Minutes of Commission III

Industrial Applications of Coal Petrology

65th ICCP Meeting, Sosnowiec, Poland

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Tuesday - 26th August

16:45-16:55 - **Opening Address**

The opening address of ICCP Commission III, Industrial Applications of Coal Petrology started with welcome and presentation of a new schedule of work for Commission III for Tuesday and Wednesday. Isabel Suárez-Ruiz presented the activities of active and inactive working groups in last year and also in the past years. First she discussed the activities of the four active working groups (Identification and petrographic classification of components in Fly ashes WG, Self-heating in coal and coal waste dumps WG, Coke Petrography WG, Microscopy of Carbon Materials WG, and Coal Blends Accreditation Program - CBAP) and than the activity of inactive working groups (Improved Image Analysis Working Group and Characterization of Gasification Products Working Group) in the last years.

The Commission III meeting started at 16:45 Tuesday August 26th and was attended by 24 participants.

16.55-17.30 - **Fly Ash Working Group** *(convenors: Isabel Suárez-Ruiz and Bruno Valentim)*

The results of this WG were presented by Isabel Suárez-Ruiz. She presented the objectives of the WG that was preparation of the Atlas of fly ash components based on images from 2007, 2009 and 2011 exercises with a level of agreement of 80-100, and distribute the Atlas among the WG participants for improvement. She presented the outline of the Atlas, the front cover, contents, contributors, introduction, provenance of fly ashes, samples, preparation and photomicrographs, selection of fly ash photomicrographs, petrographic classification of fly ashes (established in 2012), description of the optical properties of fly ash components, examples, organization of the photomicrographs and gave examples of the forms deriving from combustion of various things (coal, coal blends, pet coke, biomass, co-combustion of e.g. coal and biomass, coal blends and pet coke) in various types of boilers (pf, fluidized boilers); these forms were organic and inorganic. At the end of the Atlas will be given bibliographic references. Then she asked about comments and suggestions. Nikki Wagner asked about the title of the Atlas and Isabel Suarez-Ruiz stated that she is not happy with the title and it will be changed. Bryan Cardott asked about the rank of

coals from which the fly ashes originated. Isabel Suarez-Ruiz has that information and it will also be included in the description of the pictures of fly ashes. By then she only concentrated on the origin of fly ashes but the rank will also be included. Krystyna Kruszewska recommended putting the information about fly ashes into table and Isabel Suárez-Ruiz replied that it will be done. Then Isabel Suárez-Ruiz thanked for the comments and to all the members of the WG.

17:30- 18.10 **Coke Reflectance Measurements** *(speaker: Richard Pearson)*

Richard gave talk on the application of the new methodology of coke reflectance measurements. He presented the bireflectance mapping and reflectance density maps. He showed carbon form maps, carbon forms from bireflectance based on brightness and flashiness, coke fingerprints and reflectance, differentiation of commercial and test oven cokes with lab cokes, CSR vs. Ro_{max} reflectance regression and also pictures of fusinite, fusinite with vitrinite. He discusses coke porosity, bireflectance of deposited carbon and showed pictures of deposited carbon.

At the end of his talk he discussed sample preparation. During discussion first Georgeta Predeanu asked if after revising the standards, the standards can be included in the methodology and Richard said yes. Cornelia Panaitescu stated that the modern application method for anisotropy of coke and composition of coke must be continued because it is very important. She recommended working on cokes from blast furnaces that will give more opportunity to study the problem. Richard replied that they are trying to do it. Isabel Suárez-Ruiz asked about the practical importance of the method and Richard replied that it gives much cheaper version of CSR test and that doing reflectance measurements one can get CSR tests. Isabel also asked if he is going to license the product and Richard replied that rather not.

The meeting finished at 18.10 and Isabel closed the session.

