Report on Organic Matter Concentration Working Group (OMCWG 2010)

Convenor: João Graciano Mendonça Filho (UFRJ-Brazil)

1. Introduction:

The main objective of the Organic Matter Concentration WG was to study the effect of the isolation procedure on organic matter optical parameters. This third exercise of the WG consisted of the analysis of two samples with kerogen type I. The samples studied in this exercise were of low rank and the analyses performed were:

- ¤ Vitrinite reflectance of the whole-rock sample (WR);
- ¤ Vitrinite reflectance of the kerogen concentrate sample (KC);
- ¤ Spectral fluorescence analysis of liptinite in the whole rock sample (WR);
- ¤ Spectral fluorescence analysis of liptinite in the kerogen concentrate sample (KC);

This report includes results obtained by sixteen participants (Table 1) from 9 laboratories. The exercise was proposed in the Gramado Brazil ICCP meeting to continue the activities of the Organic Matter Concentration Working Group that began in 2008.

Table 1: List of Participants in the OMCWG.

Participant	Affiliation	Country
Araujo, Carla V.	Petrobras R&D Center	Brazil
Borrego, Angeles G.	INCAR-CSIC	Spain
Chagas, Renata B. A.	Federal University of Rio de Janeiro	Brazil
Cook, Alan	Keiraville Konsultants Pty. Ltd	Australia
Flores, Deolinda	University of Porto	Portugal
Hackley, Paul	U.S. Geological Survey	USA
Hower, Jim	University of Kentucky	USA
Kern, Marcio L.	Federal University of Rio de Janeiro	Brazil
Kus, Jolanta	Federal Institute for Geosciences and Natural Resources	Germany
Mastalerz, Maria	Indiana University	USA
Mendonça Filho, João G.	Federal University of Rio de Janeiro	Brazil
Mendonça, Joalice O.	Federal University of Rio de Janeiro	Brazil
Menezes, Taíssa R.	Petrobras R&D Center	Brazil
Ranasinghe, Paddy	Keiraville Konsultants Pty. Ltd	Australia
Souza, Igor V. A. F.	Petrobras R&D Center	Brazil
Suarez-Ruiz, Isabel	INCAR-CSIC	Spain

Previous exercises focussed on the study of samples containing terrestrial organic matter (Mendonça Filho *et al.*, 2008) and mainly marine organic matter (Mendonça Filho *et al.*, 2009) of different rank. The results of the OMCWG 2008 and 2009 are published in a paper titled: *Effect of concentration of dispersed organic matter on optical maturity parameters: Interlaboratory results of the organic matter concentration working group of the ICCP*: International Journal of Coal Geology (doi: 10.1016/j.coal.2010.07.005).

For the OMCWG 2010, the samples studied were composed of two outcrop carbonaceous lacustrine shales: one from USA (sample OMC5), Green River Formation (low rank, Type I-kerogen), Eocene age, Uinta Basin (Mahogany Ledge) and the other from Brazil (sample OMC6), Tremembé Formation (low rank, Type I kerogen), Oligocene age, Taubaté Basin.

The set of samples comprises 4 in total, numbered as follows:

Sample OMC5 (Green River Formation, Mahogany Ledge, USA): OMC5A = whole rock and OMC5B = kerogen concentrate

- ➢ Total Organic Carbon (TOC) is 6.44 wt.%
- ➢ The Hydrogen Index (HI) is 781 mg HC/g TOC and the Oxygen Index (OI) is 11 mg CO2/g TOC, indicating Type I kerogen (Figure 1);
- ➢ The T_{max} value (436°C) indicates that this sample is immature (low rank);
- ➢ The hydrocarbon source potential is very high (S2 = 54.96 mg HC/ g Rock) indicating excellent quality of organic matter for hydrocarbon generation.

Sample OMC6 (Tremembé Formation, Taubaté Basin, Brazil): OMC6A = whole rock and OMC6B = kerogen concentrate

- ➢ Total Organic Carbon (TOC) is 12.05 wt.%;
- ➢ The HI is 707 mg HC/g TOC and the OI is 12 mg CO2/g TOC, indicating Type I kerogen (Figure 1);

- The T_{max} value (436°C) indicates that this sample is immature (low rank);
- The hydrocarbon source potential is excellent (S2 = 94.75 mg HC/ g Rock) indicating excellent quality of organic matter for hydrocarbon generation.

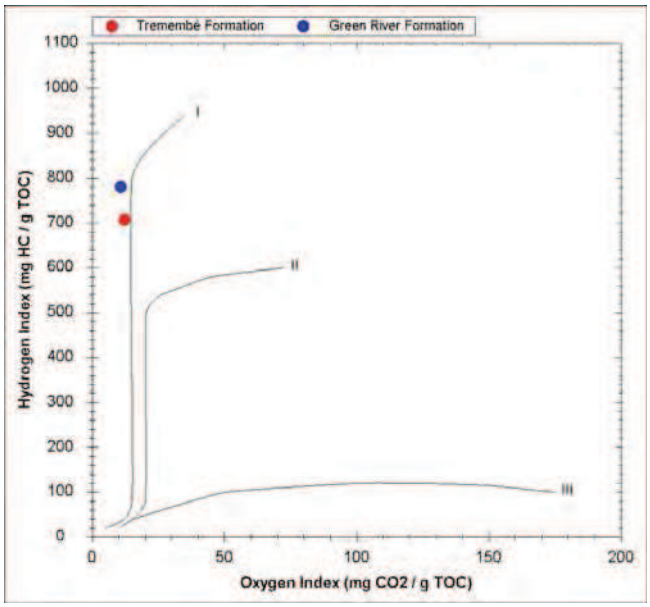


Figure 1 - Pseudo Van Krevelen plot (Espitalié et al., 1977) showing hydrogen and oxygen indices of samples from Green River and Tremembé formations.

