Influence of coke particle size on pore structural determination by optical microscopy

Mhlwazi S. Nyathi [a,*], Maria Mastalerz [a], Richard Kruse [b]

[a] Indiana Geological Survey, Indiana University, 611 North Walnut Grove, Bloomington, IN 47405-2208, USA
[b] ArcelorMittal USA, 3001 East Columbus Drive, East Chicago, IN 46312-2939, USA

ABSTRACT

Coke produced from a commercial oven was ground into various particle size ranges as a method to investigate the effect of coke particle size on pore structural parameters. The prepared samples were studied using reflected-light optical microscopy coupled with computerized and manual image analyses. Helium density and surface area measurements were performed on the prepared samples. Results showed that the use of smaller coke particle sizes for sample preparation leads to a loss of larger pores, thus, leading to an inaccurate description of the coke pore structure. This was shown by a decrease in volume porosity and average pore area; and an increase in average cell-wall thickness, coke density, and surface area that accompanied a decrease in the coke particle size. Moreover, a decrease in the ratio between pore area and cell-wall thickness, and a decrease in the ratio between pore long and short axial lengths (Feret's ratio) indicated a loss of large and elongated pores as the coke particle size decreased. However, a comparison of coke particle size ranges of 2.8–4.7 mm and 15–16 mm indicated that a particle size in a range of 2.8–4.7 mm can be employed without compromising pore structural characteristics, yet still enabling representative sampling.

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1. Introduction

Metallurgical coke remains one of the most important materials fed into blast furnaces. A principal measure of coke quality is mechanical strength, because coke strength determines its extent of degradation in the blast furnace and consequently influences coke's ability to allow optimum permeability for the flow of gaseous and molten products (Diez et al., 2002). Mechanical strength, typically measured by some type of tumble drum index at room temperature, is known as cold-strength. The coke strength after reaction (CSR) is measured by reacting coke with CO_2 at 1100 °C for 2 h (Ida et al., 1971). Surface area available for solution loss, as determined by coke porosity and micro fissuring, is one of the properties that influence coke reactivity (Chiu, 1982; Goscinski et al., 1985). Surface area is a function of coal blend, particularly its dilatation behavior, and carbonization conditions such as oven bulk density and quenching practices (Goscinski et al., 1985). As volume porosity and pore size increase, CSR decreases indicating an increase in coke reactivity towards CO_2 (Graham and Wilkinson, 1978; Grant et al., 1991). Because the coke is a relatively high porosity material, its porous structure is a source of flaws and greatly influences its strength properties (Goscinski et al., 1985; Patrick, 1983; Patrick and Walker, 1989). The largest pores of the coke pore structure can be orders of magnitude larger than other coke structural features, thus, large pores have a major impact on coke reactivity. The awareness of pore structure, especially larger pores, therefore, becomes crucial to fully characterize and understand coke mechanical strength. The use of optical microscopy accomplishes the purpose of qualitative and quantitative analyses of coke porous structure, because it covers the pore size ranges of interest, especially larger pores. Although a quantitative optical microscopy description of coke porous structure can be performed using manual methods, the use of computerized image analysis allows a sufficient number of measurements to be taken, thus ensuring representative data collection and eliminating subjective measurements. Automatic lineal analysis has helped to establish a well-defined relationship between coke structural pore volume and reactivity (Schapiro and Gray, 1963).

Given the wide range of pore sizes in a coke structure, cell-wall sizes vary greatly, from thin to thick interpore spacing values. This means that some cell-walls are more susceptible to breakage during sample preparation or in use. Although there are several quantitative studies on coke porous structure using optical microscope coupled with computerized image processing (Andriopoulos et al., 2003; Goscinski et al., 1985; Patrick, 1983), and optical microscopy remains a widely used technique in coke studies, data elucidating the effect of coke particle size upon sample preparation on the quantitative aspect of pore structural analysis is lacking. It is common in coke studies to use ≤3.0 mm particle size (Pusz et al., 2009; Pusz et al., 2010; Sharma et al., 2005) or a coke lump when preparing blocks for optical microscope analysis (Andriopoulos et al., 2003; Barriocanal et al., 1994; Lundgren, 2009; Patrick, 1991; Patrick and Walker, 1989; Sato, 1998). However, there does not seem to be a generally accepted guideline for selecting a particle size for

* Corresponding author. Tel.: +1 812 856 0617.
  E-mail address: nyathis@indiana.edu (M.S. Nyathi).

routine sample analysis. In the interest of preserving pores and other structural features during sample preparation, the use of a lump is an attractive alternative; however, it limits the possibility of having a representative sample. This paper aims to show an empirical relationship between coke particle size and quantitative measurements of pore structural parameters; namely, pore volume fraction, pore area distribution, average pore area, and average cell-wall thickness.

2. Material and methods

Coke produced from a commercial oven was ground into four different particle size ranges, 0.75–0.85, 1.0–1.18, 2.8–4.75, and 15–16 mm (Fig. 1A–D). Crushing was done manually using a hammer. All the particle size ranges were obtained from one coke sample to assure homogeneity of the original material. The samples were then Lucite-mounted in pellets using a Leco PR-15 sample press and polished on a Leco-AP-60 grinder/polisher. Samples were analyzed using a Zeiss microscope equipped with a Leica application image analysis suite. The images were generated using a reflected, white-light microscope fitted with an X–Y stage. The images were taken using a 40× oil immersion objective. For each polished block, 100 images were taken systematically by traversing the sample and taking images at 1-mm intervals. The collected images were then analyzed.

