Crossdisciplinary Applications of Microscopy Techniques, e.g. Physic-Life Science Interfaces

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Textural variations in experimental metallurgical coke: insights from optical microscopy and wavelet-based image analysis

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Carbonization of coals during coking process leads to the formation of various microscopic textures, which amongst other parameters, govern chemical and physical properties of the resulting coke. The formation of microscopic textures is a function of parental coal mix and coking conditions. The textures of coke are usually characterized on the basis of their optical behaviour in polarized light. There are three dominating textures, which have different orientation degree and, consequently, reactivity in CO/CO_2 atmosphere [1]. These textures are: highly oriented banded, mosaic and isotropic [1,2]. The shortage of a high quality coking coals, as well as environmental and economic considerations, have lead to the attempts of utilization of various additives to a coal mix. Among them - plastic [3]. This study is devoted to the characterization of major textures of experimental cokes, prepared with waste polyethylene (PE).

The samples of experimental metallurgical coke were prepared in a laboratory-scale ovens [4] using one type of coal, obtained from Ruukki Metals, Finland. The coal was mixed with varying amounts of PE, $(C_2H_4)n$. In total, six samples of coke were made with 2.5, 5, 7.5, 10 and 12.5 % of PE, and one from 100% coal (without PE). The PE plastic and the coal were comminuted to a < 5 mm fraction and then sieved. The coking process (20 - 1200^{o}C) was performed in a Nabertherm type HT08/18 chamber furnace during 16 hours and the quenching process (in nitrogen) has lasted 6 hours. The samples of coke were then sliced to 4-5 mm pieces, molded into a resin and polished (Figure 1, A) according to a procedure reported by Kokkonen & Gornostayev [5].

Optical investigations of the samples were done with an Olympus BX51 microscope equipped with a digital camera. Special software [2] was used to perform wavelet-based image analysis. According to Makkonen et al. [6], any given sample of coke can be representatively characterized with wavelet-based image analysis by studying 7-11 areas (points). In every point, 4 images under different positions (20°, -20°, 0° and 90°) of polarizing lenses should be taken. The program then divides (Figure 1, B) the images (2048x1536 pixels) into 12 sub-areas (512x512 pixels) and calculates each sub-area separately. As a result, it gives the amount (%) of isotropic, mosaic and banded textures, as well as pores and a map of textures (Figure 2) for each sub-area. Inert portions [1,6] can not be recognized by the program [6], and they should be excluded from the observations. It was also verified by Makkonen [*pers. comm*.] that the calculations presented in a research report on image analysis of industrial coke samples [6], are in agreement with traditional point counting on the same set of samples. The later method is common for characterization of coke textures [1,7,8]. In this study, each sample was photographed and subsequently analyzed in 10 areas (Figure 1, A), which gave 120 sub-areas for the image analysis, and 120 datasets per sample for subsequent statistical calculations.

The investigations of experimental samples of metallurgical coke prepared with different amounts of PE plastic have shown that the addition of PE leads to the increase of pore size, and their shape is more often has rounded outlines (Figure 2, A,C). The wavelet-based image analysis (Figure 2, B,D) has revealed general increase of isotropic texture in the PE coke, compared to PE-less coke. Also, there is a strong negative correlation (approx. -0.9) between isotropic and mosaic textures in the samples, when the increase of isotropic texture is accompanied by the decrease of mosaic texture. The latter observation can generally suggest that the isotropic texture, at least partly, can be formed for the expense of mosaic, when PE plastic is added to a coal mix. The reason for this phenomenon can be related to the activities of one or several gas phases (ethylene, propylene, isobutylene, 1-hexene and heptane) formed during thermal decomposition of PE [9]. The other related research [Heino, *pers. comm.*] has also found a correlation between porosity and cold strength of a coke with the amount of PE.

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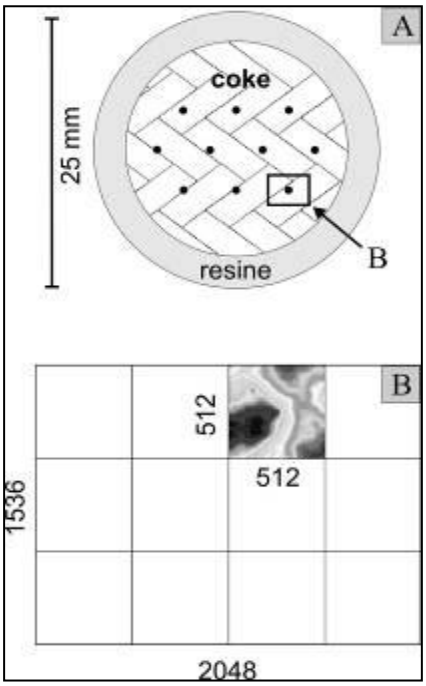

Figure 1. Polished section, locations of the points (A) and map of sub-areas for image analysis (B).

