ICCP
COMMISSION III

Self-heating of Coals and Coal Wastes WG

2020 ROUND ROBIN REPORT

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1 Introduction

The Self-heating Working Group was established during the 60th ICCP Meeting in Oviedo (Spain) in 2008. The respective information about the established working group was published in the ICCP News Letter No. 45, 2008. The aims of the Self-heating Working Group are:

- to gather examples of various forms of self-heating-related transformations of organic matter in coal and coal wastes of various ranks
- to create a classification of these self-heating induced transformations in coal and coal wastes.

Since 2008, several Round Robin Exercises on coal wastes were organized (in 2009, 2010, 2012, 2013, 2014, 2015, and 2016) and an attempt was made to classify transformation forms of oxidatively and thermally affected organic matter observed in coal disposal dumps. During the 2009 ICCP Meeting in Gramado/Porto Alegre, Brazil, it was decided that a separate classification should be prepared for oxidatively and thermally affected organic matter in coal wastes and for oxidatively and thermally affected coaly matter in coals that were subjected to self-heating processes. The classification of transformed organic matter in coal wastes was modified in a number of Round Robin Exercises owing to the great variety of form of organic particles present in coal wastes and it is published (Misz-Kennan et al., 2020).

Since 2018, the objectives of the SH WG is focused on the oxidatively and thermally affected organic matter observed in coal seams.

2 Objective of the 2020 SHWG Round Robin Exercise

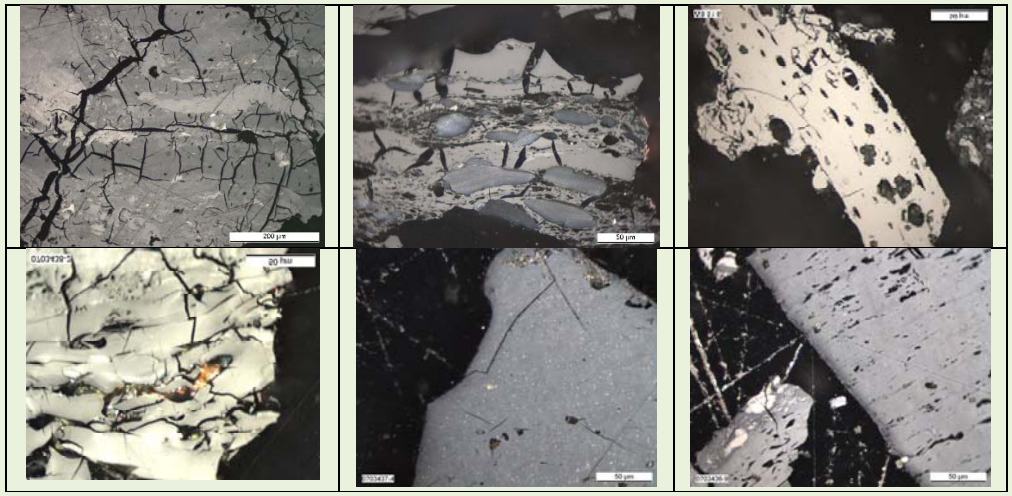
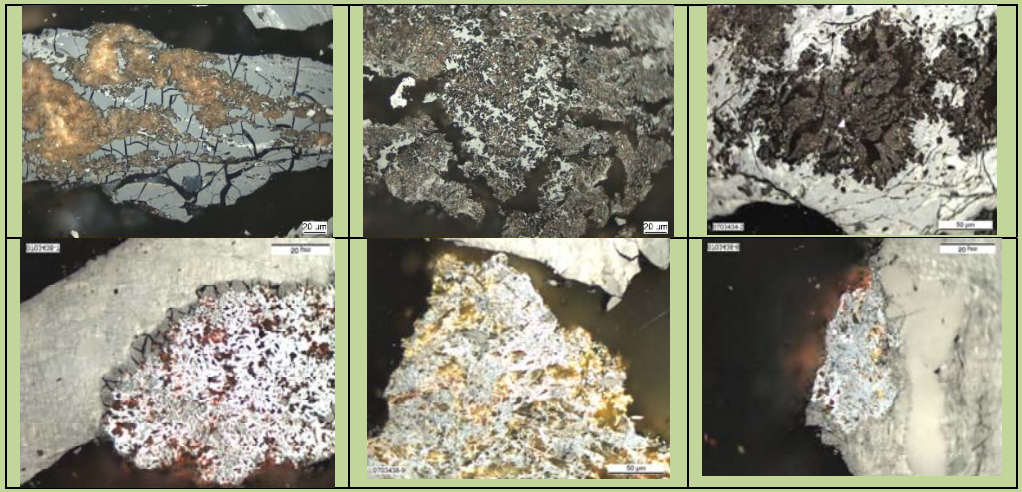
The objectives of the 2020 Round Robin Exercise are to:

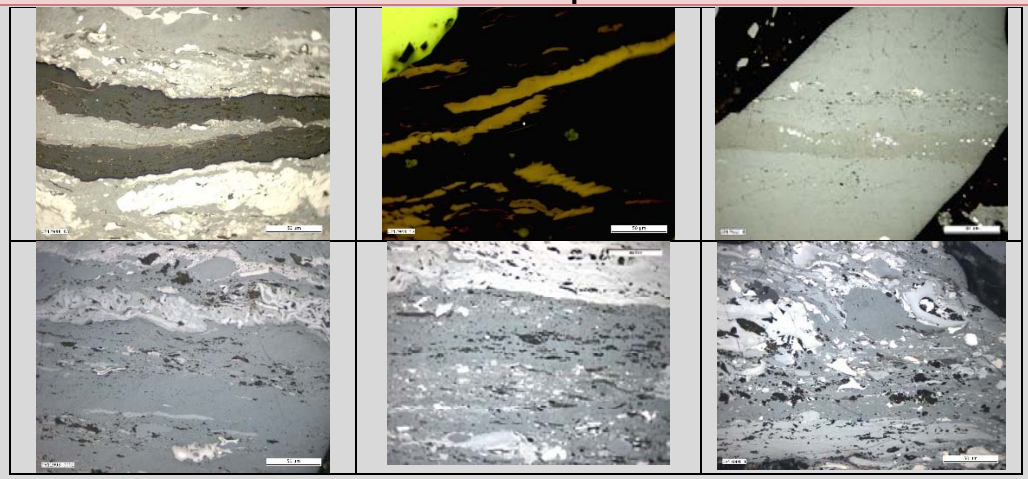
- to apply the established classification of oxidatively and thermally affected organic matter in self-heated coals.
- to test the applicability of the classification of oxidatively and thermally altered organic matter in self-heated coals.

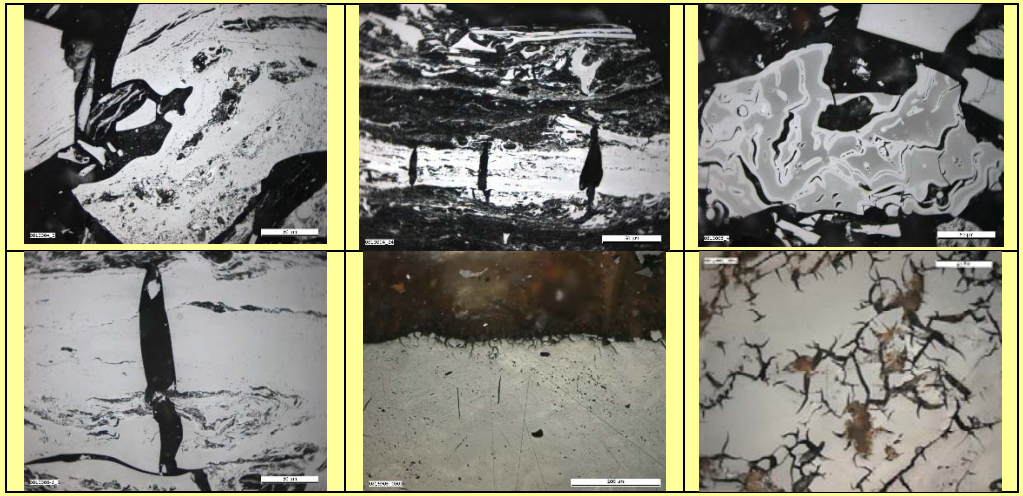
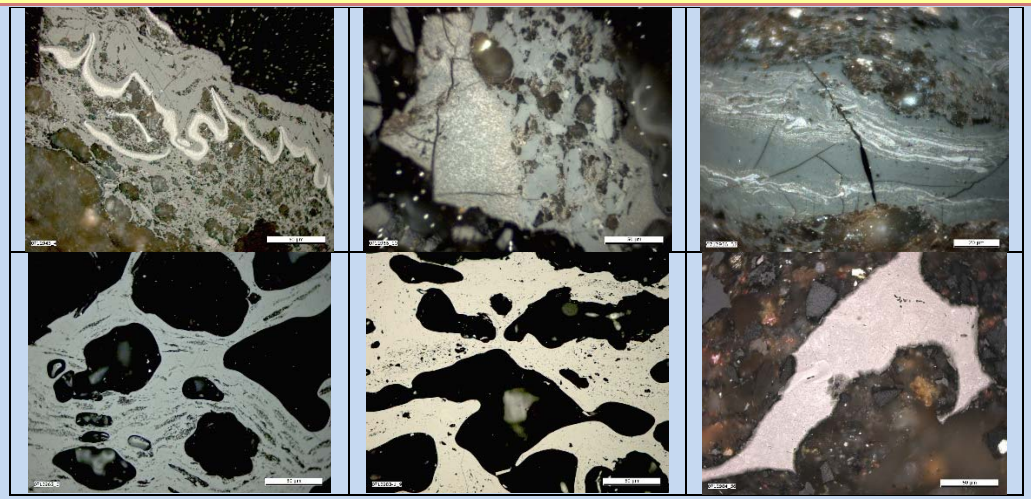
During this exercise, participants were asked to identify the selected oxidatively and thermally altered coaly particles in accordance to the established classification of oxidatively and thermally coaly particles. Secondly, participants were asked to make comments on the identified forms and on the applied classification.

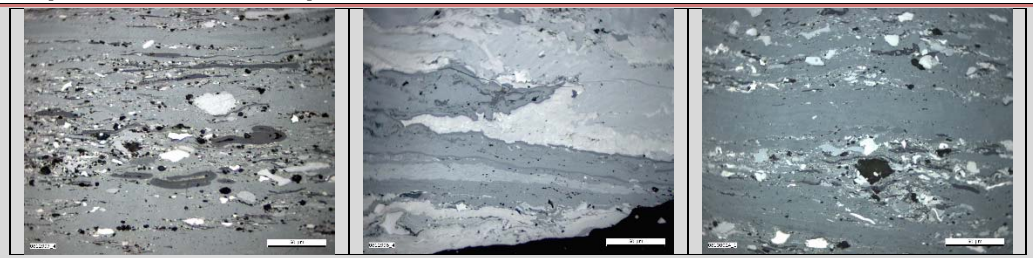
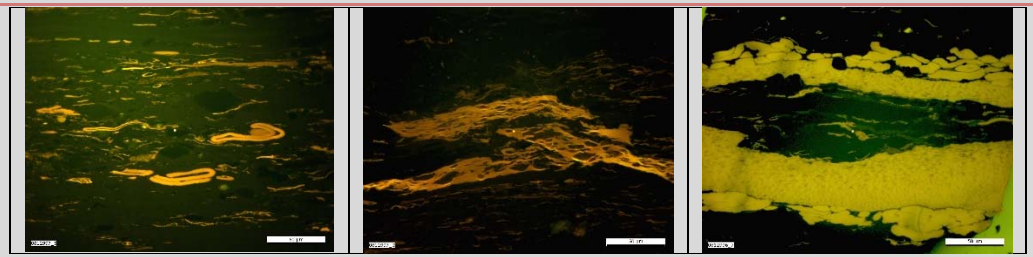
3 Classification of thermally and oxidatively affected coaly matter in self-heated coals