2. Sample Preparation:

2.1. Whole-Rock Preparation Procedure:

Studied samples were ground to approximately 2 mm size and embedded in resin. A single block was prepared for each sample.

2.2. Kerogen Concentration Procedure:

Samples were ground to approximately 2 mm size. HCl (37%) was added to the sample for 18 hrs. Following HCl the sample was washed with distilled water until the effluent was neutral. In the next step HF (40%) was added for 24 hrs, followed by a neutral wash and addition of 37% HCl for 3 hrs to remove fluorides and a final neutral wash. Samples were floated using $ZnCl_2$ (ρ = 1.9 to 2 g/cm^3) and centrifuged to separate sulphides. Following centrifugation samples were washed with HCl (10%) plus distilled water to eliminate remaining heavy liquid and air-dried. The air-dried isolated kerogen was sieved (20 m) and embedded in resin (SERIFIX-STRUERS).

2.3. Sample Polishing:

Particulate blocks were ground using progressively finer grades of wet silicon carbide papers; including 800, 1200 and 4000 grit wet silicon carbide paper. A single set of samples was sent to each participating laboratory.

3. Statistical Evaluation Criteria and Parameters

Precision and bias assessment for the analysts: an evaluation of the suitability of the data for an accreditation program (based on Borrego et al. 2006) was used to interpret the reported data. This evaluation is based on the ICCP Accreditation Program for Vitrinite Reflectance Measurements on Dispersed Organic Matter described at **http://www.iccop.org/index.php?id=29**. The parameters considered in the accreditation program are:

UMSD: refers to participant's **U**nsigned **M**ultiple of the **S**tandard **D**eviation, calculated against the group mean and standard deviation data, for each sample analyzed as per the formula below:

$$UMSD = \left| \left(\frac{X_i - \overline{X}}{\sigma} \right) \right|$$

Xi = the participant vitrinite reflectance
X = the group mean vitrinite reflectance
σ = the standard deviation of the group

SMSD: refers to participant's **S**igned **M**ultiple of the **S**tandard **D**eviation, calculated against the group mean and standard deviation data, for each sample analyzed.

AUMSD and **ASMSD** are the average UMSD and SMSD values respectively for each participant. The AUMSD value is a measure of the participant's **accuracy** and the ASMSD is an indicator of the participant's **measurement bias** in the techniques being assessed. A threshold of 1.5 separates acceptable values from those departing too much from the group mean.

4. Results and Discussion:

The participants are identified by alphabetic letters (from A to P) in this report. Sixteen participants provided results based on standard vitrinite reflectance, and nine participants provided results based on spectral fluorescence analysis of liptinite macerals.

Table 2 shows the distribution of vitrinite

reflectance for the different samples as reported by the participants. In the exercise instructions participants were asked to provide "the maximum of measurements per sample". Despite this instruction around 70% of participants for sample OMC5 and 50% of participants for sample OMC6 provided fewer than 15 values (Table 2). Figure 2 shows that it was easier for participants to find appropriate particles to measure in samples from Tremembé Fm (OMC6A and OMC6B) than in Green River Fm. (OMC5A and OMC5B) and that it was easier to find particles in the whole rock than in the kerogen concentrate. This is reflected in the fact that participants generally reported similar or higher number of readings in the whole rock than in the kerogen concentrate. Although the number of readings reported is below the desirable amount for a sound statistical evaluation (e.g., Barker and Pawlewicz, 1993) they are considered sufficient for the study of the effect of the isolation procedure on the organic matter optical parameters in Type I-kerogen. Furthermore, it was observed a large variation in the number of readings by each participant in both samples, which indicate certain difficulties in the identification/selection of the vitrinite population.

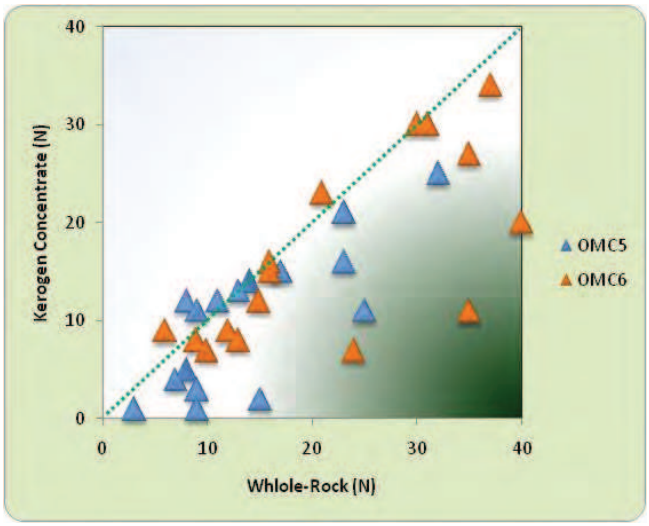


Figure 2: Comparison of number of readings for the WR and KC.

The average vitrinite reflectance value for whole-rock sample OMC5 was 0.38% and for kerogen concentrate was 0.37%. For sample OMC6, the result was 0.30% for whole-rock and 0.28% for kerogen concentrate (Plate 1). In general, the Standard Deviation (SD) values in both samples were low. It is remarkable that some participants reported SD below 0.03 even with reasonably high number of readings (Table 2).

