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APPLICATION OF AUTOMATED PETROGRAPHY TO CHARACTERIZE COKE QUALITY DISTRIBUTION IN SLOT-OVEN & NON-RECOVERY OVEN COKE[1]

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Abstract

An automated method of measuring the bireflectance of metallurgical coke has been used to determine the quality across two different types of coke beds. Maximum reflectance (Rmax), and Bireflectance (maximum - minimum reflectance), (BIR), are the two measurements required to uniquely characterize and classify the varieties of fused and unfused micro-textures, and vapour-deposited coke. A Fingerprint of coke is shown by the frequency distribution of all maximum reflectance values in a coke (>100 million values per Fingerprint), the mean of which correlates with CSR. Thus, coke quality, determined from Fingerprints and related to CSR, can be measured on 20 mm-sized pieces of coke. This paper describes the spatial variation of these measurements, made on sequential, adjacent samples cut from a slab from the wall to tar-seam in slot-oven coke, and along layering in non-recovery oven coke.

Key words: Automated petrography; Coke quality.

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1 INTRODUCTION

Traditional manual coke-texture analyses suffer from low numbers of point-counts, usually only 1000 per sample, which introduces significant uncertainty to the measured quantities. Automating the measurement process provides a method for overcoming these shortcomings. Eilertsen et al.,[1] and Crelling et al.,[2] describe a method of acquiring calibrated images of carbon materials, in which a polarizer in the incident light path was advance 18 degrees between the capture of ten sequential images. For each pixel in these images, the maximum and minimum reflectance values can be established, and by difference, the bireflectance (Rmax – Rmin), calculated.
Quantitative monochrome or false-coloured reflectance maps displaying the maximum reflectance value, the minimum reflectance value, and the bireflectance of all pixels in a field of view can be constructed (Figure 1). Although these maps superficially resemble photomicrographs, they are computer-generated maps based on grey scale, or false-colour interpolation, and represent scenes that cannot be observed directly with a microscope, nor photographed.

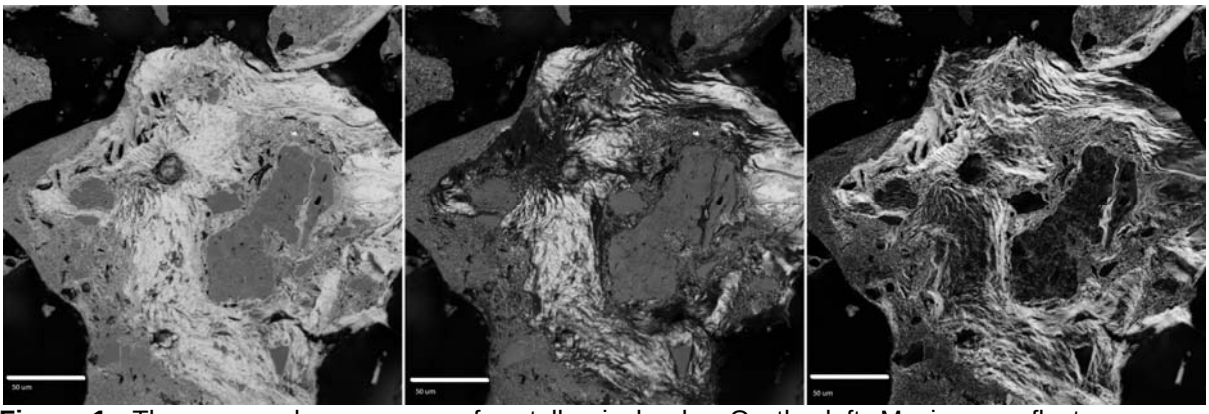

Figure 1. Three monochrome maps of metallurgical coke. On the left: Maximum reflectance map, Romax; in the center: Minimum reflectance map, Romin; and on the right: Bireflectance Map, BIR; (white scale bar: 50 µm).

2 METHODS & MATERIALS

The image-capture platform used in this study comprised a Zeiss reflected light microscope with a rotating polarizer in the incident light path, together with a 2 x 2 mega-pixel CCD camera as detector of monochromatic green (546um) reflected light, and a Marzhauser scanning stage for sample movement. Instrument calibration was performed using a Klein & Becker Strontium Titanite reflectance standard, with R = 5.41%. For each coke sample, 200 one-megapixel binned images were captured at a specific polarizer orientation to produce a file of 2000 images. Each coke sample is therefore characterized by 2 billion reflectance values ($2x10^9$), at >12 bit resolution.

