2. Materials and methods

2.1. Coal samples and size characterization

Two different coal samples taken from the feed streams to the dense medium circuits (DMCs) were received from two CHPPs (coal handling and preparation plants) in Australia. They are described as Coal A and Coal B. Coal samples were dried, sized to seven size fractions (16–32 mm, 16–8 mm, 8–4 mm, 4–2 mm, 2–1.4 mm, 1.4–0.85 mm, and 0.85–0.5 mm) using the series of sieves and procedures as specified in the Australian Standard AS 4156.1-1994.

2.2. Float and sink experiments

Float and sink experiments were conducted using the procedure described in the Australian Standard (AS 4156.1-1994). The aim was to obtain the washability data for calibrating and validating the HRXMT washability analysis. Organic liquids used to prepare the density intervals required for the experiment were bromoform (97% stab. with 3% ethanol) with a relative density of 2.81, tetrachloroethylene (99%) with a relative density of 1.62 and white spirit with a relative density of 0.78. Eight relative density intervals as specified as Australian Standard 4156.1-1994 were prepared and used: −1.3, 1.3–1.35, 1.35–1.4, 1.4–1.5, 1.5–1.6, 1.6–1.8, 1.8–2.1, +2.1. The float and sink experiments were conducted for seven size fractions, each with eight density intervals. The float and sink products were then dried and stored in sealed plastic containers for further use.

2.3. Determination of coal density and ash contents

The density of each of the float and sink products was further measured by gas pycnometry using an AccuPyc II 1340 Pycnometer from Micrometrics and helium gas together with a high precision balance. Each of the measurement was repeated at least 3 times and the averaged data were obtained and used. To obtain good statistics, the sample masses for the pycnometer measurements were chosen as heavy as possible, within the interval from 5 g to 50 g depending on the sample availability. Ash contents were determined according to the Australian Standard 1038, Part3-1979 “Methods for the analysis and testing of coal and coke, Part 3- Proximate Analysis of hard coal”. The single furnace method was used with Furnace Model UP150 from S.E.M. Ltd. The furnace temperature was raised to 815 °C in 100 min and maintained at this temperature for an additional 100 min. The ash values were determined for each of the density fractions and size fractions with weight from 1.0 to 5.0 g depending on the samples availability.

2.4. Principles of XCT for coal washability analysis

The basic principles of XCT are well-documented (Herman, 1980) and only a short overview is provided herein. The XCT scanning analysis is to examine the energy intensity, I, of an X-ray beam signal that is emerging from an object as a function of the incident X-ray energy, I_0, and the linear attenuation coefficient, μ, which relates to the local mean density of a small volume (voxel) within the object. For example, dense objects (such as minerals) capable of strongly absorbing X-rays appear white on CT images while light, soft materials (such as coal or liquid) appear grey and cavities filled with air (allowing X-rays to pass through) appear black. XCT works based on material density differences and is, therefore, suitable for determining densities of coal particles needed for analysing coal washability (without destroying the internal structure of coal samples).

The measurement quantity of XCT, $\ln(I/I_0)$, is equal to the summation of the X-ray attenuation coefficients of the material at each point along the incident line which can be measured by using an appropriate detector allowing for the measurement of the X-ray attenuation. The measured projections are manipulated by the computer software according to a specific reconstruction algorithm to produce a two-dimensional map of X-ray attenuation coefficients of the irradiated cross sections. Differentiation of features within the sample is possible because μ at each point depends directly on the electron density, the effective atomic number, Z, of the material comprising the sample, and the energy, E, of the X-ray beam. A simplified equation that describes the approximate relationship between these quantities is described as follows (McCullough, 1975; Powsner et al., 2013):

$$\mu = \rho\left\{a + b\frac{Z^{3.8}}{E^{3.2}}\right\} \quad (1)$$

where ρ is the density of the phase, a is a variable which weakly depends on E, and b is a constant. Knowing μ by measurements can be determined. For the case of transmission tomography such as XCT, the modern XCT scanners are capable of discriminating between values of μ that differ by as little as 0.1%.

2.5. Special HRXMT device for X-ray microscopy: the Xradia VersaXRM-500

Fig. 1 shows visual external and internal views of the VersaXRM-500 from Xradia, USA (now Zeiss International) used in this study. The key components are the X-ray chamber, the power supplies, the PC for running the device and processing the images and terminal panel. The device is enclosed by an insulated steel and lead-lined framework that provides protection from harmful X-ray radiation. The radiation around the device is constantly monitored by the X-ray dose meters and badges as required by the Queensland Government. The unit is equipped with safety interlocks, which automatically turn off the X-ray source when one of the access doors is opened. The light tower also visually reports status conditions, i.e., if the red light on the top is on, the X-ray source is on and X-rays are present in the enclosure. A Window 7 based computer with a monitor located outside the enclosure (the terminal panel) is used to run the device and the basic steps of processing the images. A visual light camera located behind the sample stage, at the rear of the enclosure is used to position the sample, detector, and X-ray source, via the camera window on the terminal panel. The device is also connected to an ergonomic station with a joystick for the visual positioning using the camera.

The majority of conventional X-ray (micro and nano) computed tomography systems are projection-based and use a micro or nanofocus X-ray source to project a geometrically magnified image of the sample on a large-pixel flat panel detector. They impose a significant limitation of sample size. However, this limitation is overcome in the VersaXRM-500 by the special Xradia design with a two-stage magnification