Table 2: Distribution of vitrinite reflectance as reported by the participants.

Participant	OMC5A R_r (%)			OMC5B R_r (%)			OMC6A R_r (%)			OMC6B R_r (%)		
	R_r (%)	SD	N	R_r (%)	SD	N	R_r (%)	SD	N	R_r (%)	SD	N
A	0.43	0.09	8	0.42	0.07	5	0.24	0.03	24	0.24	0.02	7
B	0.28	0.07	14	0.30	0.08	14	0.22	0.04	16	0.20	0.03	16
C	0.40	0.03	15	0.39	0.03	2	0.38	0.01	35	0.37	0.01	11
D	0.39	0.09	8	0.39	0.04	12	0.26	0.03	21	0.25	0.03	23
E	0.27	0.06	23	0.29	0.06	16	0.29	0.05	31	0.26	0.05	30
F	0.38	0.07	7	0.35	0.06	4	0.27	0.04	12	0.23	0.02	9
G	0.35	0.06	3	0.30	0.00	1	0.27	0.03	9	0.27	0.01	8
H	0.55	0.07	9	0.55	0.12	3	0.23	0.04	6	0.22	0.01	9
I	0.54	0.05	23	0.48	0.03	21	0.47	0.02	40	0.46	0.01	20
J	0.39	0.01	13	0.36	0.02	13	0.36	0.01	16	0.36	0.01	15
K	0.30	0.08	11	0.34	0.07	12	0.26	0.05	35	0.22	0.04	27
L	0.29	0.06	9	0.26	0.00	1	0.21	0.04	10	0.20	0.04	7
M	0.31	0.05	25	0.32	0.07	11	0.27	0.06	30	0.27	0.07	30
N	0.34	0.11	32	0.29	0.08	25	0.28	0.07	37	0.26	0.08	34
O	0.45	0.10	9	0.41	0.10	11	0.40	0.02	15	0.32	0.04	12
P	0.39	0.01	17	0.39	0.01	15	0.34	0.01	13	0.37	0.01	8
Average	0.38			0.37			0.30			0.28		
SD	0.08			0.08			0.07			0.07		