2.1 Coke Samples

Two 210mm coke fingers, of different coal rank, prepared in a pilot scale slot test oven were slabbed parallel to the wall, and seven 70 x 30mm rectangular sections of coke were mounted in polyester resin for petrographic analysis. The slabbed samples, are labelled 1, (closest to wall), through 7, (closest to Tar Seam), and when

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imaged more than once, the suffix L (left), M (middle) or R (right) is used to identify the area.
Non-recovery oven coke samples were collected from an oven located at the middle of Battery F at SunCoke's Vansant facility (Segers).[3] A 50 cm long finger from the top of the coke bed was cut into14 layers parallel to the top bed surface, from which numerous 20 mm CSR-sized coke pieces, were embedded in circular polyester resin mounts, for petrographic analysis. The samples are labelled as follows; T (for Top); A, (closest to coke bed surface & heat source), to N, (Tar Seam sample); 1 thru 5 for piece number. Example TK5, Top Finger, layer K, 5 fifth piece.

2.2 Carbon Type Identification Analysis

Seven carbon types are identified in crossplots of Coke Bireflectance with Coke Romax Reflectance (Figure 3), and although these are discrete populations of carbons, there is mixing at the boundaries of the groups with the adjacent groups. Algorithms were derived to discriminate these populations, and a computer now accomplishes the separation of these optically distinct components among single-cokes and coke blends automatically. The application of machine vision in the collection of data, coupled with the automated interpretation of these data, dramatically improves the accuracy of quantification of the carbon forms present, while reducing the subjectivity imposed by the human aspect of point-counting.

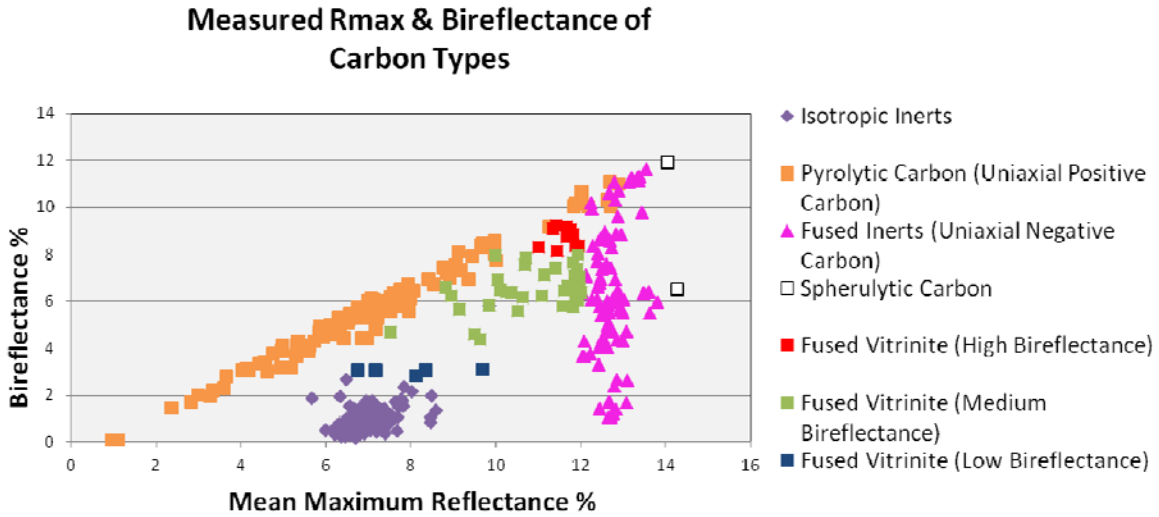

Figure 2. Seven mutually distinct carbon types can be recognized and defined on the basis of coke bireflectance and romax reflectance.

Conventional coke petrography involves point-counting the volume percent of textural components in a coke sample by applying a microtextural classification (ASTM 5061, for example). Several of these qualitative coke classifications have been proposed, and there are local variants, but each distinguishes the following carbon forms; Incipient, Circular, Lenticular and Ribbon forms of coke; Isotropic inerts, and vapour-deposited Pyrolytic and Spherulitic carbon. The approach described here is wholly quantitative, and is based on measurements of coke reflectance, but an approximate correlation with the qualitative classifications is possible, as follows; Low Bireflectance Fused Vitrinite with Circular forms; Medium Bireflectance Fused Vitrinite with Lenticular forms; High Bireflectance Fused Vitrinite with Ribbon forms.

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Interpretation of the Bireflectance maps is by automated image analysis. Algorithms have been developed to isolate and mask each carbon type, based on pixel values for Romax, Bireflectance, and Romin. In effect, each pixel is colour-coded depending on the response to masks that define the carbon types. The two maps below display the application of two techniques (Figure 3). On the left is a coloured version of the monochrome Bireflectance map shown above in Figure 1, now displaying seven colour-densities from violet through light and dark blues, green, yellow, orange, and red, corresponding with the monochrome grey scale change from black to white. This is bireflectance thresholding, but as a technique it does not separate carbon forms of different maximum reflectance. In general, low bireflectance measurements relate to inert or low-fusing material; whereas high bireflectance relates to the highly-fusible material.