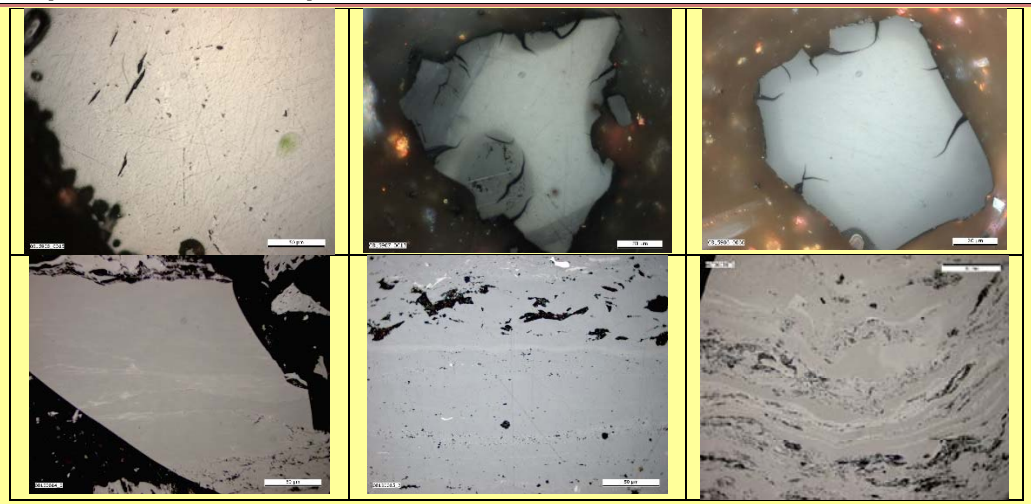
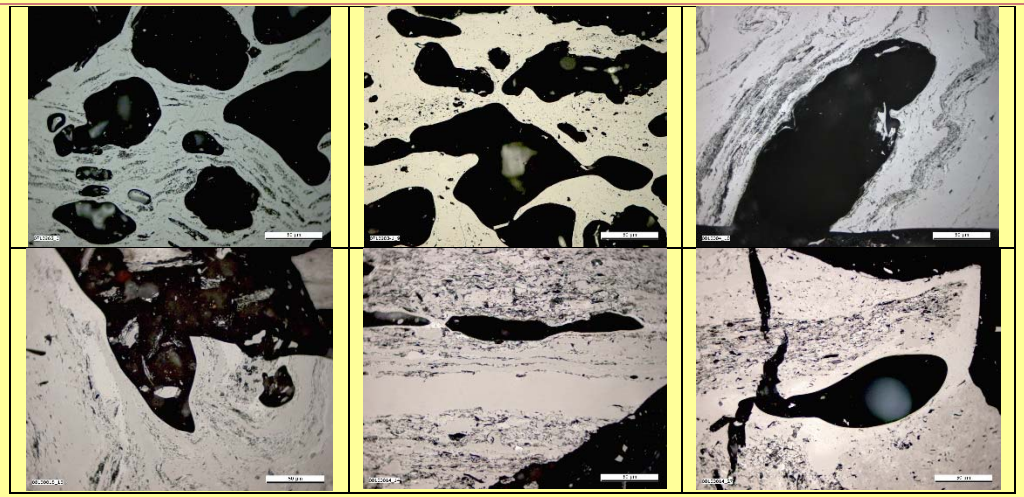
In the established classification of oxidatively and thermally coaly particles are divided into six levels:

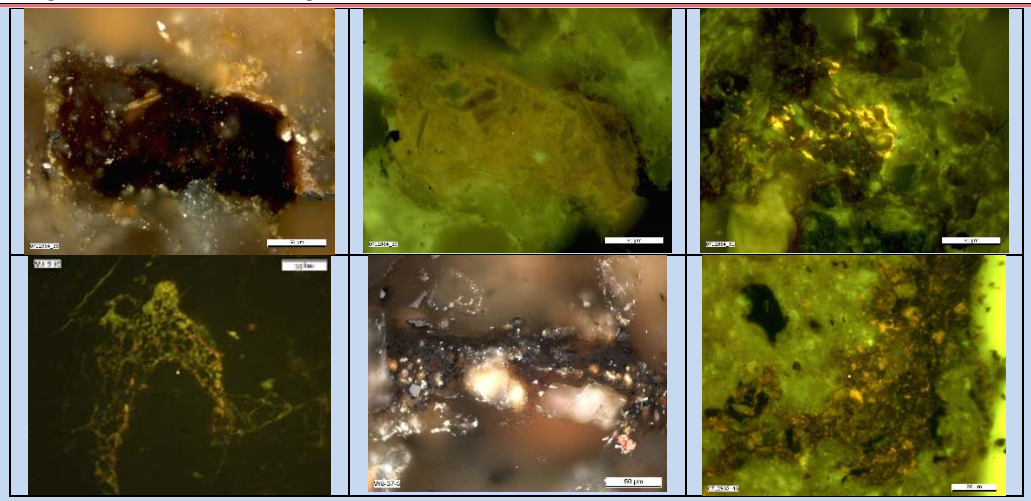
LEVEL 1	Description	Examples
LEVEL 1 **All particles**	**COALY PARTICLES** When composed of organic material	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
	MINERALS When composed of minerals and/or various mineral phases	

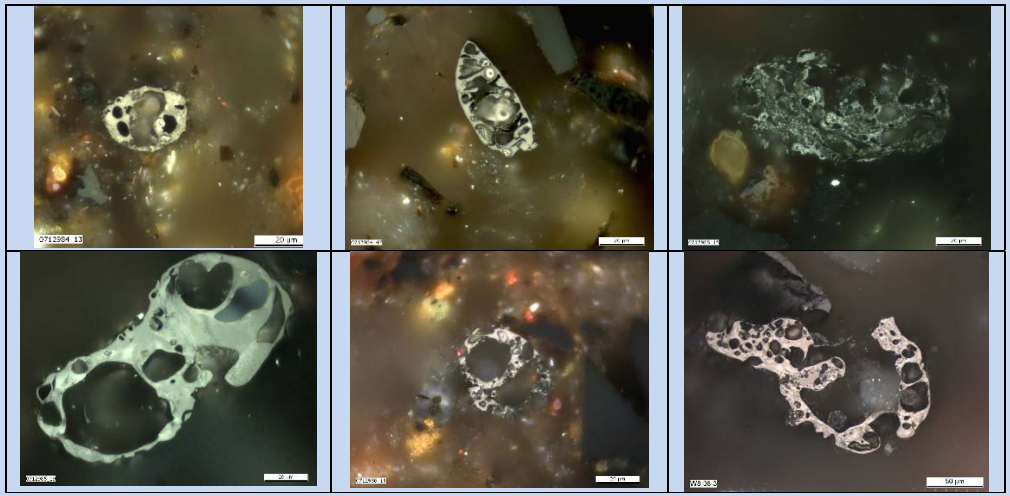
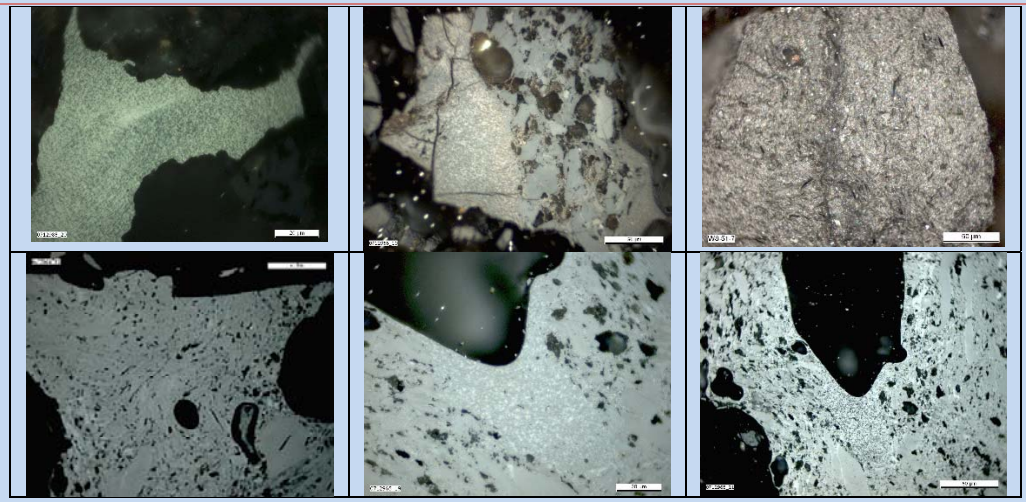
		oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
LEVEL 2	**Description**	**Examples**
LEVEL 2 NON-ALTERED	**NON-ALTERED** Coal macerals from self-heated coals displaying similar optical appearance and optical properties (morphology, micro-structure, microtexture, mean random vitrinite reflectance within the SD of the background unheated coal, appearance in reflected white light, polarised and nonpolarised light, fluorescence when irradiated) as unaltered coal macerals. Non-altered coal macerals display mean random vitrinite reflectance within the SD of the corresponding unaltered coal **and** lack any signs of alteration such as paler/darker oxidation rims, thermal fractures, thermal fissures and cracks, porosity connected with devolatilization, plasticised edges, and paler particles. For examples of thermal (shrinkage), fractures and fissures characteristic for thermally altered coals see Fig. 7 in Kus (2017). For further classification of non-altered particles see references: for huminite (Sýkorová et al., 2005), for vitrinite (ICCP, 1998), for liptinite (Taylor et al., 1998; Pickel et al., 2017), for inertinite (ICCP, 2001).	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**

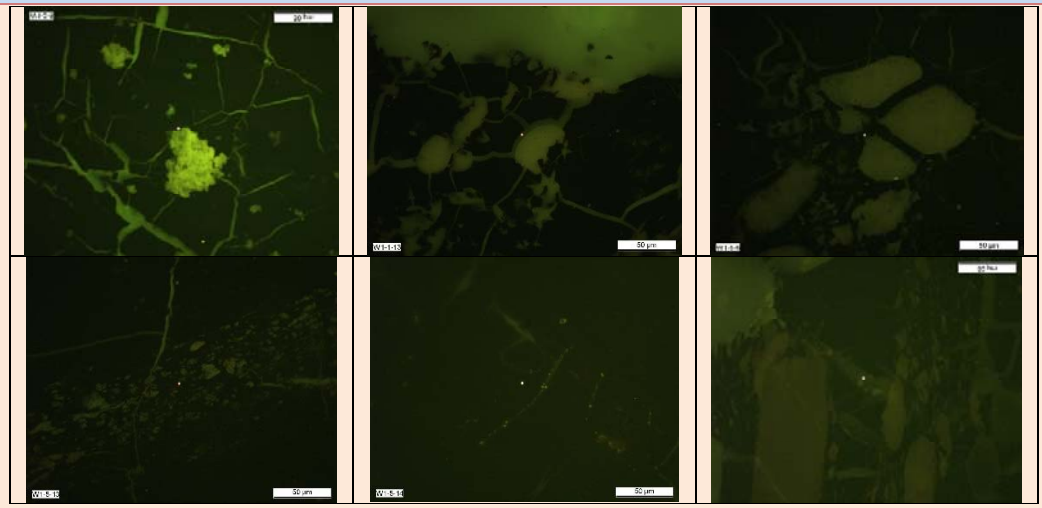
LEVEL 2 ALTERED	**ALTERED** Coal macerals with mean random or maximum reflectance equal or higher than that of the corresponding background value **and** with various signs of alteration such as: paler/darker oxidation rims, thermal fractures, thermal fissures and cracks, porosity connected with devolatilization, "pseudomicrinite", plasticised edges, and paler particles.	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
LEVEL 2 NEWLY FORMED	**NEWLY FORMED** Organic, non-maceral components or particles such as coke, pyrolytic carbon, char, and "pseudomicrinite" with mean random or maximum reflectance higher than that of the background vitrinite reflectance **and** particles displaying distinct microstructure, microtexture, anisotropy effects, signs of devolatilization, and fusibility. Newly formed bitumen is another component with mean random reflectance lower than the background vitrinite reflectance **and** of various shapes and strong yellowish fluorescence.	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**