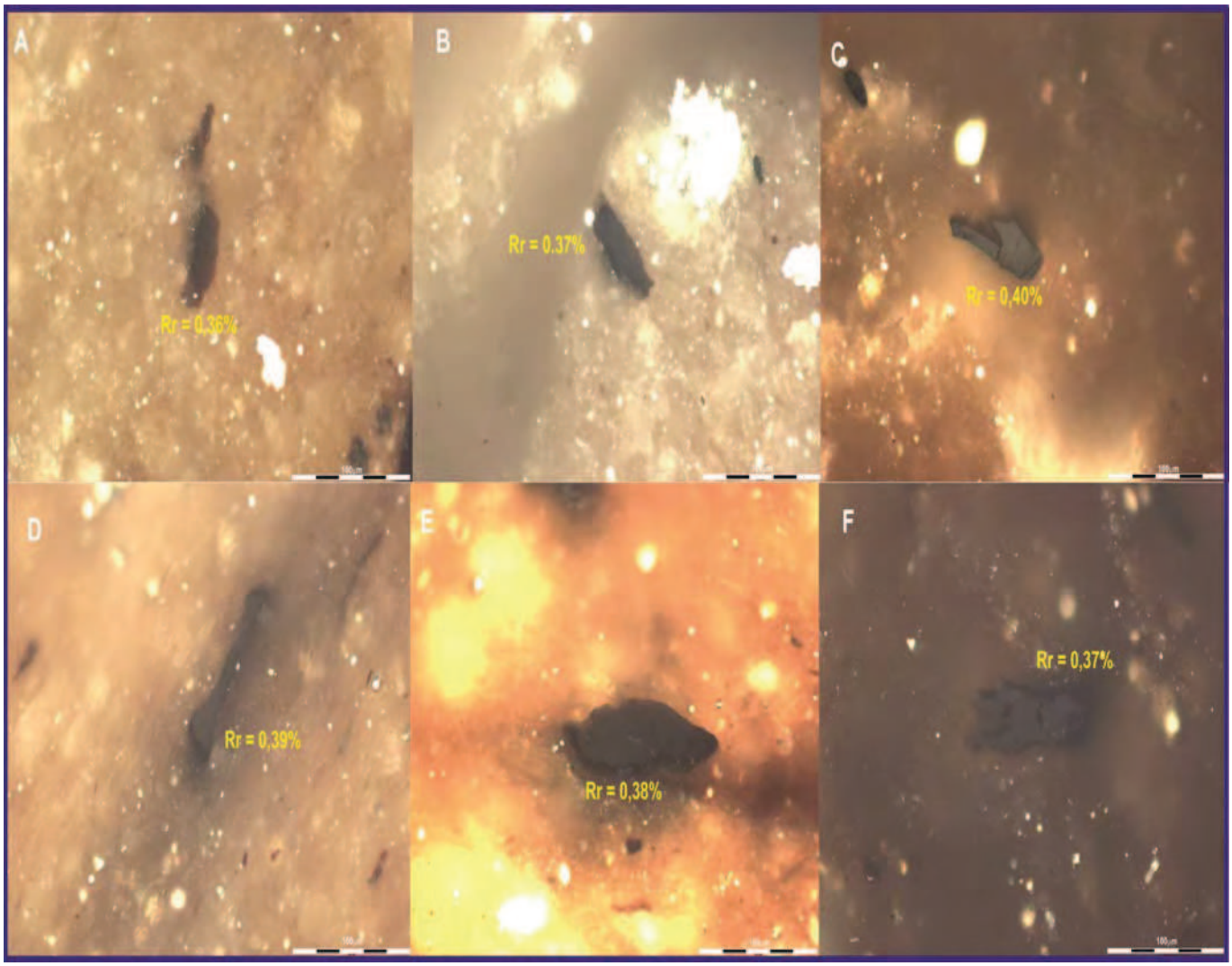


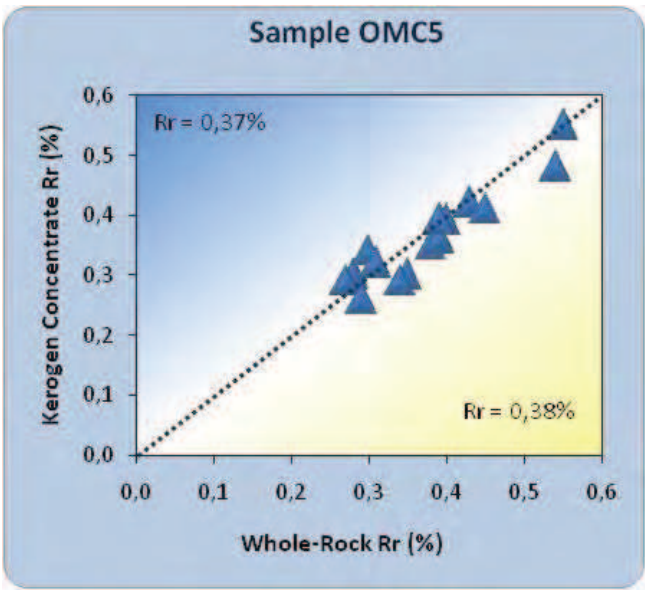
Plate 1: Examples of vitrinites. A-B) Sample OMC5A; C) Sample OMC5B; D-E) Sample OMC6A; F) Sample OMC6B. All photomicrographs were taken under white incident light, oil immersion.

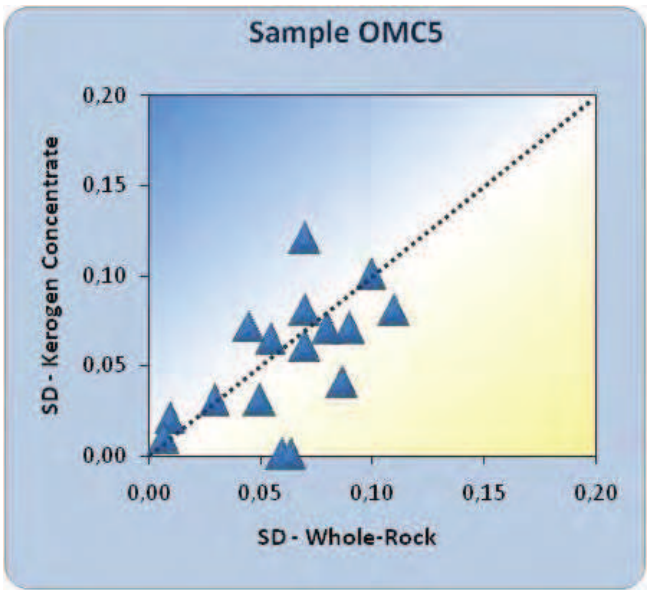
The graph of kerogen vs whole rock vitrinite reflectance values (Figure 3) allows comparison of results from the whole-rock and the kerogen concentrate samples. If the x and y axes have the same dimensions and the results were equivalent, all the points should be on the linear regression or closer. In both samples the reflectance tended to be slightly higher in the whole-rock, where it can be observed clearly that most of the points are slightly below the linear regression.

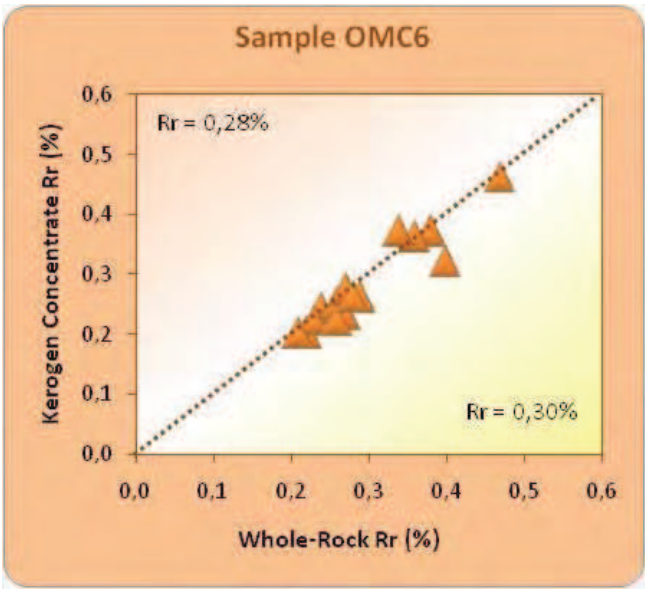
Standard Deviations in reflectance values between WR and KC are shown in Figure 4. If SD values were always higher in one than in the other, this would indicate a greater difficulty to identify the population of measurable particles. The graphs show larger SD in sample OMC5 than in sample OMC6 and also larger differences in SD between the WR and the KC for OMC5. In sample OMC5 the SDs scattered quite randomly around the linear regression, except for a couple of values (participants G and L who only reported one reading having SD=0). In sample OMC6 the SDs tended to be slightly higher in the WR than in the KC.