The image on the right has been interpreted using different algorithms that better discriminate between carbon forms with markedly different optical characteristics. For example, the areas of Fused Inerts (Uniaxial Negative carbon) shown pink, have high maximum reflectance (see left image, Figure 1), and are differentiated from the unfused Isotropic Inerts, shown in violet, which also share a low bireflectance. Each image of a sample is analysed using these techniques, and the volume percent of the carbon forms is compiled from hundreds of millions of pixels, accurately, in a repeatable, quantitative way.

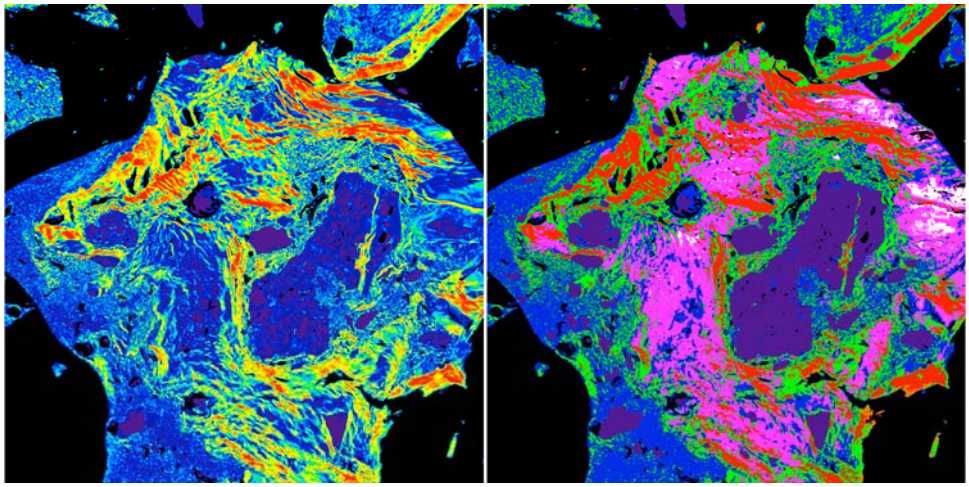

Figure 3. Bireflectance Maps showing interpretation by Bireflectance thresholding, on the left, and a more complex algorithm on the right. On the right, pink – Fused Inerts (Uniaxial Negative); violet - Isotropic Inerts; dark blue/green/red – Fused Vitrinite with low-, medium-, & high-bireflectance.

The proportions of the seven carbon types are computed and displayed within the histogram of Romax Reflectance (Figure 4). These diagrams are distribution plots of carbon types within the romax reflectance profile, and are Fingerprints of coke. The distribution and layering of the carbon types is distinctive. Isotropic inerts, and incipient carbon (unfused low-rank, higher volatile “soft coking coal”), are always to the left; fused inertinite, spherulitic carbon (from vapour-phase deposition), and high bireflectance fused vitrinite, are always to the right.

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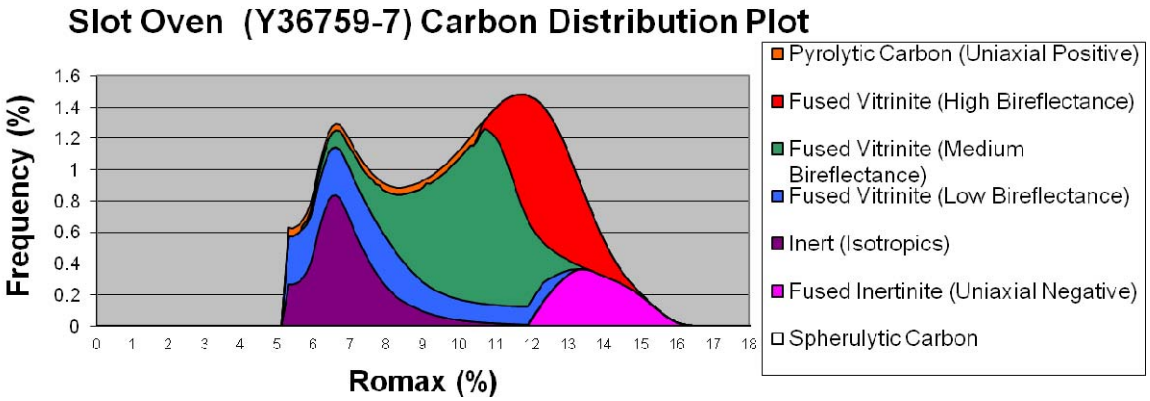

Figure 4. Carbon type distribution plot within a Romax Fingerprint profile.