Wednesday, August 28, 2013

The meeting started at 9.10 with 33 participants. Isabel Suárez-Ruiz presented the schedule for the Commission III for this day and announced that at the end of the meeting of this commission Nikki Wagner will present the activity of Gasification WG.

9.15 - 10.00 **Coal Blend Accreditation Program** *(convenor: Isabel Suarez-Ruiz)*

Isabel Suárez-Ruiz presented objectives of the CBAP. She presented the items that have to be determined, evaluation process, blend and the statistical parameters that are calculated in the program. She discussed the problems with the data base. Final report of CBAP from this year exercise will be delivered to participants early in September 2013. She presented evaluation of results in 2013 Exercise and presented the comparison of results from this year exercise and results from 2007, the year of the first CBAP exercise. At the end of her talk she presented conclusions from 2013 exercise. The next exercise will be proposed by the end of 2014 or early 2015. She also presented recommendations for future CBAP exercises. She made emphasis on the problems with CBAP data base. Then she asked for

single coals that can be used in the accreditation program. People having such coals should contact Isabel. The sending fees will be paid by the ICCP. She thanked to all participants of the CBAP exercise. Then she asked for comments/discussion. As there were no comments, she remarked that she is very happy with participants in CBAP as they are very professional and sent their results within deadline.

10.00 - 11.00 **Self-heating Working Group** *(convenors: Magdalena Misz-Kennan, Jolanta Kus, Deolinda Flores)*

First Magda Misz-Kennan presented the aims of the Self-heating Working Group and past activity in the period 2008-2012. Then she briefly discussed the problems with terminology and current problems connected with presentation of the forms that has to be recognized (square marking a field within particle or cross hair or arrow or another way). She presented the 2013 Round Robin Exercise that was based on 32 photomicrographs taken from coal wastes from Lower Silesian Coal Basin (Poland) in which organic matter was of 0.6-0.7% R_r. She presented the forms on which there was general agreement and the most troublesome forms. The problems with 2013 Round Robin Exercise was that in case of altered particles, not all participants marked all the three categories, i.e. appearance, structure and texture and the most troublesome forms were taken in polarized light with analyser. At the end of her presentation she concluded that (1) the results obtained this year were good and proofed that giving the vitrinite reflectance is necessary to differentiate between thermally altered and unaltered organic matter and (2) the difficulty was of a technical nature: how to mark the form. After Magda's presentation the discussion started how to present the form. Stavros Kalaitzidis asked what is important: a form or a whole particle? Isabel Suárez-Ruiz suggested that as for normal point counting analysis we should use cross-hair. Nikki Wagner suggested to use a square because we see what happens to the particle. Cornelia Panaitescu paid attention on the importance of temperature and that in coal wastes sometimes we might have coke and sometimes chars. Jola Kus replied that commonly we are unable to measure temperature we are usually absent in the please where self-heating and where altered forms occur. Deolinda Flores said that the heating temperatures were measured in Portugal directly and also indirectly using the method of newly formed minerals. That was part of the project that aim was to develop the way of measuring the temperatures but it is very difficult. Stavros Kalaitzidis suggested putting the R_r value of unaltered organic matter on every microphotograph. Georgeta Predeanu also suggested using square for recognizing the forms. At the end of the discussion on self-heating Jola Kus read the e-mail from Peter Crosdale about possible cooperation and it was strongly recommended.

The meeting finished at 11.00 and was followed by coffee break.