In general, low values of SD were observed in the two analyzed samples; however results from sample OMC5 contained a larger scatter of the readings, which could indicate more difficulty to identify the vitrinite particles in sample OMC5 than in sample OMC6.

The scatter of results also is observed in Figures 5 and 6 where mean reflectance reported by each participant is plotted with error bars corresponding to the individual standard deviation (SD). The scatter of the results is reasonable and most of the values are within the 1.5SD threshold of the ICCP Accreditation criteria.







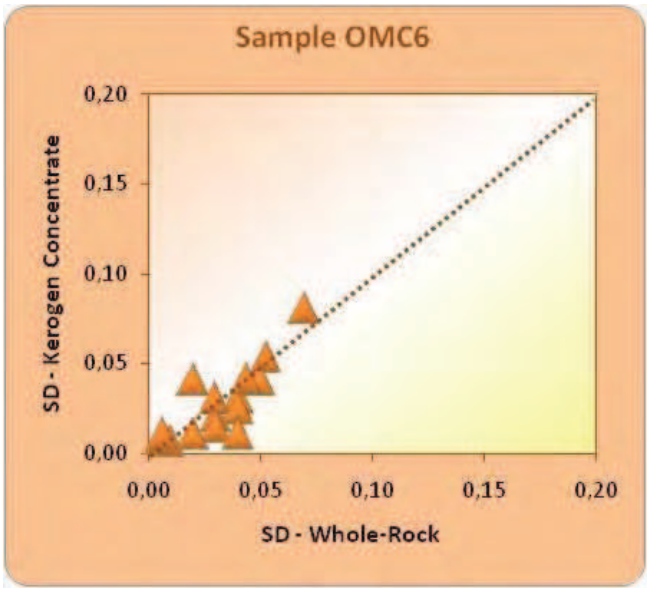


Figure 3: Comparison of mean reflectance values between WR and KC.

Figure 4: Comparison of standard deviation (SD) between WR and KC. (Scatter of data in the analyzed samples)

The average reflectance considering all data was 0.38% for sample OMC5A (Whole-Rock) and 0.37% for sample OMC5B (Kerogen Concentrate). Participant H reported values higher than 1.5SD of the group mean in both samples and Participant I reported values higher than 1.5SD of the group mean in sample OMC5A (WR) (Figure 5). Some participants noted the presence of two vitrinite populations in sample OMC5, the higher of which the average reflectance was around 0.60% (Figure 6). However, one participant reported the presence of a low reflecting inertinite population (0.45%) in the same sample suggesting those particles could just as well be interpreted as vitrinite (Figure 7).

The high reflectance values measured by participants H and I with low SD could indicate that they refused to measure the low reflecting population measured by most of the participants. Participant H reported slight orange fluorescence in the low reflecting population indicating a perhydrous character. This could have been the reason for refusing the measurement of the low reflecting population with mean values around 0.35% (Figure 8).

The group mean considering all the data was 0.30% for sample OMC6A (Whole-Rock) and 0.28% for sample OMC6B (Kerogen Concentrate) (Figure 9). Participant I reported values higher than the group mean plus 1.5SD in both samples.

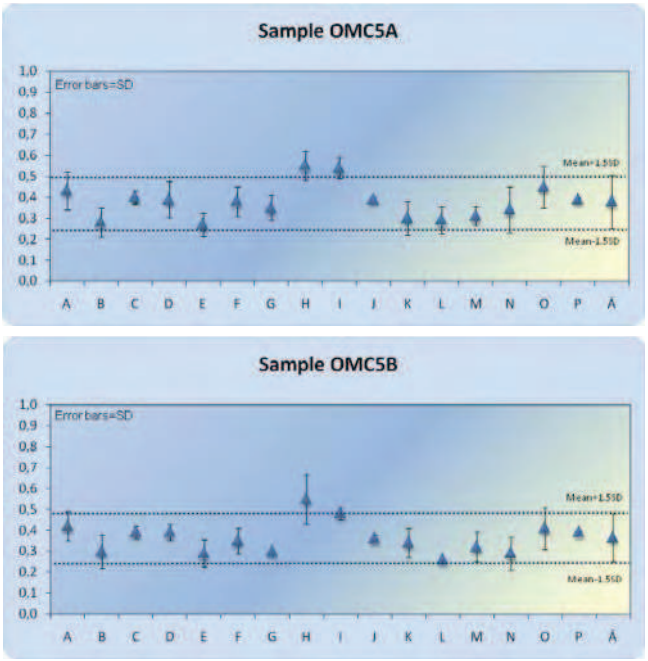


Figure 5: Average Rr (%) values for samples OMC5A (Whole-Rock) and OMC5B (Kerogen Concentrate).

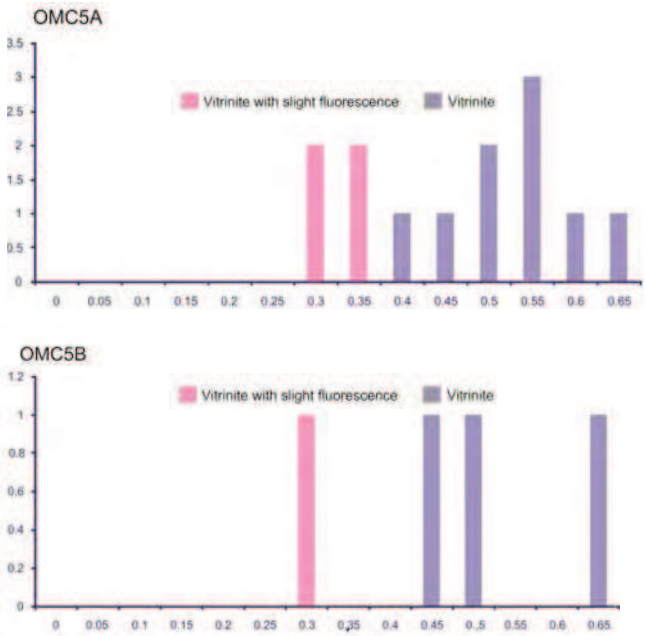


Figure 8: Histograms showing a low reflecting vitrinite population in samples OMC5A (WR) and OMC5B (KC) measured by the participant H.