2.3 Coke Fingerprints

Superficially similar to coal Fingerprints, Coke Fingerprints are frequency distributions of Romax reflectance of coke. They are unique and specific, and can be used to demonstrate product consistency (Figure 5), or highlight compositional variations. Fingerprints of cokes derived from high-volatile rank coals are characterized by a large singular peak between 6% & 10% Romax (Figure 5). Cokes produced from low volatile rank have a small peak between 6% and 8%, caused by unfused Inertinites from the parent coal, while the fused vitrinites generally occur between 9% & 12% (Figure 5). The location of fused vitrinites in cokes made from medium volatile coals, are intermediate between these two higher and lower types.

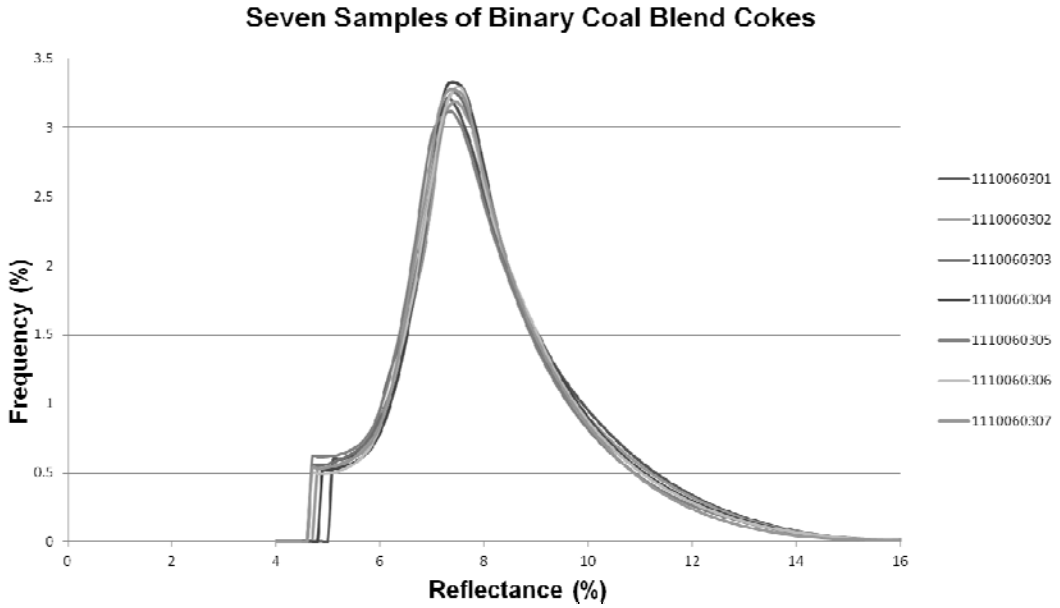

Figure 5. An example of product consistency illustrated by Coke Fingerprints of seven crushed samples of a binary coke blend.

3 RESULTS

3.1 Coke Fingerprints

Coke Fingerprints for the three different sets of coke samples are shown in Figures 6, 7 and 8.

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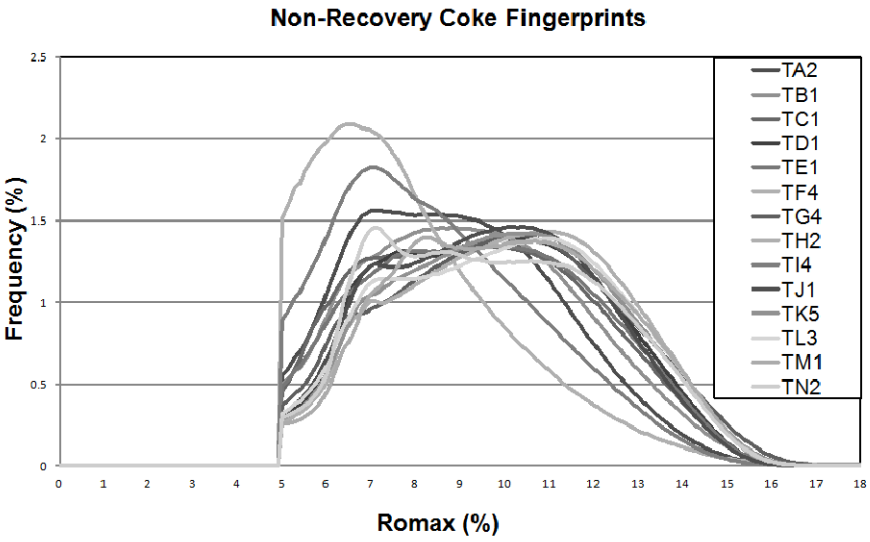

Figure 6. Coke Fingerprints from Non-Recovery Cokes.