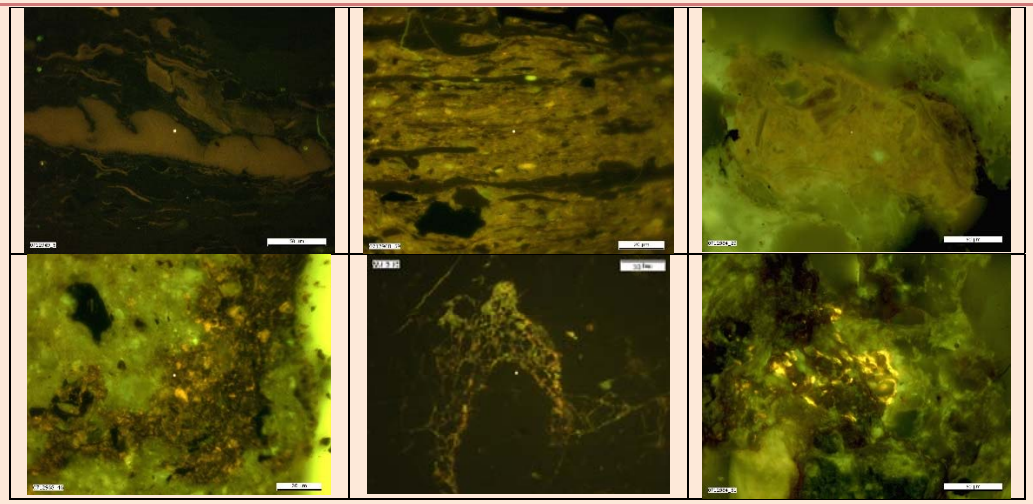
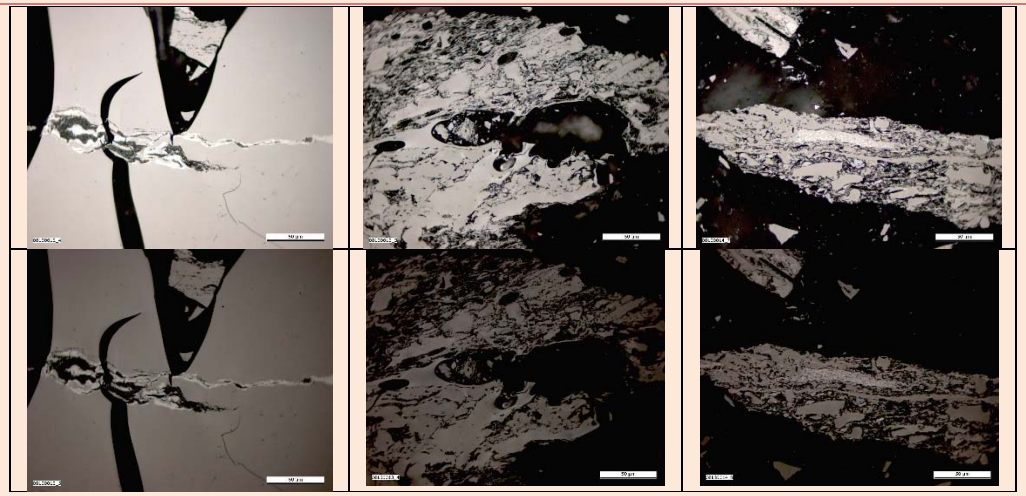
LEVEL 3 NON-ALTERED	**VITRINITE – NON-ALTERED**	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
	LIPTINITE – NON-ALTERED	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
	INERTINITE – NON-ALTERED	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**

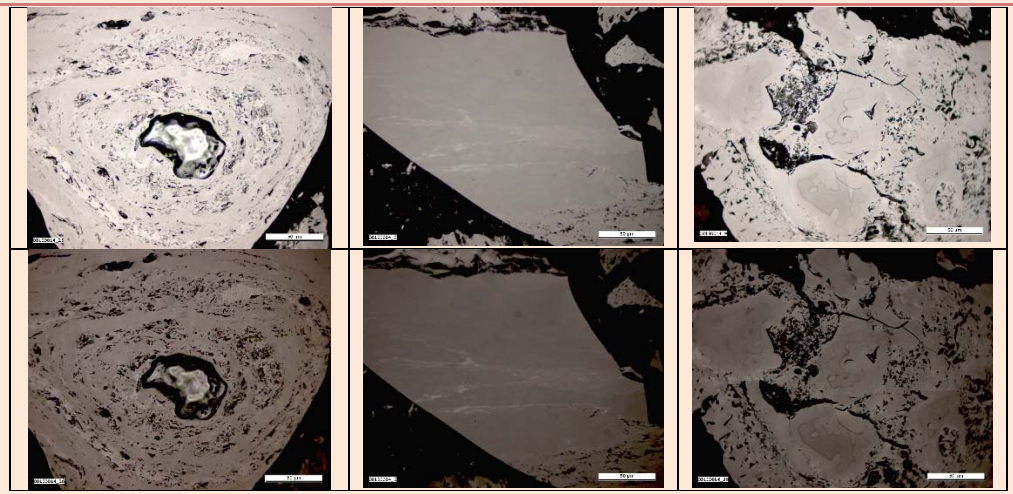
LEVEL 3 ALTERED	**<u>MASSIVE –</u> ALTERED** Area without Devolatilisation Pores	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
	<u>POROUS –</u> ALTERED Area with Devolatilisation Pores	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**

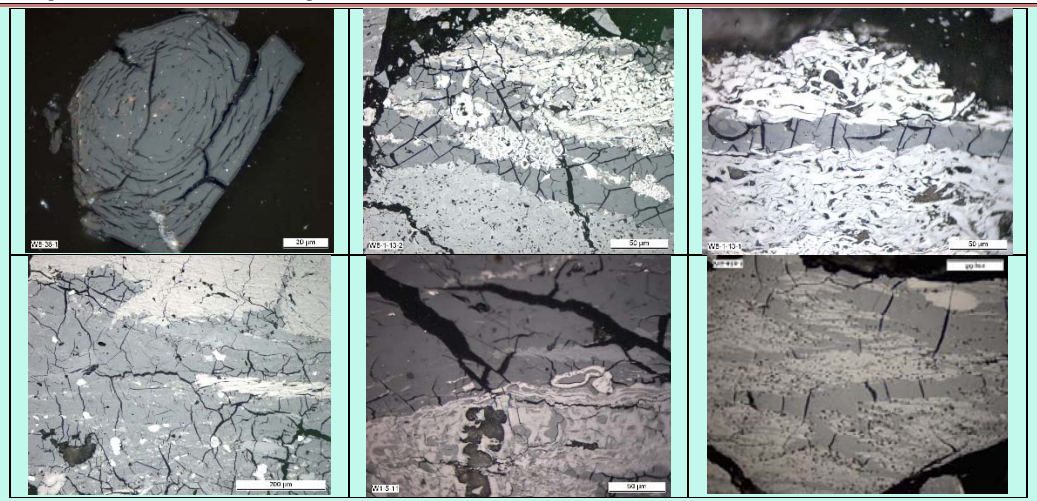
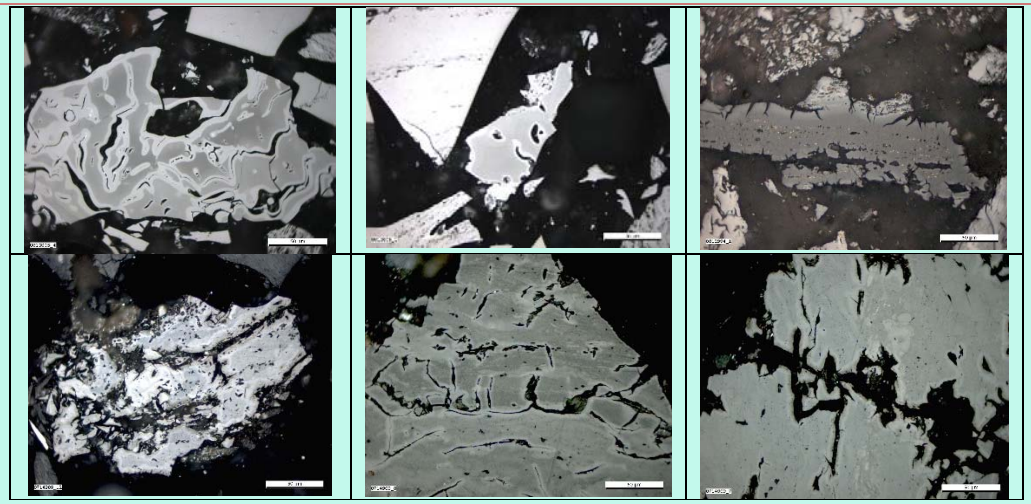
LEVEL 3 NEWLY FORMED	**BITUMEN – NEWLY FORMED** Expulsions of hydrocarbons generated during self-heating processes largely from liptinite macerals; they have various shapes: droplets, thread-like structures or are of irregular nature; commonly they co-occur with minerals; with mean random reflectance lower than the background vitrinite reflectance and of strong yellowish fluorescence; for further reference see Alpern et al. (1992).	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
	PYROLYTIC CARBON – NEWLY FORMED Strongly anisotropic organic matter occurring in the form of ribbons, thin laminae; it occurs separately, on the edges of forms or within strongly thermally altered particles; for further references see Taylor et al. (1998) and Kwiecińska and Pusz (2016).	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**

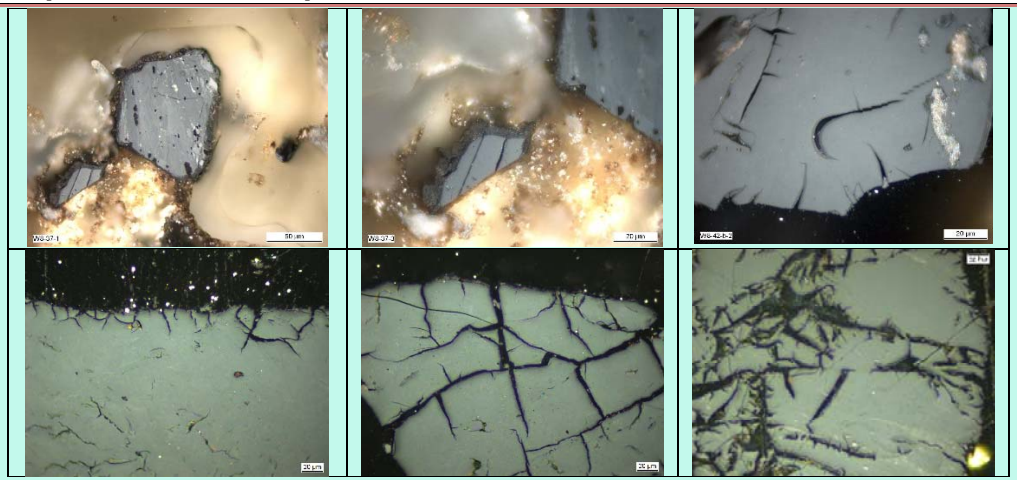
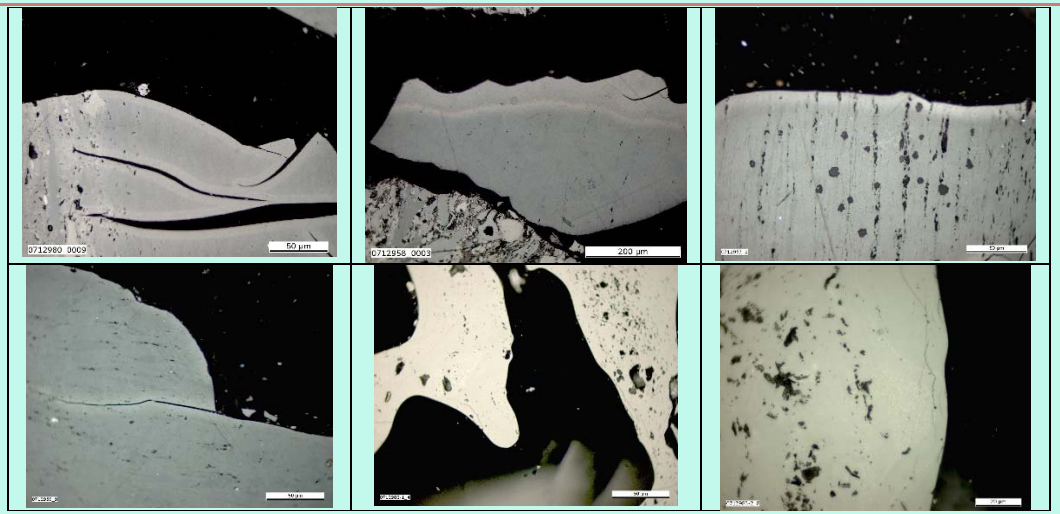
LEVEL 3 NEWLY FORMED	**<u>CHARS –</u> NEWLY FORMED** Organic particles with pyrolysis char morphology characterised by randomly distributed pores and varying porosity, shape, and size; their colour in reflected white light is light grey to white, and the reflectance is always higher than the prime huminite/vitrinite; Kwiecińska and Petersen (2004).	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
	<u>COKE –</u> NEWLY FORMED Coal heated in the absence of air, vesiculated and harden into coke. For further reference on similar textures and structures in metallurgical coke see Suárez-Ruiz and Crelling (2008), on natural coke Kwiecińska and Petersen (2004).	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**