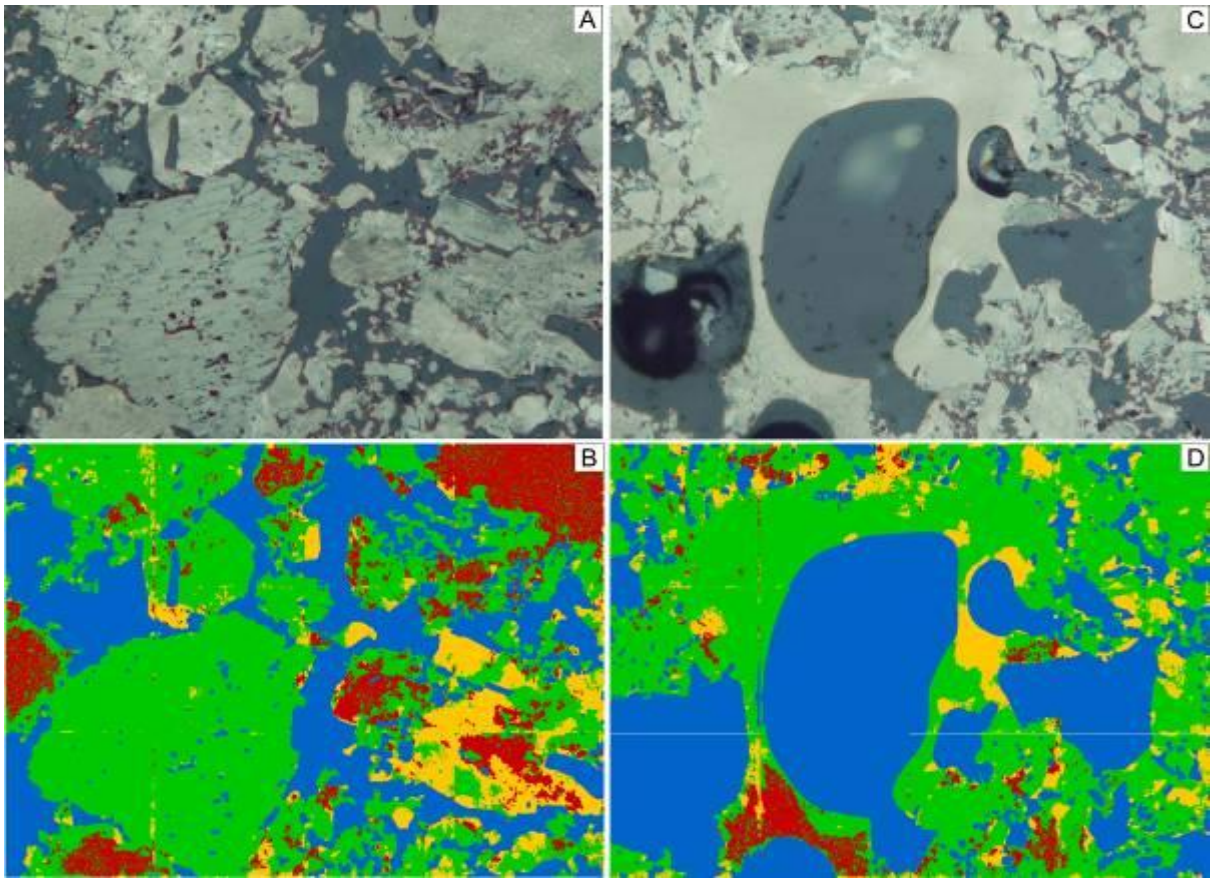

Figure 2. Wavelet-based image analysis of experimental metallurgical coke. A,B - 100% RI coal, C,D – 87.5% RI coal, 12.5% PE. Magnification - x20. Textures: isotropic – green, mosaic – red, banded – yellow, pores - blue.