11.30 - 11.55 **Coke petrography working group** *(convenor: Lauren Johnson, presented by Magdalena Misz-Kennan)*

Magda Misz-Kennan presented the results of 2013 Round Robin Exercise on behalf of Lauren Johnson. Magda presented the aim of the Coke petrography working group that is to classify the carbon forms in images of coke given a reference document with some photographs, the outline of the exercise that was based on 20 images from four cokes, and the results. The success of this exercise was limited based on the image quality which was made worse by the fact that images were sent as a PDF to reduce the file size. Lauren suggested sending coke blocks to participants of taking microphotograps of what they consider the various constituents of the coke, with the intention that this would raise some spirited discussion. She also suggested working with Dave Pearson on reflectance of coke. After the presentation the discussion started. Cornelia Panaitescu suggested stronger collaboration with people that have more experience with coke petrography. She also remarked that atlas of coke was published in Romania. Dave Pearson said that they got polished blocks from Lauren and the surface of the blocks was poor and that that is why the pictures were very poor. She used much greater resolution than Dave was using. Walter Pickel suggested putting invitation to this Working Group in ICCP News Letter.

11.55-12.15 **Gasification WG** *(convenor: Nikki Wagner)*

First Nikki Wagner presented the activity of the Gasification Working Group. from 2007 when the WG was proposed. A number of participants wanted to join the WG because they wanted to learn and not because they were working on gasification. There was a problem with getting the samples. After the first Round Robin Exercise Nikki got response from four participants. It is necessary to compare the chars from gasification with chars from combustion and then to decide if there is a necessity for creating a new char classification. In 2012 it was suggested to close the WG or to get more samples. During the meeting in China it was suggested that the Gasification WG will continue but no more samples were obtained despite of some promises. Nikki also mentioned that the part of ICCP web page dealing with commission III has not been updated for a number of years. She asked again for samples and suggested closing the WG in 2014 if no additional samples be obtained. In discussion Magda Misz-Kennan remarked that as secretary of Commission III she sent request twice to all conveyors of active and past WG and she got few responses. Kaydy Pinetown suggested supplying samples from gasifiers what was also recommended by Isabel Suárez-Ruiz.

12.15-12.25 **Proposal of New WG** *Isabel Suárez-Ruiz*

Two years ago in the light of very good results from Application of reflectance in estimation of Structural Order WG it was proposed that Sławka Pusz, who conveyed the WG, should convey the Accreditation Program in that area. Unfortunately Sławka did not have time to conveyed the program. Now Sandra Rodriques agreed to be a convenor of the Structural Order Accreditation Program. Barbara Kwiecińska stated that Sandra will be an excellent convener. It was also suggested that Sławka should help Sandra if she be need. Isabel Suárez-Ruiz asked how many people were in this WG and Sławka replied seven. Isabel suggested

putting the information about the Structural Order Accreditation Program into the ICCP News Letter. David Pearson suggested that we do not need be accredited in bireflectance of materials. And he found organization of such accreditation program as inappropriate. Isabel stated that anisotropy is applied to many things and related e.g. to Raman Spectroscopy. David Pearson replied that there is no need for this accreditation program. Deolinda Flores suggested that it is necessary to know who is interested in this accreditation program and that it is necessary to send e-mail to all participants of Commission III and not to publish in news letter because people do not read news letters. Isabel suggested sending an e-mail about with information about the Structural Order Accreditation Program to all ICCP Members and also to include a small note in News Letter.

After lunch the works of Commission III continued.

13.30 - 14.30 **Carbon Materials Working Group** *(convenors: Georgeta Predeanu, Cornelia Panaitescu)*

The WG started after lunch at 13.30. Georgeta Predeanu presented the objective of the WG that is directed to the microscopically characterization of carbon materials derived from coal and petroleum, to consolidating and completing the existent methods developed for carbon materials structural and textural characterization.