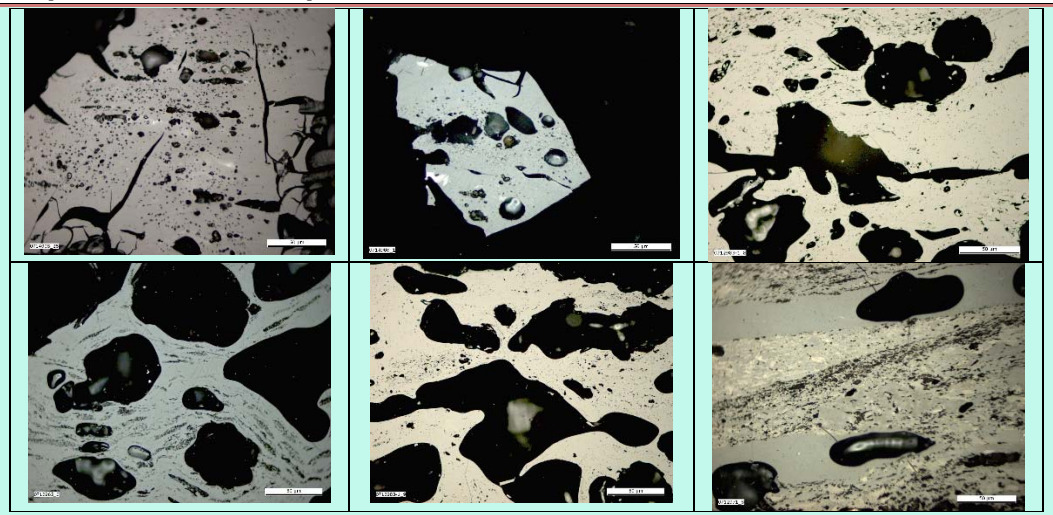
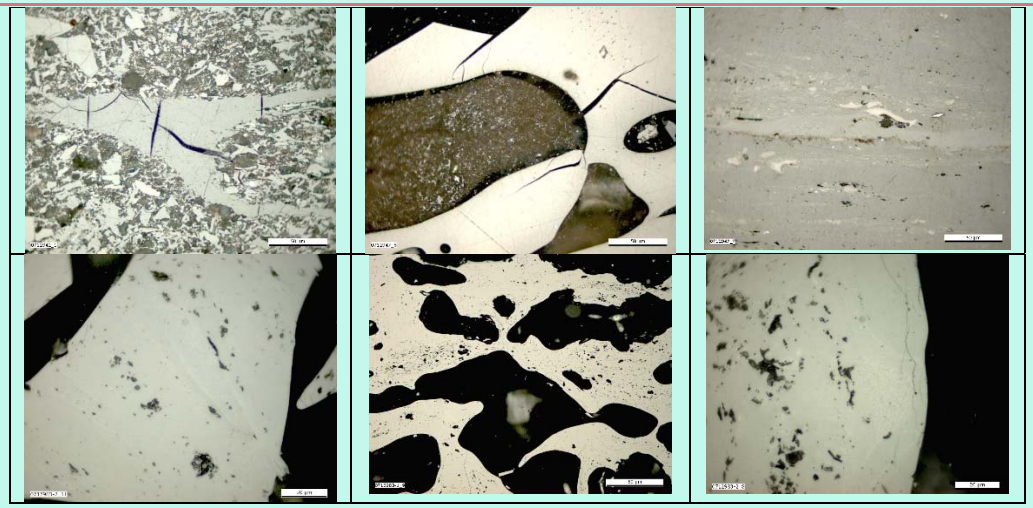
LEVEL 3 **NEWLY FORMED**	**<u>"PSEUDOMICRINITE" –</u>** **NEWLY FORMED** Rapid heating of liptinite during the self-heating process can lead to formation of substance with optical appearance resembling that of micrinite. It is called now "Pseudomicrinite" The newly formed "Pseudomicrinite" mimics the shape, outline of former (unaltered) cutinite, sporinite and other liptinite macerals. It occurs as fine-grained material chara-cterised by a high reflectance.	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
LEVEL 4 **NON-ALTERED**	**<u>NON-FLUORESCENT –</u>** **NON-ALTERED** **ALTERED** **NEWLY FORMED** Coal grains lacking fluorescence.	

ALTERED NEWLY FORMED		oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
	<u>FLUORESCENT –</u> NON-ALTERED ALTERED NEWLY FORMED Coal grains displaying fluorescence.	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
LEVEL 5 NON-ALTERED ALTERED NEWLY FORMED	**<u>ISOTROPIC –</u> NON-ALTERED ALTERED NEWLY FORMED** Coal grains with lack of optical anisotropy in polarised light.	

		oil immersion (n = 1.518) in reflected monochromatic **non-polarized and polarized incident light, 50x, respectively**
	<u>NON – ISOTROPIC</u> – **NON-ALTERED** **ALTERED** **NEWLY FORMED** Coal grains with optical anisotropy in polarised light.	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized and polarized incident light, 50x, respectively**

LEVEL 6 ALTERED NEWLY FORMED	**THERMAL FRACTURES, FISSURES, CRACKS – ALTERED NEWLY FORMED** Non-inherent and non-tectonic fractures and fissures; single to bifurcated, dendritic to biconcave fractures and fissures, occurring at edges of coal grains or within coaly particles; irregularly distributed; can extend across coal macerals although often stop at boundaries with inertinite macerals or mineral matter (Kus, 2017).	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
	PALER OXIDATION RIMS - ALTERED NEWLY FORMED Paler rims of higher reflectance occurring along the edge of particles, at fractures, fissures or around devolatilisation pores.	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**

LEVEL 6 ALTERED NEWLY FORMED	**<u>DARKER OXIDATION RIMS</u> - <u>ALTERED</u> <u>NEWLY FORMED</u>** Darker rims of higher reflectance occurring along the edge of particles, at fractures, fissures or around devolatilisation pores.	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
	<u>PLASTICISED EDGES</u> – <u>ALTERED</u> <u>NEWLY FORMED</u> Particles with plasticised or mouldable external, rounded to sub-rounded edge shape; smooth contours; gradual in contour and lack, for the most part, the abrupt discontinuity at the edge; often associated with paler oxidation rim.	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**

LEVEL 6 ALTERED NEWLY FORMED	**<u>DEVOLATILISATION PORES</u> – <u>ALTERED</u> <u>NEWLY FORMED</u>** Particles with pores that are usually round or oval in shape, sometimes they are elongated; their size is from a few to tens of µm; only pores related to thermal processes are included; porosity being an original feature of maceral, e.g. porosity of fusinite, is not included here.	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**
	<u>PALER PARTICLE</u> – <u>ALTERED</u> <u>NEWLY FORMED</u> Vitrinite grains of higher reflectance compared with the background value.	 oil immersion (n = 1.518) in reflected monochromatic **non-polarized incident light, 50x**

The following classification of thermally and oxidatively altered coaly matter in coal (Fig. 1) has been adapted and modified from the classification of thermally and oxidatively altered coaly matter in coal wastes published by Misz-Kennan et al. (2020). The modification included the following: in the Level 3, the category “micrinite” has been replaced by the category “pseudomicrinite” and in the Level 6 the category “bands” has been removed.

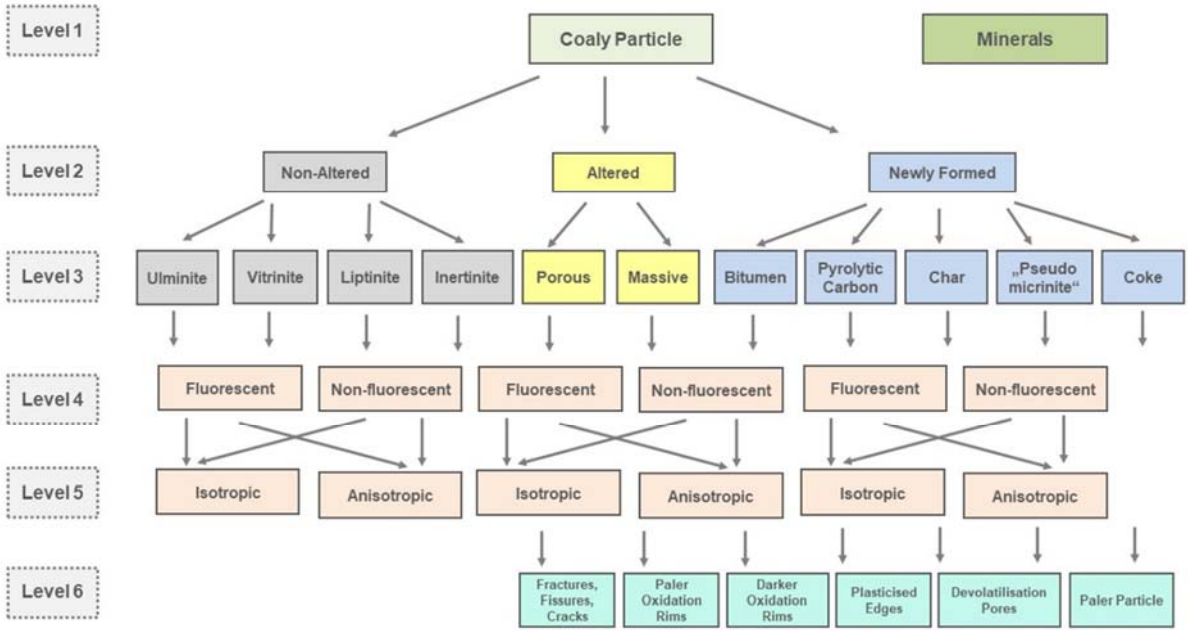


Fig. 1. Classification of thermally and oxidatively altered coaly matter in self-heated coals adopted from classification of thermally and oxidatively altered coaly matter in coal wastes (modified after Misz-Kennan et al., 2020).

4 Round Robin Exercise

The 2020 Round Robin Exercise was based on 52 particles in 210 slides that were prepared from high volatile coals with the background mean random vitrinite reflectance of 0.94% VRr and with standard deviation of 0.04, corresponding to the reflectance value of an unaltered coal. Each particle under examination in the respective slide is taken under reflected non-polarised white-light (RL), UV fluorescence as well as under white-light polarised at 0° and polarised at 90°. The slides contain also information on the mean random, mean maximum, and mean minimum vitrinite reflectances of the sample (not on the particle under examination!). In addition, in the right upper corner a small window is attached with the background colour of an altered high volatile bituminous coal.

Participants were asked to:

- Determine the form of organic matter in the rectangle in accordance to the established classification (see chapter 2) and mark the answer in the attached Excel file (2020 Round Robin Exercise SHWG.xls).

How to mark answers in the attached Excel file according to the established classification (see chapter 2):

- In case of **Level 1: Coaly Particle of Minerals**, please mark **one** of them.
- In case of **Level 2: Non-Altered, Altered and Newly Formed,** please mark **one** of them.
- In case of **level 3: Non-Altered – Vitrinite, Liptinite, Inertinite,** please mark **one** of them.
- In case of **level 3: Altered – Porous, Massive**, please mark **one** of them.
- In case of **level 3: Newly Formed – Bitumen, Pyrolytic Carbon, Chars, "Pseudomicrinite", Coke,** please mark **one** of them.
- In case of **level 4: Altered – Fluorescent and Non-Fluorescent**, please mark **one** of them for the selected option in Level 3.
- In case of **level 5: Altered – Isotropic and Non-Isotropic**, please mark **one** of them for the selected option in Level 3.
- In case of **level 6: Newly Formed – Fractures, Fissures; Paler Oxidation Rims; Darker Oxidation Rims; Plasticised Edges; Devolatilisation Pores; Paler Particle**, please make **multiple answers**, if adequate for the Altered and Newly Formed categories.

5. Results of the 2020 Round Robin Exercise

The 2020 Round Robin Exercise involved participation of 17 parties, mainly from academia with Magdalena Misz-Kennan, Ali İhsan Karayiğit, Georgeta Predeanu, Ivana Sýkorová, Joana Ribeiro, Kimon Christanis, Nikki Wagner, Sławka Pusz, Stavros Kalaitzidis, Zeynep Buckun, Paul Hackley, Dragana Životić, Małgorzata Wojtaszek-Kalaitzidi, Piotr Wojnowski, Sandra Rodrigues, Tara Congo, and Georgia Petratou. The aspects, which are discussed in the sub-sequent chapters, are pointed out.

6. Evaluation of results

The evaluation of results obtained in the 2020 Round Robin Exercise followed three different evaluation schemes, which were as follows:

1. **Raw Agreement Indices** with only the Overall Level of Agreement being evaluated for each of the categories in this report.
2. **Standard Statistical Evaluation** with the minimum, maximal, and average level of agreement values being evaluated.
3. **Detailed Statistical Evaluation**

6.1 Raw Agreement Indices

The raw agreement indices were applied to categories identified by participants and were established by Uebersax (2001) as well as used by Predeanu et al. (2015). The evaluated index for the obtained results is the overall level of agreement (LOA). LOA considers both the correct identified answers (positive identification) as well as lack of response, but identified and not marked (negative identification), as well as incorrect responses (disagreement). The LOA gives a broader information of the evaluation status and is more accurate. The equation used is given as:

$$Po=a+d/a+b+c+d=a+d/N$$

where:
a is the number of positive agreement;
d is the number of negative agreement;
b and c are the numbers of disagreement

The results obtained by each of the participants point towards in general to a generally satisfactory outcome.

Non-Altered Particles

The LOA for the category Non-Altered Particles as displayed in Fig. 6 and as identified by each of participants reveals well-comparable results among the participants. The LOA ranges between 87% and 100% and its average amounts to **95.8%**.