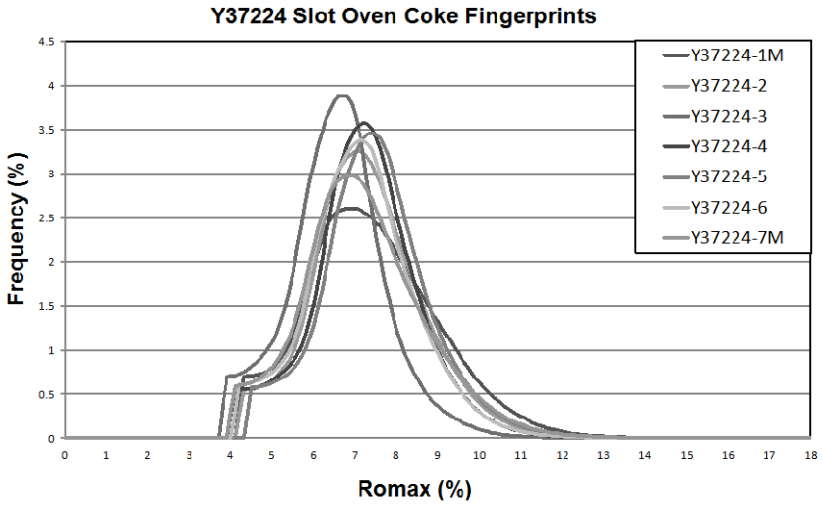

Figure 7. Coke Fingerprints for Y37224 Slot-Oven Cokes.

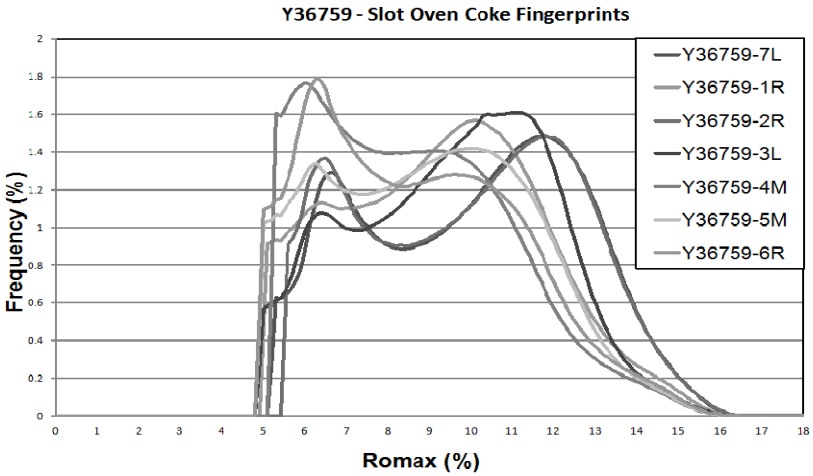

Figure 8. Coke Fingerprints for Y36759 Slot-Oven Cokes.

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Figure 6 presents Fingerprints of a single top-to-centre piece of non-recovery coke that was sectioned into 14 layers to make CSR-sized pieces. The Romax frequency distribution indicates a blend of coals

3.2 Romax Variation

The mean Romax Reflectances for the three different sets of coke samples are shown in Figures 9, 10 and 11.

3.3 Spatial Variation in Carbon Forms Component

The variation in the Isotropic Inerts component and other carbon forms of the three different sets of coke samples are shown in Figures 12, 13 and 14.

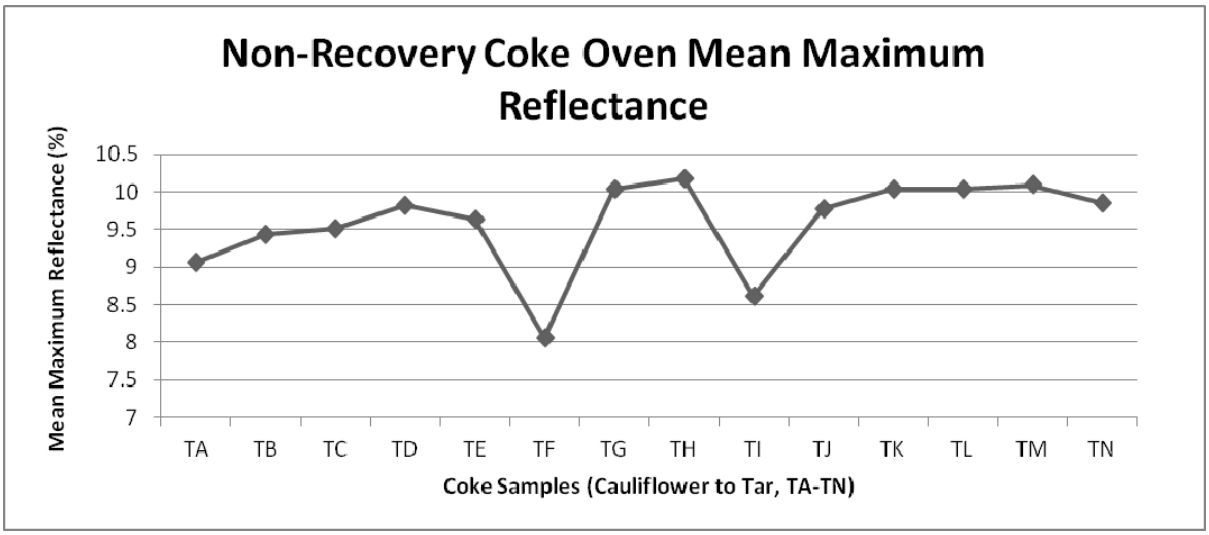

Figure 9. Mean Reflectance of Non-Recovery Cokes.

