

Inter-laboratory comparison II: CO₂ isotherms measured on moisture-equilibrated Argonne premium coals at 55 °C and up to 15 MPa

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Received 5 September 2006; received in revised form 22 January 2007; accepted 26 January 2007

Available online 1 February 2007

Abstract

Sorption isotherms, which describe the coal's gas storage capacity, are important for estimating the carbon sequestration potential of coal seams. This study investigated the inter-laboratory reproducibility of carbon dioxide isotherm measurements on moisture-equilibrated Argonne premium coal samples (Pocahontas No. 3, Illinois No. 6, and Beulah Zap). Six independent laboratories provided isotherm data on the three moisture-equilibrated coal samples at 55 °C and pressures up to 15 MPa. Agreement among the laboratories was good up to 8 MPa. At the higher pressures, the data among the laboratories diverged significantly for two of the laboratories and coincided reasonably well for four of the laboratories.

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Keywords: CO₂ sorption; CO₂ storage capacity; CO₂ isotherm; Argonne premium coal; Carbon sequestration

1. Introduction

Carbon dioxide storage in coal seams has recently received increased attention as a potential option to

reduce greenhouse gas emissions to the atmosphere. Accurate measurements of CH₄ and CO₂ sorption isotherms are vital for the optimum development of techniques to either sequester CO₂ or to combine CO₂ storage with an enhancement of CH₄ recovery (Clarkson, 1999; Gasem et al., 2001). For a given coal seam, sorption isotherm measurements provide information about the storage capacity, the overall economics of the process, and the types of operating

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conditions that can be used (White et al., 2005). The storage capacity of a coal seam is traditionally estimated from manometric, volumetric, or gravimetric isotherm measurements (Ruppel et al., 1972; Lu et al., 1995; Humayun and Tomasko, 2000). Since there is no standard procedure or technique for measuring the isotherm, results obtained from different laboratories need to be compared to determine their accuracy. In order to better understand the variation in estimates of the storage capacity of coal seams, the U.S. Department of Energy-National Energy Technology Laboratory (DOE-NETL) initiated an inter-laboratory study where the sorption isotherms measured by laboratories throughout the world were compared. In that first study, the CO₂ sorption isotherm measurements on dry coals at 22 °C were compared (Goodman et al., 2004). The isotherm data varied widely among the laboratories, with the greatest variation being observed for the lower rank coal samples. The results of that study suggested that the drying procedure for removing moisture from the coal samples played a large role and that the isotherms reflected different degrees of residual moisture content. The development of a strict procedure for drying coals in order to obtain isotherm measurements on dry coals was recommended.

The DOE-NETL then initiated a second inter-laboratory isotherm comparison of coals where CO₂ sorption isotherms were collected on moisture-equilibrated coals at temperatures and pressures relevant to CO₂ sequestration. Results of this second comparison among laboratories for moisture-equilibrated coals are reported here. Each laboratory used the same coal samples and followed the same general procedure; however, each laboratory used their own apparatus and isotherm measurement technique. Currently, there is no standard CO₂-coal isotherm method as assumed by ASTM procedure E-691 (ASTM E 691–99, 2000). Thus, these results should be considered a discovery process which was undertaken to determine if stricter control of variables is needed to obtain good inter-laboratory precision for CO₂-coal isotherm measurements. The data are examined for possible sources of error, but only as a guide for future investigations, not to provide unequivocal answers to specific questions. Our intent is to address the issue of whether isotherm measurements reported by laboratories worldwide are comparable. This work provides guidance for estimating the reproducibility that might be expected when comparing published sorption isotherms on moisture-equilibrated coals from different laboratories.

2. Experimental

Approximately a dozen laboratories that are measuring sorption data worldwide were invited to participate in this study. Because of their funding or time constraints, the following six independent laboratories volunteered to participate at their own expense, excluding the cost of the coal samples: CSIRO, Australia; RWTH Aachen University, Germany; Oklahoma State University, USA; TICORA Geosciences, USA; University of British Columbia, Canada; and DOE-NETL, USA. Table 1 lists the participants and their affiliations. Located throughout the world, the participants included academic, government, and industrial laboratories. The data each laboratory contributed are reported anonymously, being referred to as laboratory 1, 2, 3, 4, 5, or 6 throughout the paper, an order that is different from the listing in Table 1.

For this study, DOE-NETL chose a set of three coal samples from the Argonne Premium Coal Sample Program: Pocahontas No. 3 (low volatile bituminous), Illinois No. 6 (high volatile bituminous), and Beulah Zap (lignite) (Vorres, 1990). The Argonne premium coal samples provide the research community with the highest quality samples for basic research. Samples for a specific coal are as chemically and physically identical as possible. The coals are well-characterized and are stable over long periods of time because they were prepared and stored under inert gas. This ensures that all participants received identical, homogeneous coal samples and any variations in the measured results were unlikely to be due to sample variability. The

Table 1
Inter-laboratory comparison II participants

Participants	Affiliation
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B.M. Krooss, A. Busch, Y. Gensterblum	Institute of Geology and Geochemistry of Petroleum and Coal, RWTH Aachen University, Germany
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