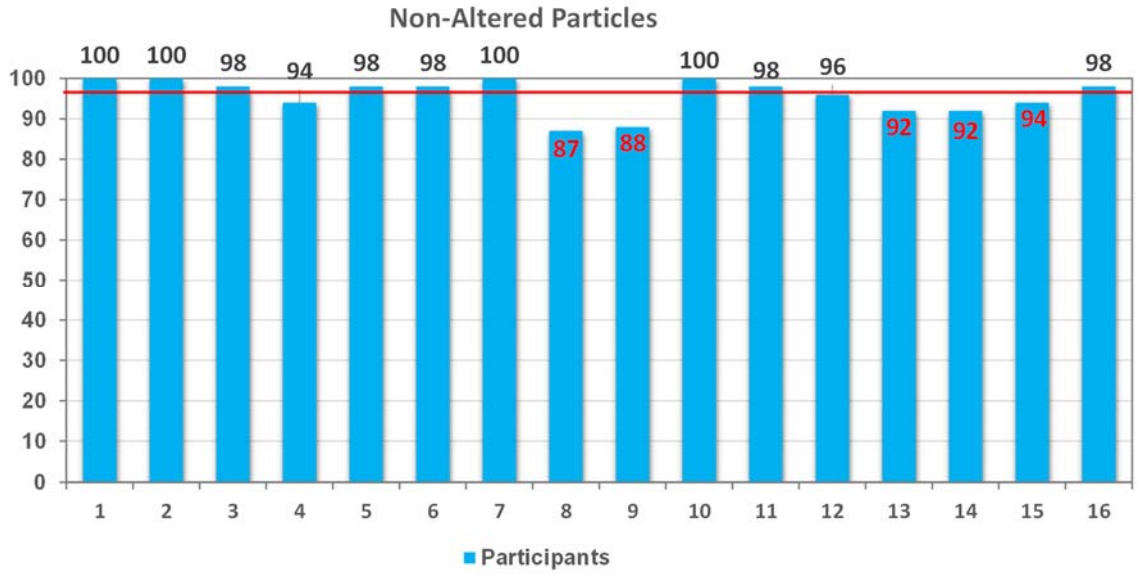


Fig. 6. Level of overall agreement for the category Non-Altered Particles obtained by the participants in the 2020 Round Robin Exercise.

Altered Particles

The next category Altered Particles displays a slightly higher range of the data for the 17 participants with values ranging between 77% and 100% and the average LOA of **95.1%** (Fig. 7).

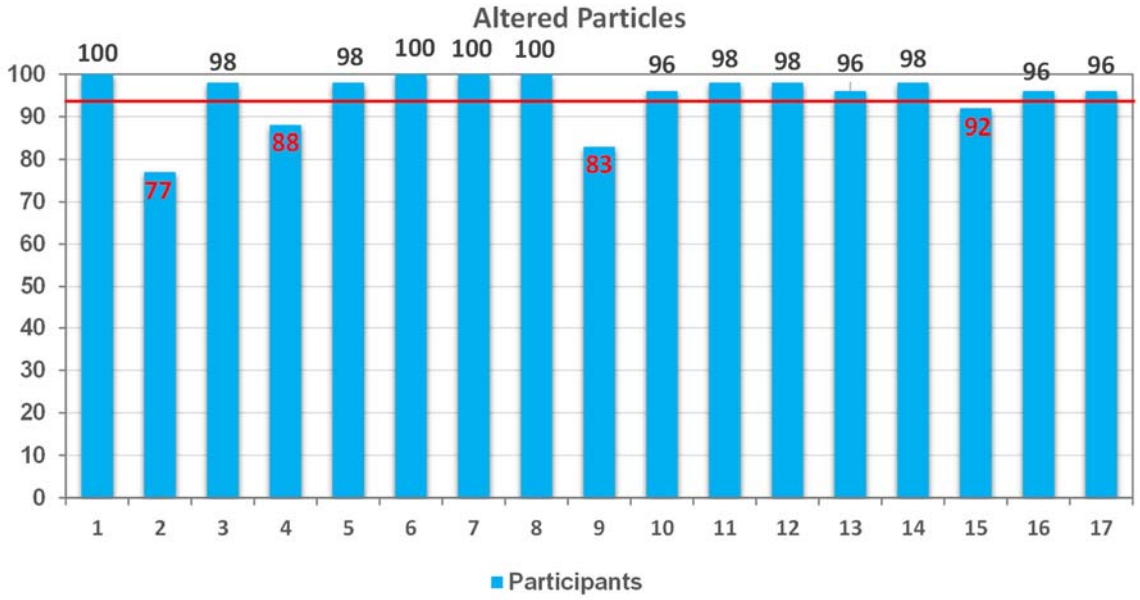


Fig. 7. Level of overall agreement for the category Altered Particles obtained by the participants in the 2020 Round Robin Exercise.

Newly Formed Particles

A better scenario of LOA is shown for the Newly Formed Particles category with a range between 83% and 100% and the average LOA of **95.7%** (Fig. 8).

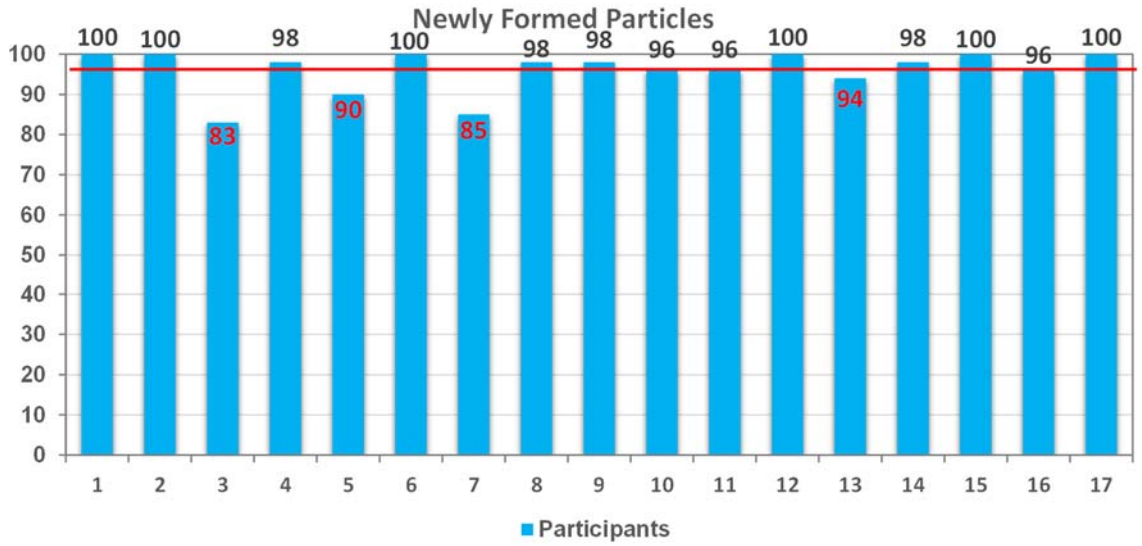


Fig. 8. Level of overall agreement for the category Newly Formed Particles obtained by the participants in the 2020 Round Robin Exercise.

Vitrinite Non-Altered

The LOA for the category Vitrinite Non-Altered as displayed in Fig. 9 shows well-comparable results among the 17 participants with an average of LOA of **98.8%**.

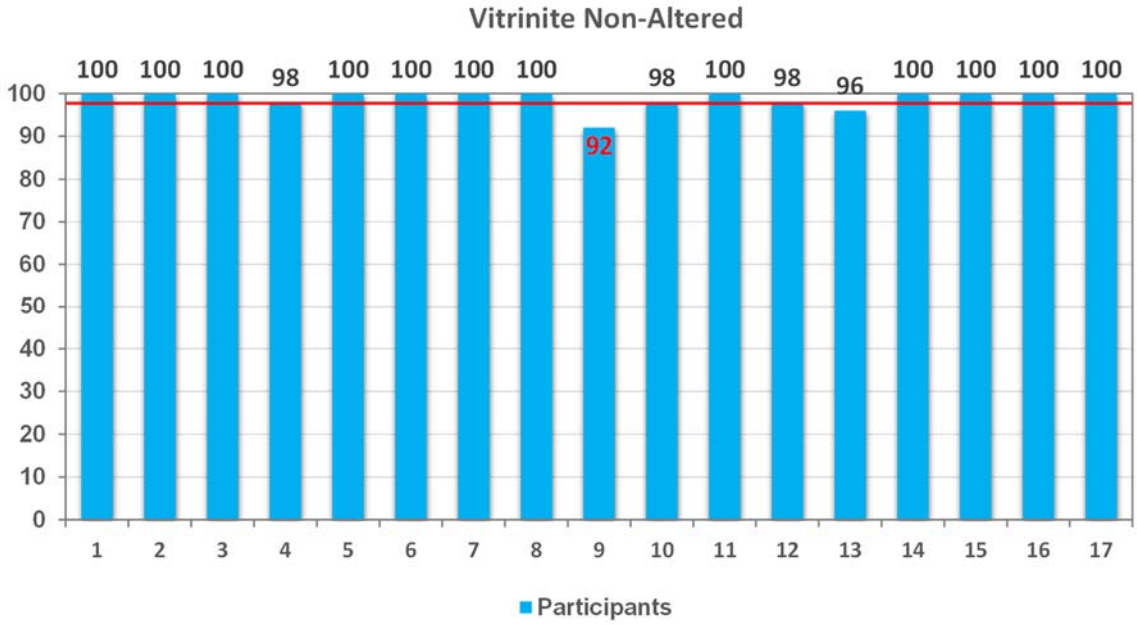


Fig. 9. Level of overall agreement for the category Vitrinite Non-Altered obtained by the participants in the 2020 Round Robin Exercise.

Massive

A still satisfactory, although slightly lower LOA is shown for the category Massive with a range between 75% and 98% and the average LOA of **92.9%** (Fig. 10).

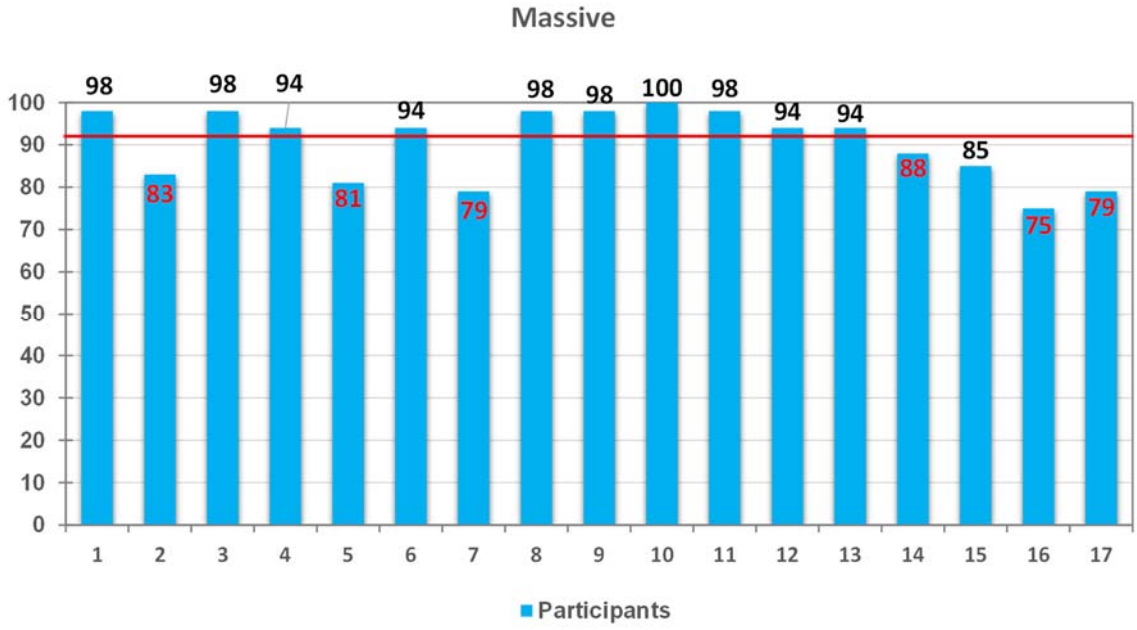


Fig. 10. Level of overall agreement for the category Altered Vitrinite obtained by the participants in the 2020 Round Robin Exercise.

Porous

A still satisfactory, although markedly lower LOA is shown for the category Porous with a range between 63% and 100% and the average LOA of **87.1%** (Fig. 11).