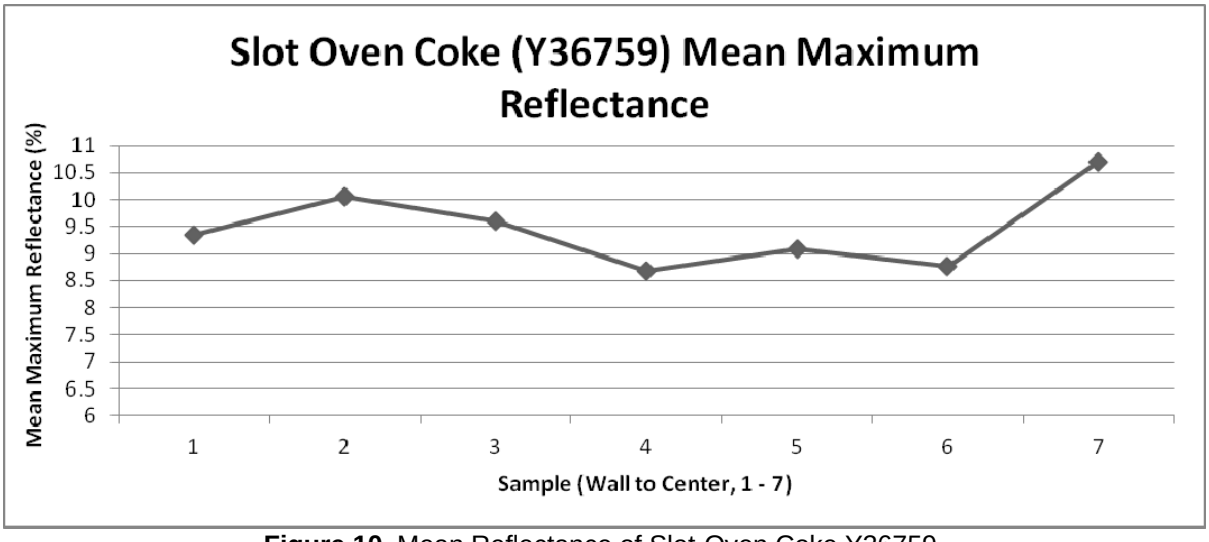

Figure 10. Mean Reflectance of Slot-Oven Coke Y36759.

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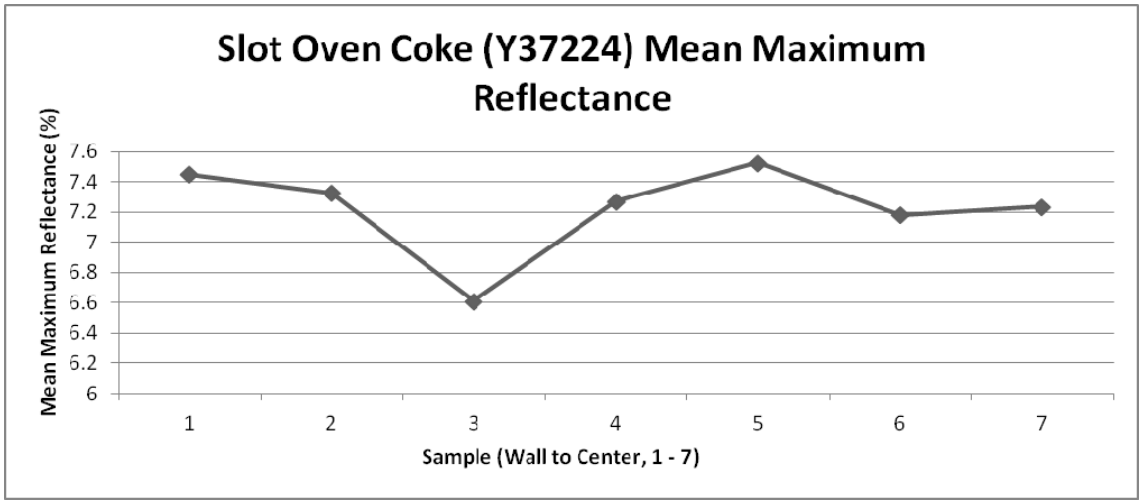

Figure 11. Mean Reflectance of Slot-Oven Coke Y37224.