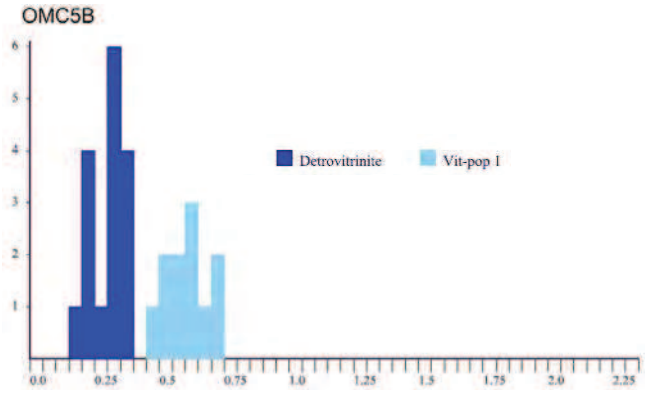


Figure 6: Histogram showing a high reflecting vitrinite population in sample OMC5B (KC).

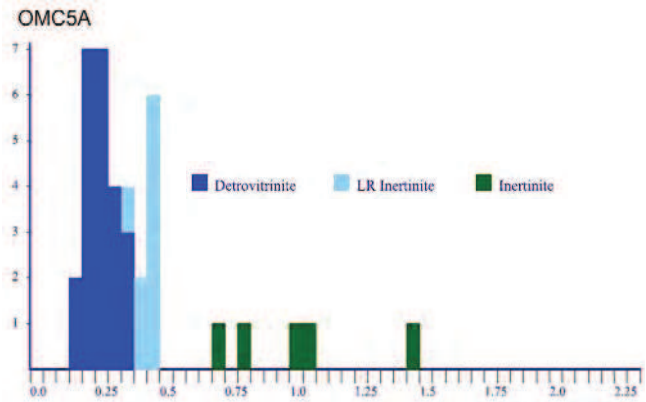


Figure 7: Histogram showing a low reflecting inertinite population in sample OMC5A (WR).

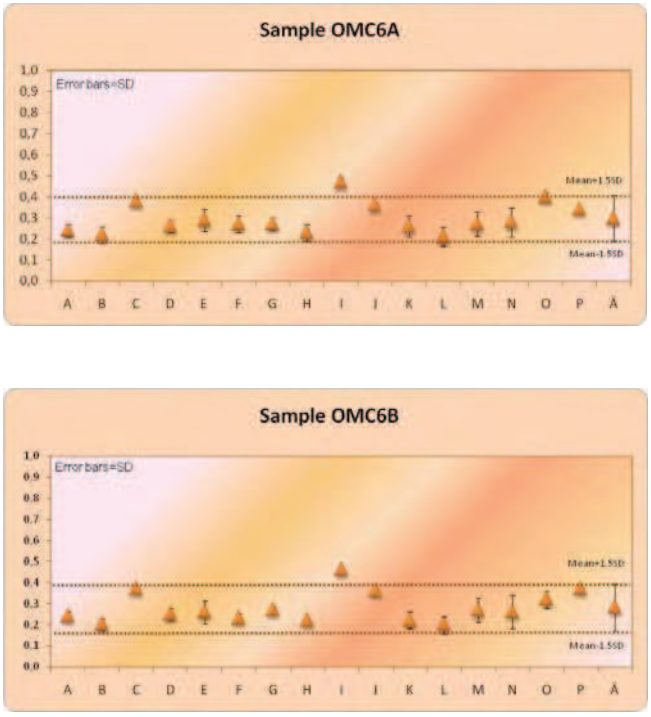
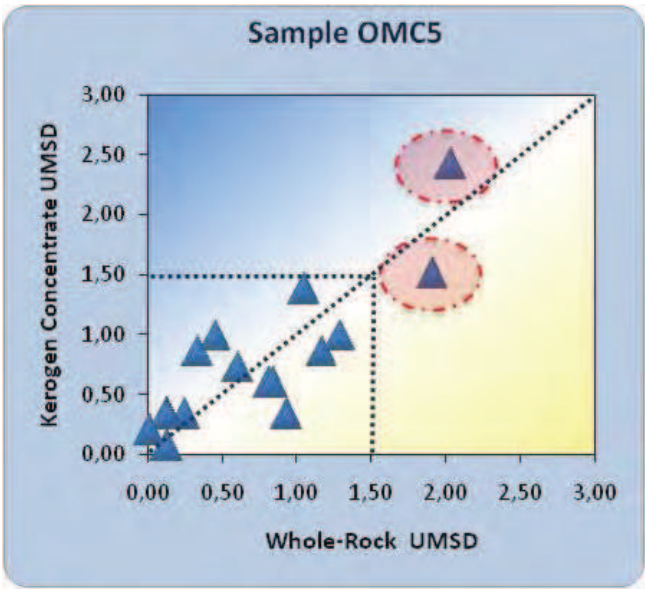


Figure 9: Average R_r (%) values for samples OMC6A (Whole-Rock) and OMC6B (Kerogen Concentrate).