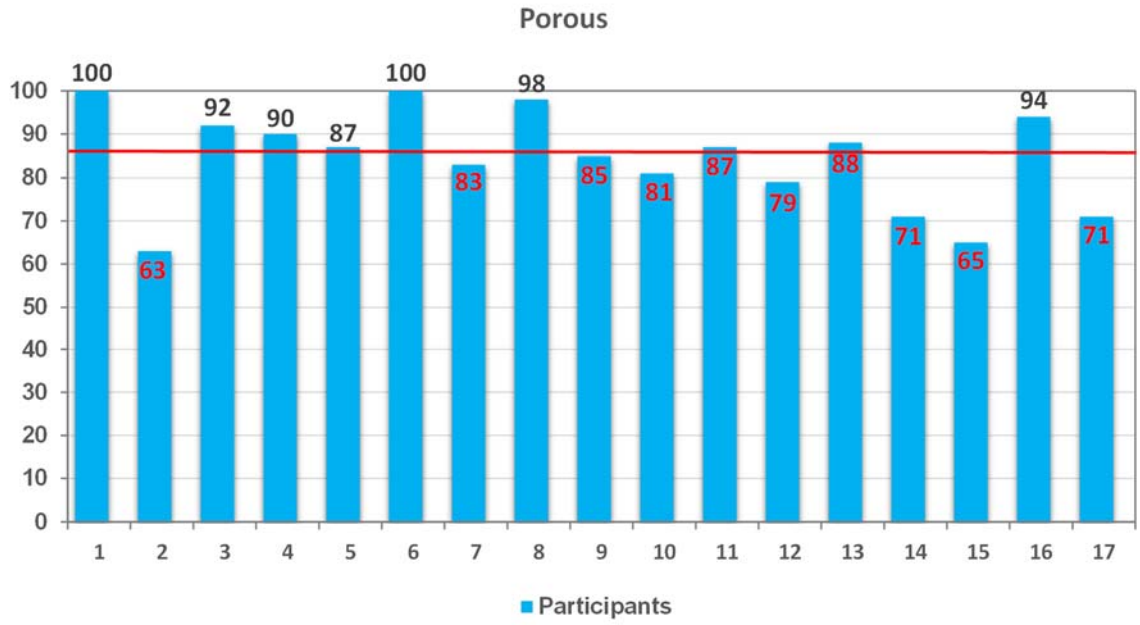


Fig. 11. Level of overall agreement for the category Porous obtained by the participants in the 2020 Round Robin Exercise.

Isotropic

A well-comparable outcome among the participants was obtained for the category Isotropic as displayed in Fig. 12. The average LOA amounts to **92.8%**.

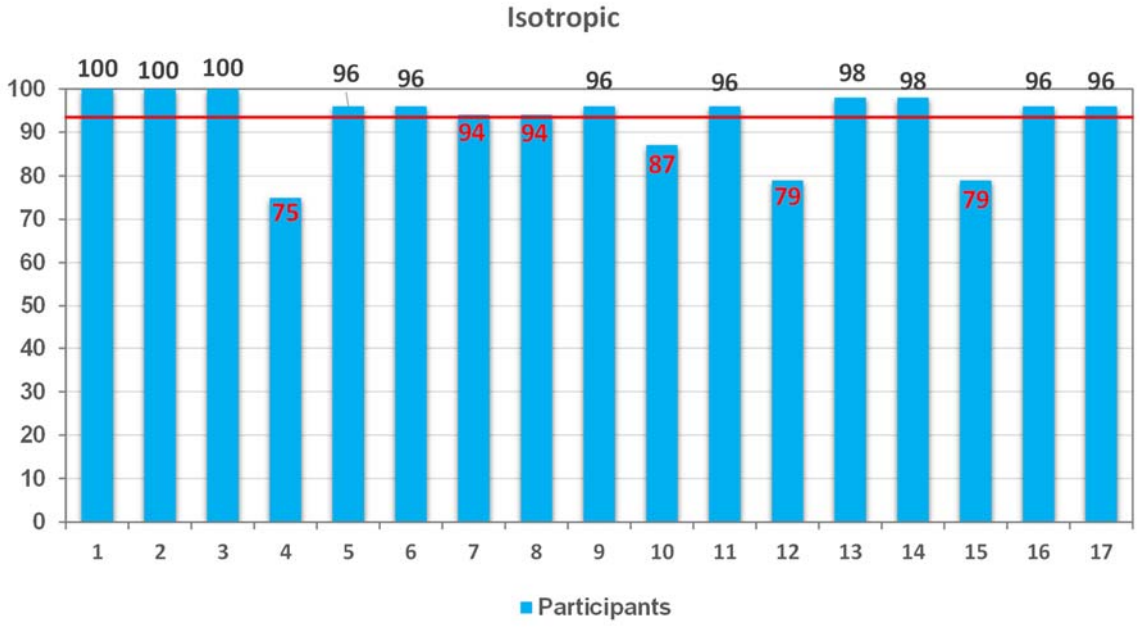


Fig. 12. Level of overall agreement for the category Coke obtained by the participants in the 2020 Round Robin Exercise.

Non Isotropic

A less satisfactory level of LOA is shown by the category Non isotropic with the lowest average level of LOA of **74.6%** (Fig. 13).

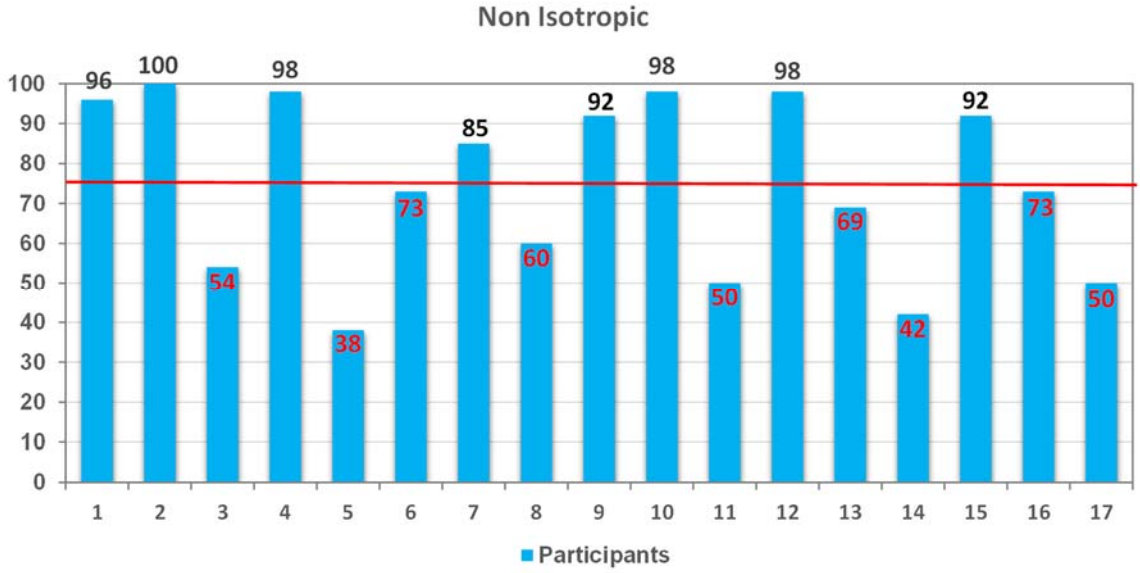


Fig. 13. Level of overall agreement for the category Isotropic obtained by the participants in the 2020 Round Robin Exercise.

Paler in Colour Particle

The most homogeneous distribution of LOA among the participants with the average of LOA of **99.5%** is displayed for the category Paler in Colour Particle in Fig. 14.

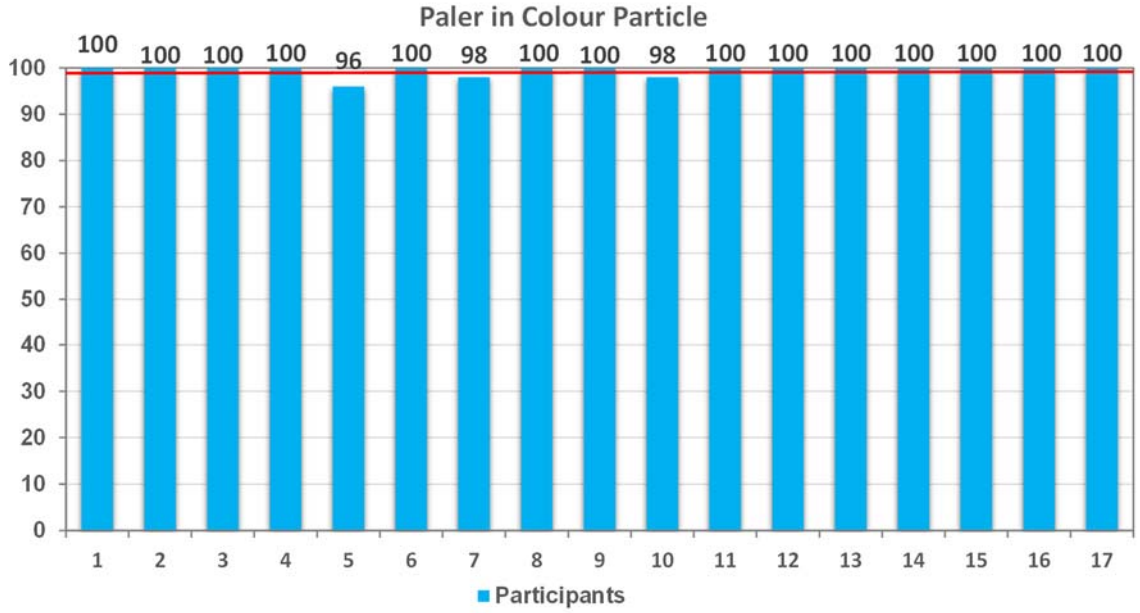


Fig. 14. Level of overall agreement for the category Paler in Colour Particle obtained by the participants in the 2020 Round Robin Exercise.

6.2 Standard Statistical Evaluation

Standard statistical evaluation of the obtained results follows a different approach, namely a comparison of minimum, maximum, and average level of agreement (in %) obtained by all participants for categories evaluated in images (Fig. 15).
For the most categories, the average level of correctly identified forms to amounted to equal or more than **90%** as marked by the blue colour. For the categories marked in yellow, on average, between **87.4%** and **88.0%** of the following categories: Altered, Fractures, fissures, Plasticised edges, and Devolatilisation pores were correctly identified. In the remaining five categories: Massive, Porous, Isotropic, Non-isotropic, and Paler in Colour Particles the corresponding particle posed distinct difficulties. On average, only **59.4%** and **78.8%** of these forms were correctly identified. These categories display also a relatively high dispersion of data sets.

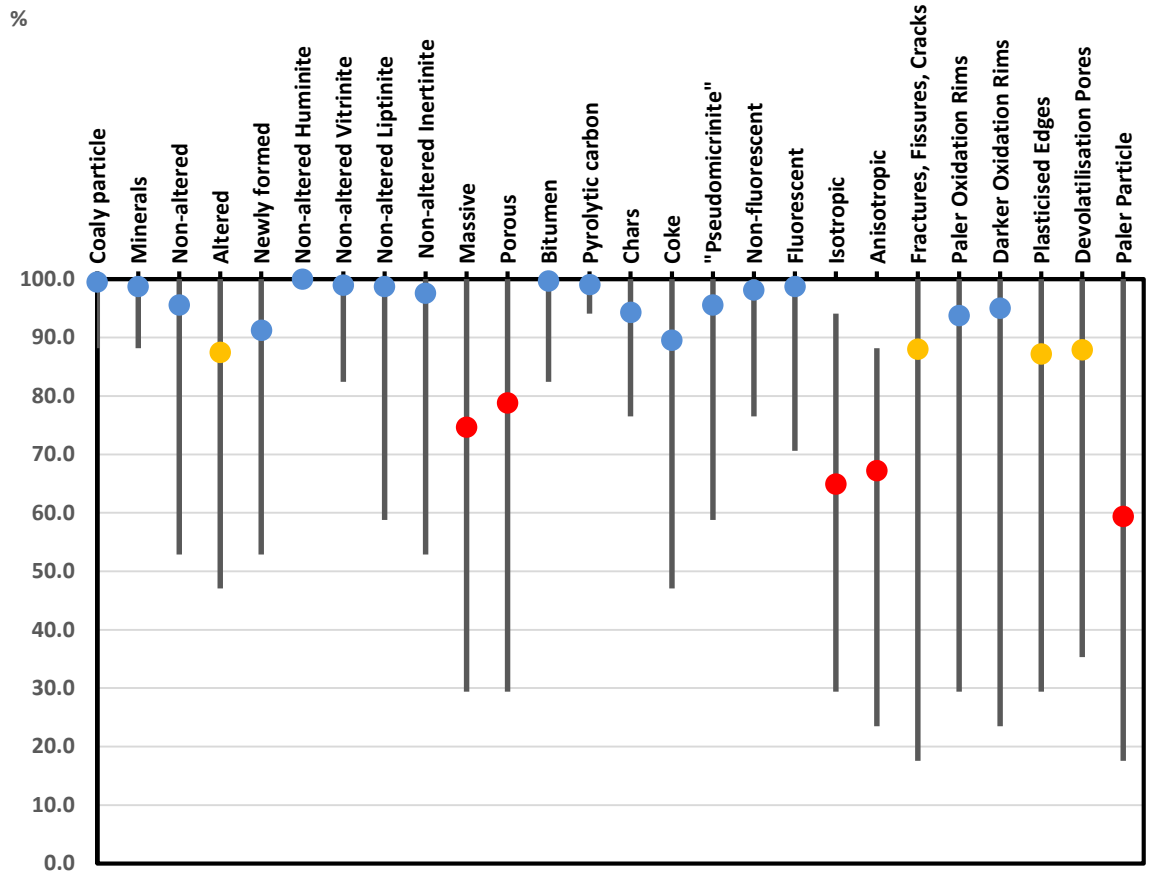


Fig. 15. Minimum, maximum, and average values (in %) of correctly identified forms obtained for individual morphological forms of organic matter in thermally and oxidatively altered coals by participants of 2020 Round Robin Exercise.