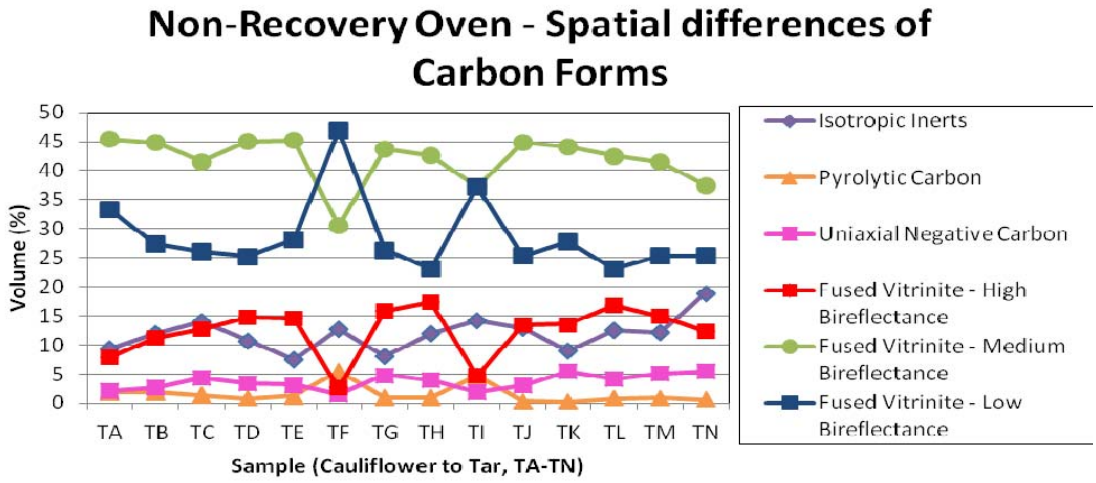

Figure 12. Variation of Isotropic Inert Component of Non-Recovery Cokes.

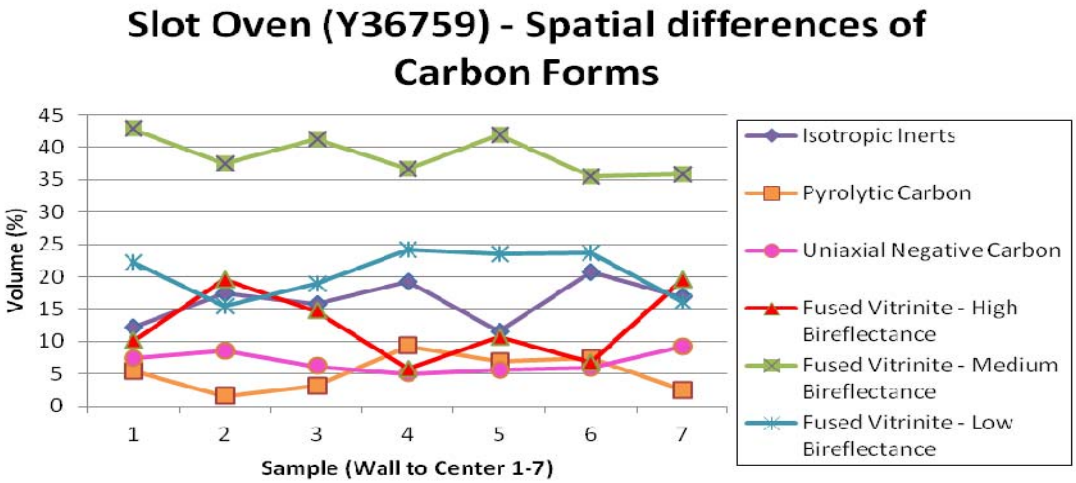

Figure 13. Variation of Isotropic Inert Component of Slot-Oven Coke Y36759.

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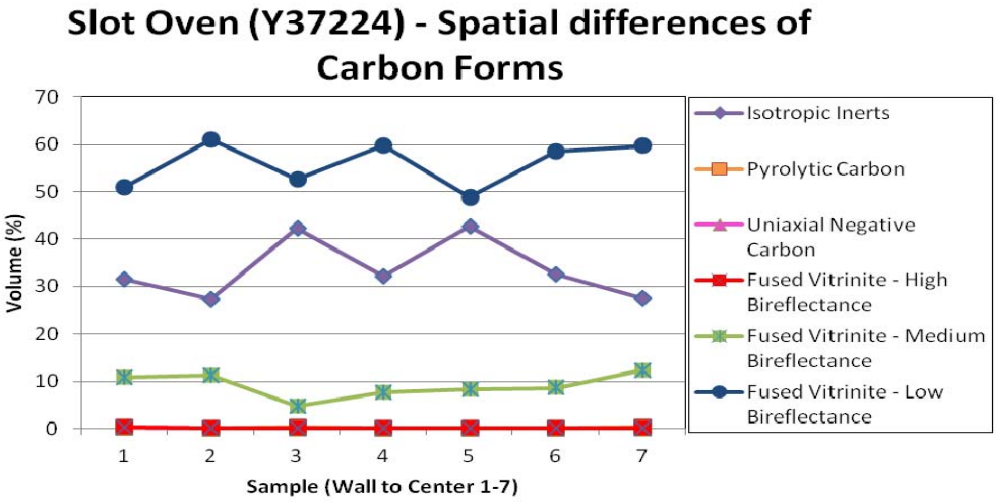