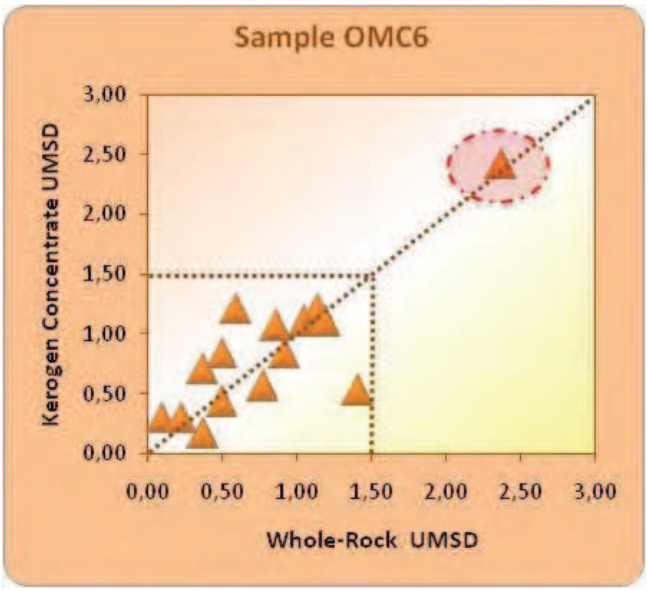


Figure 10: UMSD (Unsigned Multiple of the Standard Deviation)[1].

In Figure 10 it can be observed that in general the UMSD of participants were evenly dispersed around the linear regression in samples OMC5 and OMC6. In sample OMC5 two analysts (Participants H and I) presented results outside of the threshold considered acceptable for precision (±1.5SD of the group mean) in both the KC and the WR samples. In sample OMC6, only Participant I presented results departing more than 1.5SD of the group mean.

Using the criteria and parameters applied for the DOMVR ICCP accreditation program, www.iccop.org, (Table 4), good results were obtained in this exercise (Table 5). Nevertheless this assessment does not address the problem of selecting different vitrinite populations. Two participants had an AUMSD value over 1.5 and one participant had a very high ASMSD value, probably related to problems with vitrinite identification. The majority of participants presented consistent results and their analytical techniques were acceptable.

Table 4: Coal Reflectance Analysis Criteria (ICCP).

Parameters	Precision and bias for the analysts	
ASMSD	< ± 0.5	Low
	± 0.5 < ± 1.0	Medium
	± 1.0 < ± 1.5	High
	> ± 1.5	Very High
AUMSD	< 1.5	Your analytical technique is acceptable
	> 1.5	Your analytical technique is not acceptable

Table 5: Accuracy of results calculated against the group mean and standard deviation data, for each sample analyzed: SMSD (Signed Multiple of the Standard Deviation), AUMSD and ASMSD.

Participant	SMSD	AUMSD	ASMSD	Remarks (bias)
A	-0.32	0.68	-0.08	Low
B	-4.56	1.14	-1.14	High
C	2.64	0.66	0.66	Medium
D	-0.79	0.35	-0.20	Low
E	-3.02	0.76	-0.76	Medium
F	-1.58	0.40	-0.40	Low
G	-2.07	0.52	-0.52	Medium
H	2.46	1.58	0.62	Medium
I	8.02	2.00	2.00	Very High
J	1.70	0.51	0.43	Low
K	-2.95	0.74	-0.74	Medium
L	-4.71	1.20	-1.20	High
M	-2.28	0.57	-0.57	Medium
N	-2.32	0.58	-0.58	Medium
O	3.11	0.78	0.78	Medium
P	1.96	0.49	0.49	Low