6.3 Detailed Statistical Evaluation

Ewa Szram, a colleague of Magdalena Misz-Kennan from the Department of Natural Sciences at the Silesia University in Sosnowiec, Poland applied a scheme of a detailed statistical evaluation to the results obtained by participants for the categories and forms in images.

A. The following statistics refers to evaluation of data with respect **to how participants identified given category in the examined slides.**

Detailed statistical evaluation embraces the so-called Grubb's test and the box-and-whisker diagram and were applied to check for outliers and extreme values and to identify these deviations in the data sets.

The **Grubb's test** checks for outliers by comparing maximum values of the absolute differences between the values and their mean. It evaluates the presence of outliers in a univariate data set, assuming the data are described to have a normal distributed population. Thus, the results indicate a probability that the evaluated data belongs to the core population. The applied level of significance was established to **0.05** for number of slides that were identified: **n=52**. In Tab. 1 results of the Grubb's test for evaluating the forms in images are displayed. Based on the applied level of significance and number of identified forms, the critical value is equal to **2.97**. Values, which are lower than **2.97** indicate lack of outliers and are highlighted in Tab.1. These include among others Massive and Porous forms. The remaining values suggest the presence of deviance and statistical irregularities.

Table 1. Results of the Grubb's test applied to categories in the evaluated categories in images.

Coaly Particle	Minerals	Non-Altered	Altered	Newly Formed Particle	Non-Atered Huminite	Non-Altered Vitrinite	Non-Altered Liptinite	Non-Altered Inertinite	Massive	Porous	Bitumen	Pyrolytic carbon	Chars	Coke	Pseudomicrinite	Non-fluorescent	Fluorescent	Isotropic	Anisotropic	Fractures, Fissures Altered	Paler in colour oxidation rims	Darker in colour oxidation rims	Plasticised edges	Devolatilisation pores	Paler in Colour Particles
5.76	3.91	4.48	3.22	3.67	**NA**	5.12	6.13	5.83	**2.17**	**2.45**	7.07	2.32	3.96	3.33	3.68	4.88	5.23	**1.93**	**2.66**	3.63	5.69	5.26	3.37	3.27	**2.19**

In the **box-and-whisker diagram**, the lower and upper limits are used as diagnostic tools to identify observations that might be outliers and extreme values. Observations, which lie below or above these limits, are termed outliers and extreme values.
For the categories identified in images by participants as displayed in Tab. 1, box-and-whisker diagram in Fig. 16 shows a markedly high numbers of min and max values, outliers, and extreme values. This might be caused by the fact that majority of the evaluated categories follows a non-normal distribution as confirmed by the outcome of the Kolmogorov-Smirnov, Lilliefors, and Shapiro-Wilk tests at a level of confidence of **0.05**. The presence of the outliers and extreme values can significantly alter or misrepresent statistical results. The box-and-whisker diagram for the recognized categories provides also other statistical information. Sample size of data evaluated for each of the identified categories in Fig. 16 does not affect the appearance of the diagram as the population, i.e., the sample size is more than 20. With regard to the skewness, only the data in categories Massive, Isotropic, Anisotropic, and Paler Particle is left-skewed, whereas Porous are right-skewed.

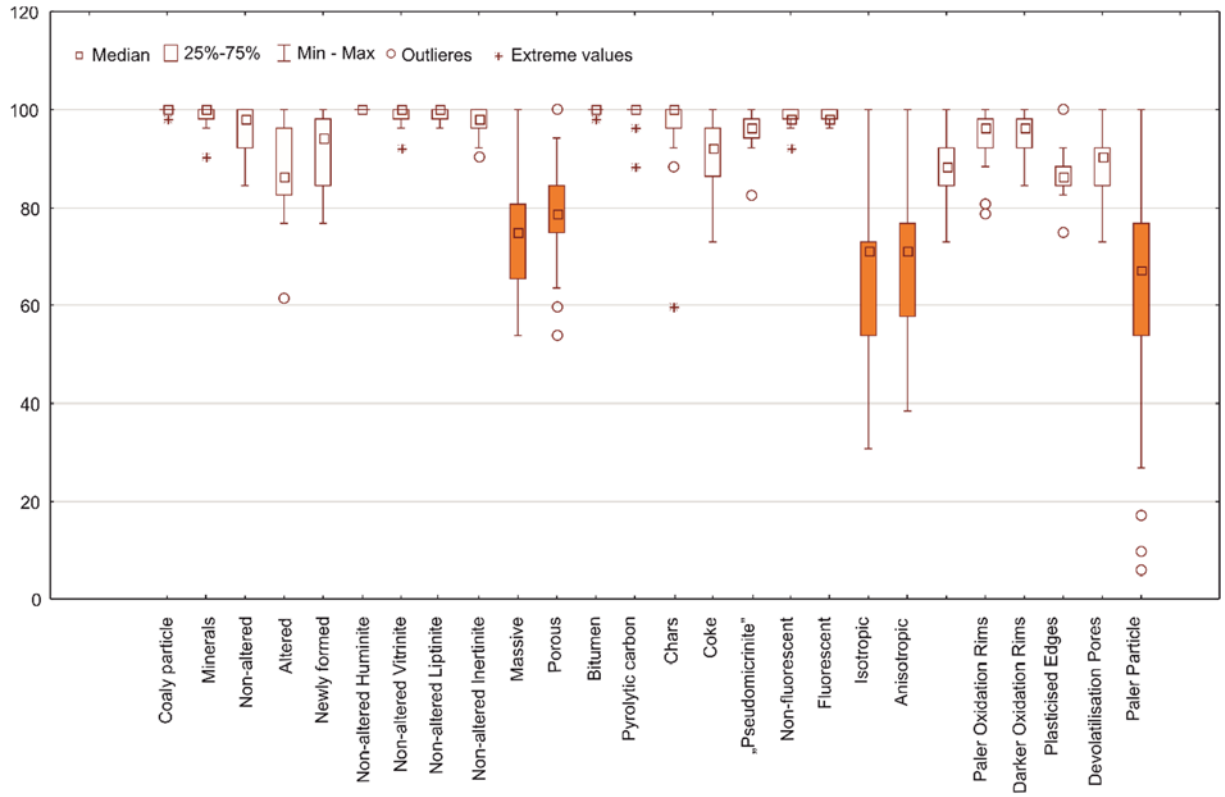


Fig. 16. The box-and-whisker diagram with extreme values, outliers, median and non-outlier range (25%-75%), obtained for 25 categories (Coaly particle to Paler in colour particle) in thermally and oxidatively altered coals in the 2020 Round Robin Exercise.

The range between the highest and the lowest value shows the highest degree of dispersion (spread). It gives an idea of how much variation is observed in the displayed categories. The highest range is observed in the category Paler Particle. As the range takes the count of extreme values only, it sometimes might not reflect an adequate degree of variability. The Interquartile range (IQR) is a more representative measure of data spread as it not affected by outliers and extreme values. The highest IQR is also noted and Paler in Colour Particle.

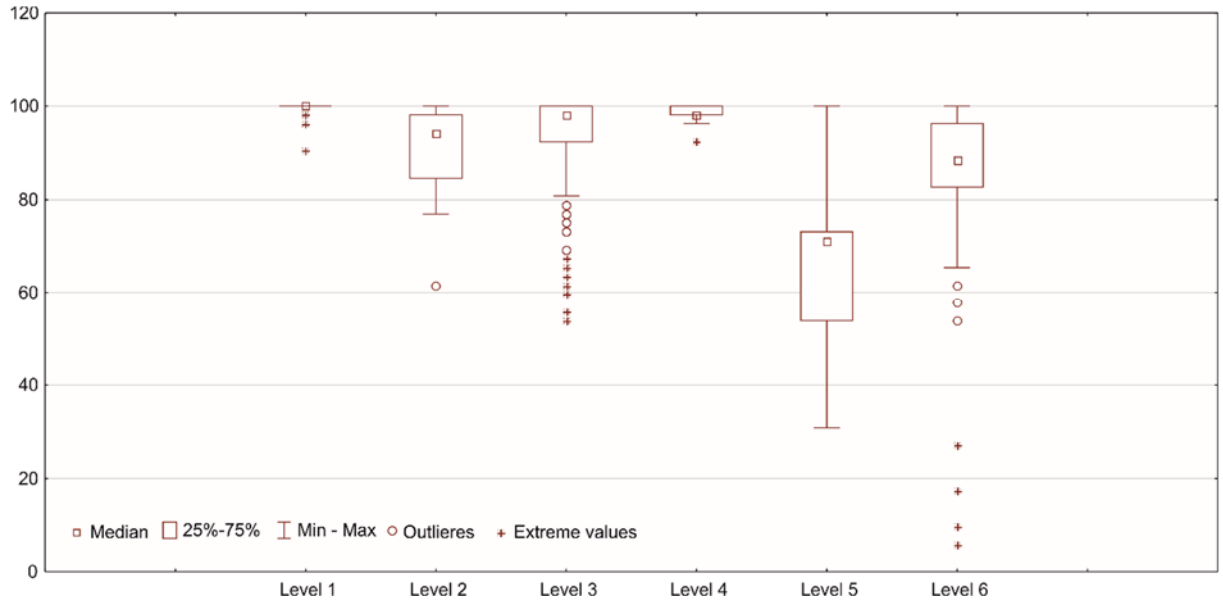


Fig. 17. The box-and-whisker diagram with extreme values, outliers, median and non-outlier range (25%-75%), obtained for categories and grouped into levels (1 to 6) in thermally and oxidatively altered coals in the 2020 Round Robin Exercise.

Based on the box-and-whisker diagram of the identified categories grouped into Levels 1 to 6, the highest range is observed in the Level 5 with the highest IQR also documented for the Level 5 (Fig. 17).

7. Conclusion drawn from the evaluation with respect to how participants identified given category in the examined slides:

- Based on the arithmetic mean, participants obtained the highest score (interval of 95-100%) for the following best identified forms: “Pseudovitrinite”, Non-altered and Fluorescent categories as well as other 8 categories. The variation of coefficient was lowest for Coaly Particle and Bitumen.
- The category Non-Altered formed in the Level 2 was best identified as opposed to category Altered.
- The category Bitumen in the Level 3 was best identified as opposed to category Coke.
- The category Darker in colour oxidation rims in the Level 6 was best identified as opposed to category Paler in Colour Particle.

The following statistics refers to evaluation of data with respect to how forms were identified in the examined photomicrographs.

The results of the Grubb’s test shown in Tab. 2 reveal the critical level of 2.97 at the level of significance of 0.05 and number of identified categories being equal to 26. Values, which are lower than 2.97 indicate lack of outliers and extreme values and include the following photomicrographs: 1, 2, 3, 4, 7, 11, 16, 18, 21, 24, 25, 26, 27, 30, 33, 34, 35, 39, 41, 44, 45, 46, and 48.

Table 2. Results of the Grubb's test applied to photomicrographs. Numbers from 1 to 52 define the order of photomicrographs that were identified.

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25
4.489286	3.986115	3.654302	3.510646	**2.942922**	**2.231396**	3.128177	**2.520489**	**2.050944**	**2.558870**	3.030207	**2.744494**	**2.968322**	**2.665955**	**2.648099**	3.356877	**2.745110**	3.036320	**2.894193**	**2.584402**	3.043536	**2.681989**	**2.445942**	3.402920	3.008501
26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50
3.272839	3.308632	**2.705946**	**2.272936**	3.609233	**2.359238**	**2.752752**	3.250096	3.420147	3.131505	**2.338053**	**2.917587**	**2.374118**	3.228467	**2.287217**	3.732324	**2.369401**	**2.562818**	3.462328	4.203749	3.518991	**2.211883**	3.025767	**2.455526**	**2.705074**
51	52																							
2.552775	**2.381612**																							

For the same photomicrographs as displayed in Tab. 2, box-and-whisker diagram in Fig. 18 shows a decidedly higher numbers of outliers and extreme values.