Figure 14. Variation of Isotropic Inert Component of Slot-Oven Coke Y37224.

4 DISCUSSION

An unexpected result of the Coke Fingerprinting analysis is the amount of observed variation and inconsistency of the Reflectance Profiles when compared to the Fingerprints of the seven crushed cokes of Figure 5, but this could be an inherent difference between two markedly different types of sample preparation; coke blocks versus crushed particulate pellets. The smallest amount of variation is seen in the Slot-Oven cokes from High Vol coal (Y37224) Figure 7. We had not expected to see concentration variations, which are new to our experience.

Each set of Coke Fingerprints shows this unexpected feature – variation in the concentration of Isotropic Inerts, and other carbon forms. The range of Isotropic Inerts contents, for example, is greatest in the higher rank Slot-Oven coke (Y36759), at 16%, followed by the Non-Recovery coke with 12%, and the High Vol Slot-Oven coke (Y37224) at 10%. The mean Isotropic Inert content is highest for the low rank Slot-Oven coke (Y37224) at 29.6% (SD=3.9%), because it includes incipient, non-fused, low rank Vitrinite. This is followed by high rank Slot-Oven coke (Y36759) with 10.5% (SD=0.9), and the Non-Recovery coke at 13.9% (SD=6.42).

The variation in the content of Isotropic Inerts impacts the mean reflectance and bireflectance of the cokes; higher levels of Isotropic Inerts reduce the mean reflectances, which are predictors of the CSR parameter. The mean reflectance, and standard deviation, of the three different cokes are, in ascending order; low-rank Slot-Oven coke (Y37224) at 7.06% (SD=0.07); Non-Recovery coke with 8.97% (SD=0.14; high rank Slot-Oven coke (Y36759) with 10.00% (SD=0.66).

4.1 Coke Strength After Reaction

Previous work by Gill et al. [4] has shown a strong relationship between coke mosaic size index (CMSI, a measure of the average size of the textural components in the coke), ash chemistry and CSR. CMSI is based on different mosaic classification system by:

$$CMSI=(a+2b+3c+4d+5e)/(a+b+c+d+e)$$

In this equation a, b, c, d and e represent percentage of very fine, fine, medium, coarse and elongate domains respectively, as determined by manual point counting

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techniques. However, later work by Brown et al.,[4] showed the CMSI is related to the bireflectance of the sample. In the current study ash chemistry was not available for all the samples and so an absolute value for CSR cannot be estimated. However, variation in CSR is implied by the reflectance variation, and if we assume for a given coke the ash chemistry is consistent throughout the sample an estimate of the variation in CSR can be made from the variation in reflectance / bireflectance. The relation derived in Gill et al.,[5] is

$$CSR=14.09CMSI – 53.16F + 22.22 \pm 5.8$$

Where, F is an ash basicity factor.
The spatial variations in CSR can be estimated to be as large as 8 or more points for both the NRCO and the higher rank slot oven coke; and 6 for the lower rank slot oven coke. This is comparable to the 10-20 points in CSR variation reported in commercial ovens, for example, Khan, Gransden and Price,[6] and Amamoto[7].
Taken together, these data demonstrate a variation along the length of the coke fingers, in a direction perpendicular to the oven wall, in the coke quality parameters, mean Rmax, BIR, mean Isotropic Inerts content %, and percentages of other carbon forms, and predicted CSR. Unfortunately, the sample series in each case was discontinuous, that is, there were spaces between adjacent sample sites. Another, already-planned study, will use continuous samples selected perpendicular to the wall, and attempt to identify a cause, and perhaps a fabric in the coke that is responsible for this observed variation.

5 CONCLUSIONS

This study of the coke petrography of three different coke fingers has demonstrated the following:

- The new technique is a major advance in coke petrography dramatically reducing the error and providing a quantitative evaluation tool for future work.
- The technique is both accurate and rapid, and the reflectance measurements relate directly to coke quality.
- The method uniquely characterizes, and ranks, cokes.
- The application can provide new and unique insight into coke quality. For example, as evidenced by this study, it has quantified the extent of variation in quality distribution wall-to-centre coke samples, of different origin.

Acknowledgements

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