The convener presented past activities of the Microscopy of Carbon Materials Working Group since its establishment in 2008 during the ICCP Meeting in Oviedo, giving a short review of the three performed exercises: identification of individual raw materials as such as are used in technological processes (2009-pitch coke, petroleum coke, anthracite, 2010-evolution of optical characteristics of two pitches during heating) and mixtures of these raw materials as they are incorporated into various finished products (2011-on different technological steps of steel electrodes, 2013-on different products anodes/cathodes). Comparative evaluation of the results in the 2009-2011 ICCP Carbon Materials WG exercises show high average levels of overall agreement over 80%. In 2013 in order to provide the participants with a suitable carbon materials used in aluminum industry, two sets of samples of anodes (the electrode) and embedding cathodes blocks used in the electrolysis vats from the industrial flow sheet of carbon products manufacturing Electrocarbon Slatina of Romania, were prepared. Following identification and characterization of the optical appearance of petroleum coke, calcinated anthracite and pitch coke both the optical type (isotropic/anisotropic), texture and size were carried out. The results show a high level of over 82% agreement of the analysts, compared to the conveners. The most easier identified textures (over 95% of 38%) were those of punctiform, mosaic and fine fiber; than follows the textures identified with a lower score (85-95% representing 15%) as being fiber medium and fine); between 75-85% (38%) were those of anisotropy, ribbon, domain, including the origin macerals of vitrinite and inertinite; and the lowest identification score (under 75% of only 8%) belonging to isotropic type. Georgeta presented that in 2013 is ending a series of 4 exercises addressed to the main industrial consumers of raw materials of carbon

source that can be investigated microscopically. She proposed that beyond 2013 the activities of the WG will highlight in some more exercises how microscopy can be a method of investigating raw materials of coal origin and waste lignocellulosic materials (biomass) in the stages undergoing pyrogenetic reactions involving the obtaining of char and then of the activated carbons, with interesting structures that can be widely analysed through microscopy. Georgeta and Cornelia made a call to everyone within the ICCP to come up with new ideas/proposals, according to their research activities to find new sources of petrographic research interests to carbon materials. They also propose to enlarge the conveners group with a new co-convener, to strengthen our working group, and who could provide samples. The conveners presented the intention to prepare a draft version of a manuscript discussing the results of the four exercises of the WG that will be sent to participants. The final version of the manuscript is to be completed by the end of December for submission in a peer-reviewed journal.

Isabel thanked Georgeta for the great job. She suggested providing her with samples of carbon forms (fly ash and chars from biomass combustion). Krystyna Kruszewska asked about the quality of the material and Georgeta replied that coke was made from coal and mixtures of coals and carbon material had very low porosity, low developed pore structure.

When the discussion on Carbon Material WG finished Isabel asked for ideas for new Working Groups. It was suggested that the web page of commission III should be ready by the end of this year.

14.30-14.45. **Commission III Concluding remarks** *(Isabel Suárez-Ruiz, Magdalena Misz-Kennan)*

The Meeting of Commission III took place on Tuesday August 27 and Wednesday August 28. First she presented the objectives of the Fly Ash WG, the outline of the Atlas of Fly Ash, the classification of fly ash components, organization of the photomicrographs in the Atlas and gave examples of the forms deriving from combustion in various type of boilers. The atlas will include particles from combustion of coal of various rank. Richard Pearson presented application of Coke Reflectance Measurements.

On Wednesday the Meeting started with report on Coal Blend Accreditation Programme given by Isabel Suárez-Ruiz who presented the report from this year accreditation exercise and compared the results with results obtained during the first exercise in 2007. She also presented the problems connected with evaluation of results and data base. In 2013 a new Round Robin Exercise was prepared in Self-Heating WG . The current problems are how to present the forms of organic matter to be recognized. In future cooperation with Peter Crosdale is planned.

Magda gave presentation of Coke Petrography WG on behalf of Lauren Johnson. It was recommended her to cooperate with people who have more experience and invite more people to this WG.

Nikki Wagner gave reasons of the problems in Gasification WG. The group will continue and Kaydy will provide her with samples from various gasifiers.

A new accreditation program was proposed on the Application of reflectance in Estimation of Structural Order. The convenor will be Sandra Rodrigues. Information about this program will be sent by e-mail to all ICCP members and published in ICCP News Letter.

The last presentation was by Geogeta Predeanu who gave report of the activity of Carbon Material WG. In future the activity will concentrate on other materials.