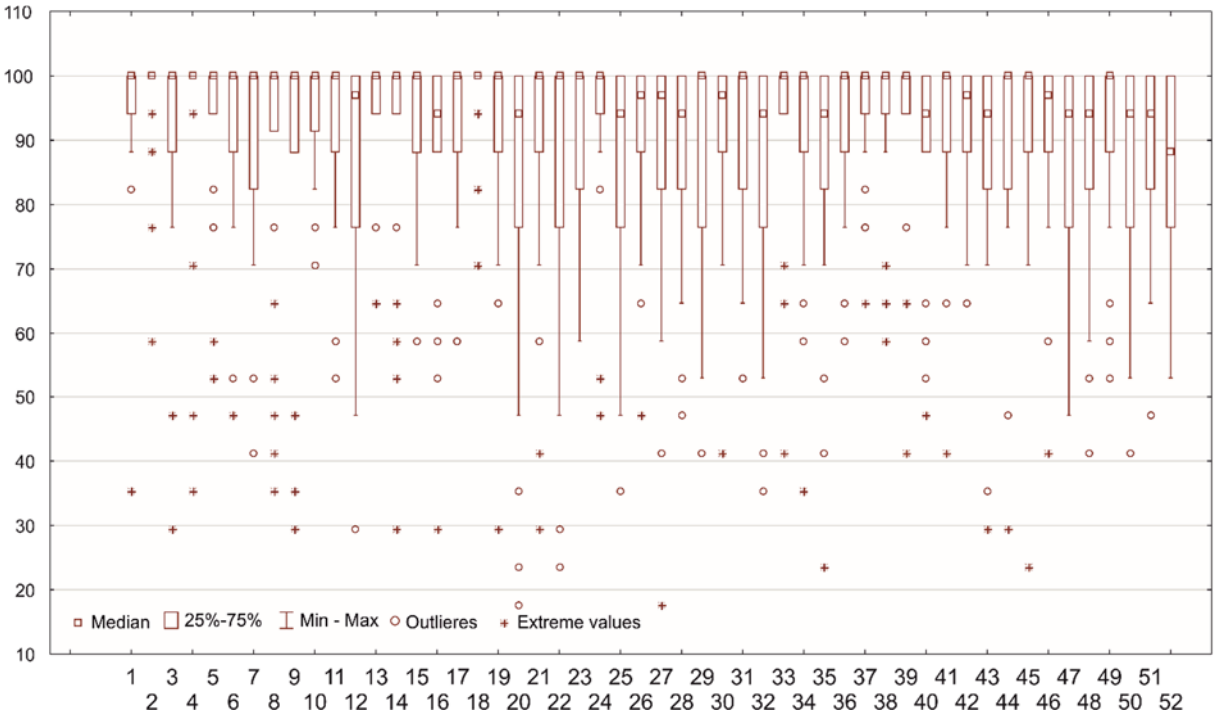


Fig. 18. The box-and-whisker diagram with extreme values, outliers, median and non-outlier range (25%-75%), obtained for photomicrographs (1 to 52) in thermally and oxidatively altered coals in the 2020 Round Robin Exercise.

Based on the arithmetic mean, the best results were recorded for photomicrograph 18 (98.38%) and 2 (98.38%) and the worst for photomicrographs 9 (83.37%), 20 (83.71%), and 29 (85.97%). The statistical mode, or the most common numbers in the data set refers to the value of 100% and occurred in 2 and 4 (numerical account of mode: 21 and 20, respectively). The lowest numerical account obtained for the

photomicrograph 52. The lowest range, i.e., the best recognizability was calculated for photomicrographs 18 and 10 with the range of 29.40 and photomicrographs 42, 37, and 13 with the range of 35.30. The highest range was documented for photomicrographs 20, 27 with a range of 82.40. The variance and standard deviation are not referred to as all data sets display non normal distribution. The highest variation coefficient was obtained for 9, 20, and 22 and the lowest for 18, 2, and 10 photomicrographs.

Based on the box-and-whisker diagram for Levels 1 to 6 displayed in Fig. 19, the highest range is observed in the Levels 5, with the the highest IQR is noted for the Level 6.

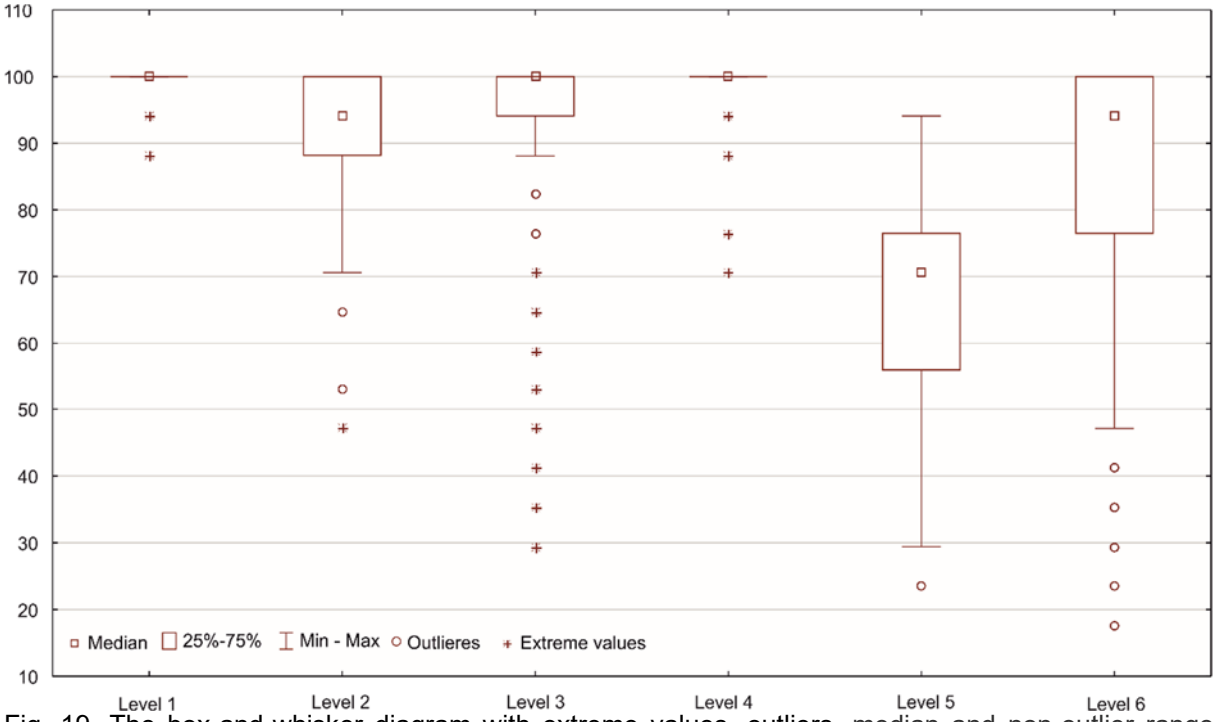


Fig. 19. The box-and-whisker diagram with extreme values, outliers, median and non-outlier range (25%-75%), obtained for 52 photomicrographs grouped into levels (1 to 6) in thermally and oxidatively altered coals in the 2020 Round Robin Exercise.

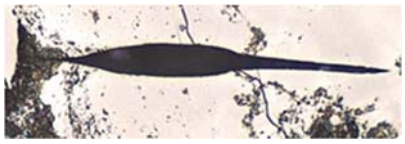
Conclusion drawn from the evaluation with respect to forms as identified in each of the photomicrographs:

- Based on the arithmetic mean, the best results were recorded for photomicrograph 18 (98.38%) and 2 (98.38%) and the worst for photomicrographs 9 (83.37%), 20 (83.71%), and 29 (85.97%).
- However, based on the range, the best recognizable forms were displayed in photomicrographs18 and 10 with the range of 29.40 and photomicrographs 42, 37, and 13 with the range of 35.30 and worst recognizable forms were seen in photomicrographs 20, 27 with a range of 82.40.

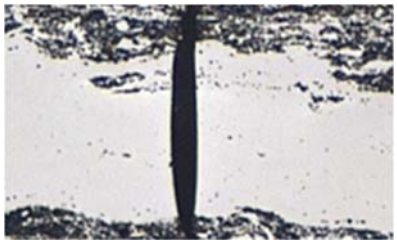
8. Remarks from Participants

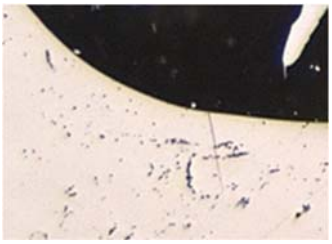
Participants made numerous comments and suggestions, which are displayed as follows:

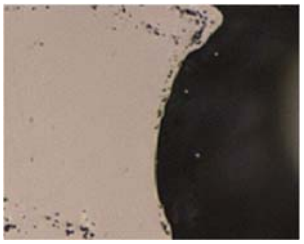
- Porous is any hole in the material so where the rectangle falls over fissures, cracks, or vacuoles it is always porous.
- From all the images there is no data to tell if the material in rectangle is isotropic or anisotropic. For that you would need the Ro values from the material in the rectangle to be on the 2 polarized light images at 90 degrees to each other.
- For the identification of 'paler particle' in level 6 - if the material in rectangle is brighter than 0.94 image in corner or the VRo values given is >0.94% then one can make the assumption it is a paler particle. I don't think I marked all the images for that but it would be most of them.
- I have some doubts about identification of "porous" structure when there are volatilization vacuoles. In addition, I also have some doubts in the identification of paler particles; should be the particles with reflectance above the background?
- Slide 49 - Check slide 95 for the ultraviolet light, and slides 96 and 97 for the polarisation. I am going to assume that it is a non altered particle and that it since it is a cutinite has fluorescence.
- I defined the sample/grain as anisotropic most often on the basis of the given reflectance values. Rarely, anisotropy was evident in 0° and 90° polarised light photographs.
- Sometimes it is difficult to distinguish between pores and cracks/gaps caused by temperature, e.g.: slide 5, 6, 8.



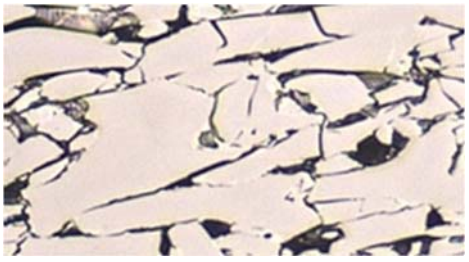
- How to label massive areas on the boundary of a large pore. As massive or as porous. E.g. slide 11, 15 - I have labelled as porous but have doubts.







- In the case of areas involving inertinite, I described them as porous, although the pores there appeared to be primary and not temperature-induced. e.g.: slide 12, 32.
- Slide 2 - Based on the classe I can note that this sporinite is FL. Correct?
- A cut-off value is needed (e.g. vol.%) of porous ???
- I added both crack and dev pore, as I think it cracked and also lost something.
- The first one deals with the picture in the right upper corner of the slides: in chapter 3 of your guidelines it is written that this is 'an altered high-vol. bit. coal' but my opinion is that it shows an unaltered coal. Please advise!
- The second disagreement: You provide Rmax, Rr, Rmin values of the coal under the microscope and not under the red parallelogram. Correct?
- Slide 6 - Very hard to tell if this is isotropic or anisotropic.
- Slide 12 - Few pores may be related to thermal processes; many pores may be an original feature of maceral.
- Slide 25 - Was deciding between pseudomicrinite and coke. Went with pseudomicrinite because I thought from the shape it may have previously been cutinite.
- Slide 9, 45, 46 - Darker oxidation rim or dirty sample / poor polish?
- Slide 49 - ERROR? Slide 10 of the background information has this image labelled as cutinite. It should fluoresce?
- Slide 50 - Not sure whether the pores are devolatilisation pores or not, they appear to be filled with minerals?



5.6. Comments by Convenors

- The conveners proposed to modify the description of **Porous** and **Massive** categories and to provide an additional graphical example to clarify definition of this category.
- The conveners suggested to amend definition of the **Isotropic** vs **Anisotropic** forms, and proposed to add the Rmax/Rmin values to each of grain under examination in each of the slides for the next Round Robin Exercise in support of the identification.
- The definition of the category **Paler in colour particle** will be modified.

Acknowledgment:
The Convenors of the Self-Heating WG would like to thank all Participants for participation in the 2020 IDOM Round Robin Exercise and for providing detailed descriptions of the observed optical characteristics of coals subjected to oxidative and thermal alteration. The author is very grateful Ewa Szram, Faculty of Natural Resources, University of Silesia, Katowice, Poland for great support in statistical evaluations of the data.

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