The analyses were carried out using automated and manual image analyzing programs. For computerized image analysis, an ImageJ program was used, whereas manual image analysis was performed using a Leica image analyzer. ImageJ allocates each feature in an image with<!——> a brightness value, expressed as a fraction of the range between white (maximum output) and black (zero output). A threshold is then adjusted in order to make sure a correct black and white representation of features in the image is obtained (Fig. 2A–B). Threshold
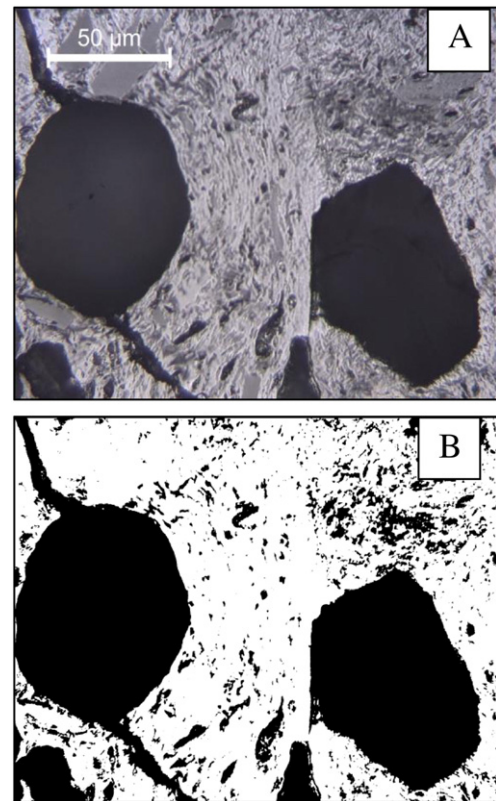

Fig. 2. An example of optical micrograph (A) before and (B) after conversion into a black and white representation using the ImageJ program.
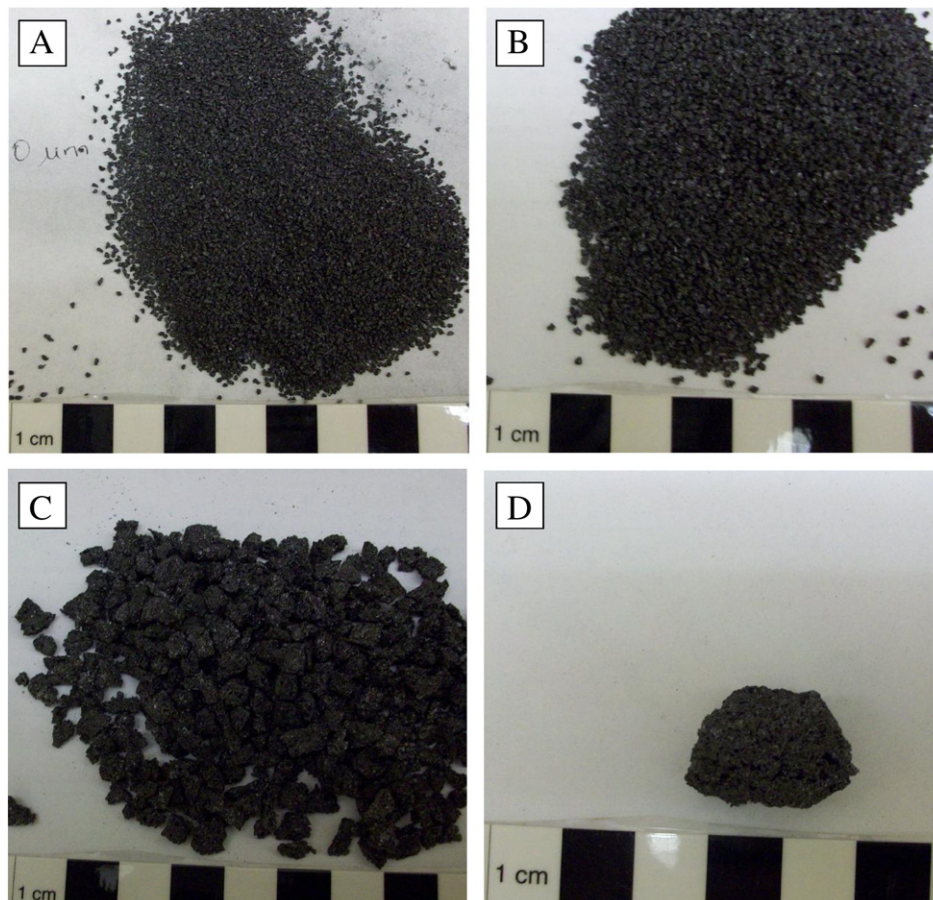

Fig. 1. Images of coke ground into particle size ranges of (A) 0.75–0.85 mm (B) 1.0–1.18 mm (C) 2.8–4.7 mm and (D) 15–16 mm.