Porosity measurement using X-ray microtomography and mercury intrusion porosimetry: Assessment of their correlation in South African coal

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Summary: Micro-focus X-ray tomography is widely being applied to assess porosity also in coal, but needs to be calibrated and correlated with the well-established and traditional Mercury porosimetry method in order to be accepted, within limits, to provide an accurate valuation of the pore space. This study assesses the two techniques as being applied to South African coal samples.

1. INTRODUCTION

Mercury intrusion porosimetry (MIP) has its practical importance to obtain physical information in porous media such incremental, cumulative and differential pore size distributions for mesopore and macropore ranges (5 orders of magnitude from 0.4 mm to less than 4 nm.) in addition total pore area, pore area distributions, median pore diameter determinations, bulk density, apparent density and porosity [1]. MIP is widely and generally accepted as the recognized baseline laboratory analytic technique to which e.g. helium pycnometry, light optical microscope (LOM) images, scanning electron microscope (SEM) and radiation scattering method are compared and calibrated to [2]. However, although mercury porosimetry detects open porosity with high precision, its application towards the bulk porosity determination is destructive, it measures not the actual inner size of a pore but only the largest entrance towards a pore as well as no information is obtained from isolated/closed pores or pore connectivity [2][3][5][6].

Klobes et al (1997) used X-ray microtomography (µXCT) in rock porosity studies before and after MIP to determine the effect of MIP on the physical characteristics [4]. Kovářová et al (2012) compares the possibilities MIP and µXCT for detailed identification of the influence of the weathering processes of sandstone internal structures which includes pore size determination [5]. Cnudde et al (2009) made an assessment comparing MIP and µXCT- also evaluating the measuring range of the two techniques including intrusion porosimetry in building stones and concrete. Importantly, Cnudde concluded that direct correlation of the obtained test results from µXCT and MIP on total porosity and pore-size distribution is difficult and in some cases showed large discrepancies in porosity values. However, µXCT provide supplementary and valuable data about the pore-size distribution in the range above 10 µm but the combined µXCT/MIP techniques allows analysis in the pore-size range which includes the air voids can thus be of high value for durability studies.

It is only after the introduction in 2011 of µXCT in South Africa that South African coal samples were extensively investigated using µXCT - also in evaluation of coal microstructure, which includes porosity and cracks on coal breakage characteristics [7]. Coetzee (2015) used both MIP and µXCT to quantify the degree of swelling of large particles, while comparing them in the study of conventional swelling characteristics of powdered coals originating from South African coal mines [8]. It is thus foreseen that the development and utilisation of µXCT will become more sophisticated also to obtain some physical properties (eg porosity) of coal without using the traditional MIP method. This study focus on the calibration and correlation of µXCT with the well-established and traditionally utilised MIP method in order to be accepted, within limits, to provide an accurate evaluation on porosity of South African laboratory prepared coal samples.

2. EXPERIMENTAL METHOD

The experiments were performed on several well prepared (spherical shaped) South African low volatile coal samples at the South African Nuclear Energy Corporation (Necsa) on its MIP and Micro-focus X-ray Radiography

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and Tomography (MIXRAD) instruments. Due to the non-destructive nature of µXCT, the experiments were conducted first using µXCT at a potential and current of 100kV and 100mA respectively. Thereafter the samples were subjected to MIP.

3. RESULTS

Preliminary averaged MIP results of 5.7 ± 0.5% bulk porosity indicating a relatively small porosity fraction for the low volatile coal samples. The µXCT 2D virtual slice in a 3D tomogram of a sample is being displayed in Fig. 1 showing also the distribution of pores (up to 5microns) in a thin section being highlighted in black. A full analysis, correlation, comparison and evaluation of MIP and µXCT in terms of porosity results obtained, will be presented.

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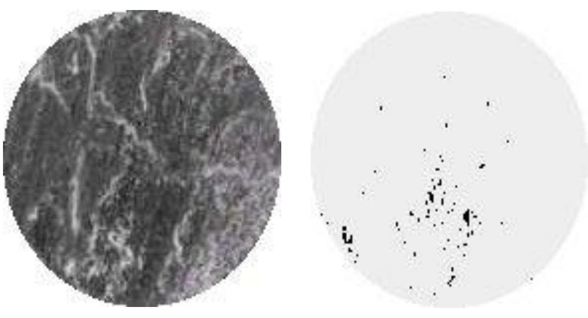

(a) (b)

Figure 1: Non-destructive power of µXCT visualizing porosity (>5micron) in a 2D slice of a 3D tomogram of a low volatile South African coal sample (a) 2D slice (b) Electronic enhancement with porosity in black