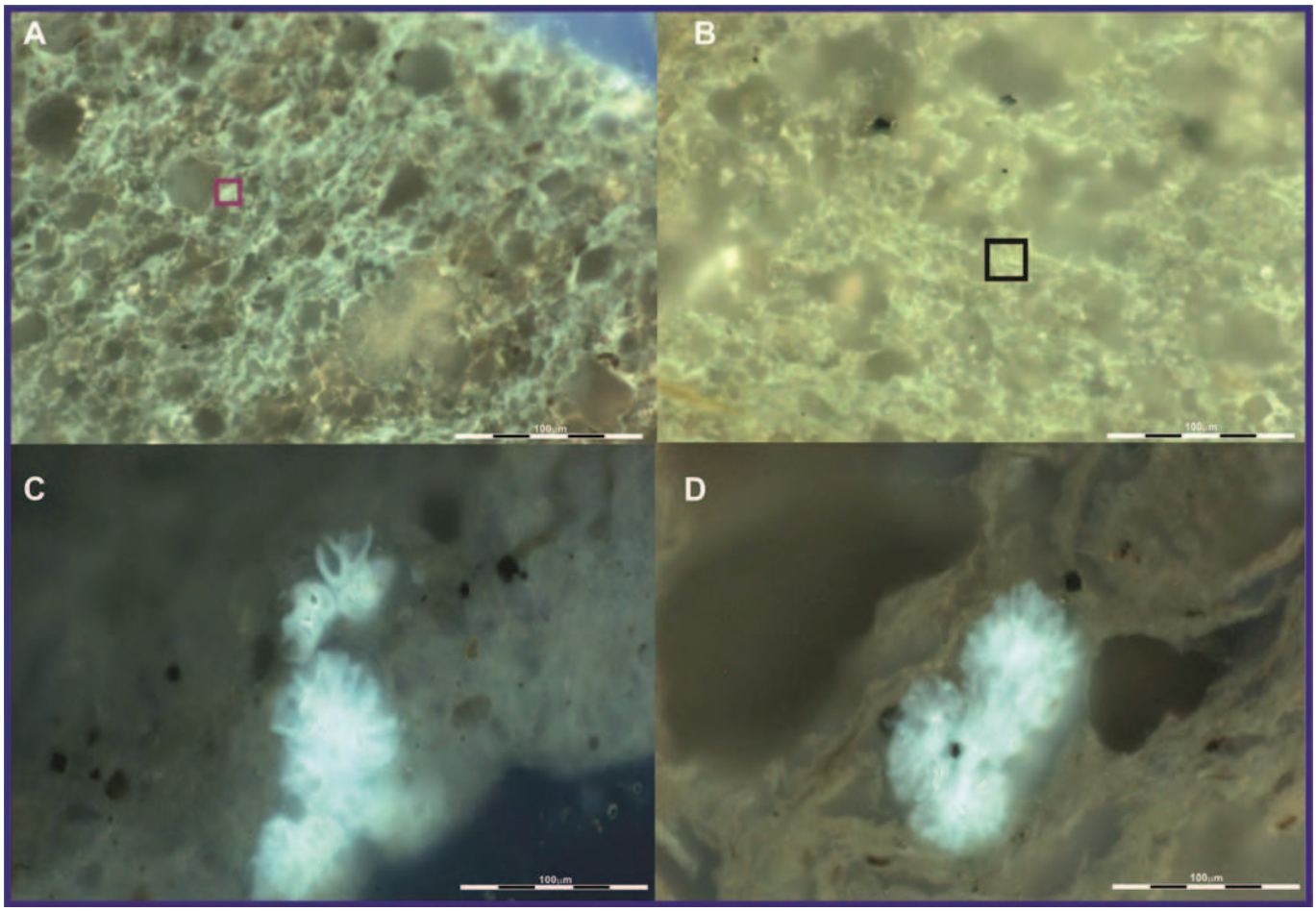
[1]calculated against the group mean and standard deviation data

5. Spectral fluorescence analysis:

Nine participants provided results on spectral fluorescence measurements. Nine participants gave spectral curves for Unstructured Fluorescing Organic Matter (AOM, Plate 2, Photos A and B) of the Green River Fm. (OMC5). For sample OMC6 (Tremembé Fm.) seven participants provided results for telalginite (Botryococcus algae, Plate 2, Photos C and D) and three participants provided results for lamalginite. This will not allow comparison of the fluorescence properties of a single component in both samples but does allow investigation of the effect of kerogen isolation on fluorescence parameters. In general, the λ_{max} results for sample OMC5 confirm the low rank of Green River Fm. (Table 6 and Figure 11). There was no pattern in the spectral fluorescence results for the samples OMC5A (WR) and OMC5B (KC) (Table 6, Figure 12). In general, the results of vitrinite reflectance equivalent calculated from the spectral fluorescence, based on Mukhopadhyay (1994), are higher than the values from measured vitrinite reflectance (Table 7).

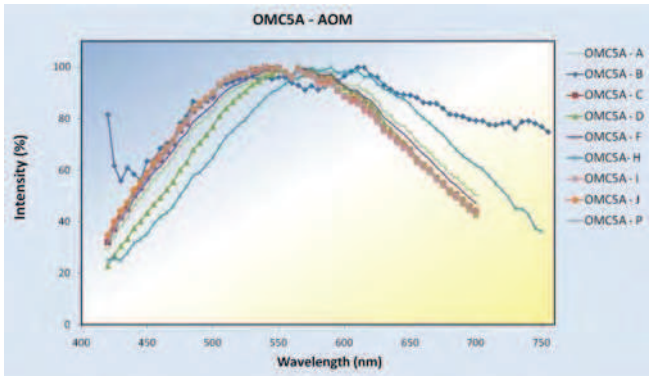
Table 6: Spectral fluorescence parameters obtained for Green River Fm. (OMC5).

Participant	OMC5A (AOM) λ (nm)	OMC5B (AOM) λ (nm)
A	565	565
B	610	530
C	540	564
D	565	565
F	565	565
H	590	575
I	540	565
J	540	550
P	540	540



Plates 2: A) Sample OMC5A: Unstructured fluorescing organic matter (AOM); B) Sample OMC5B: Unstructured fluorescing organic matter (AOM); C) Sample OMC6A: Telalginite (*Botryococcus* algae); D) Sample OMC6B: Telalginite (*Botryococcus* algae) and Lamalginite. All photomicrographs were taken under fluorescence mode.

In the case of sample OMC5A (WR), these results suggest that unstructured fluorescing organic particles (AOM) are not appropriate for this analysis, probably owing to that fluorescing groundmass can be derived from degraded algal material and/or bacterial biomass and commonly appear intimately intermixed with mineral groundmass (Plate 2). Other option is that vitrinite reflectance is suppressed as commonly reported in organic-rich rocks. Alternatively, the results could indicate the wrong selection of the vitrinite population measured because estimated vitrinite reflectance values are closer to those of the high reflecting population selected by some participants, and refused for measurement by others (Table 2). For sample OMC5B (KC) besides the differing origin of organic particles, results could have been affected by the kerogen isolation procedure as reported in Mendonça Filho et al. (2009, 2010).



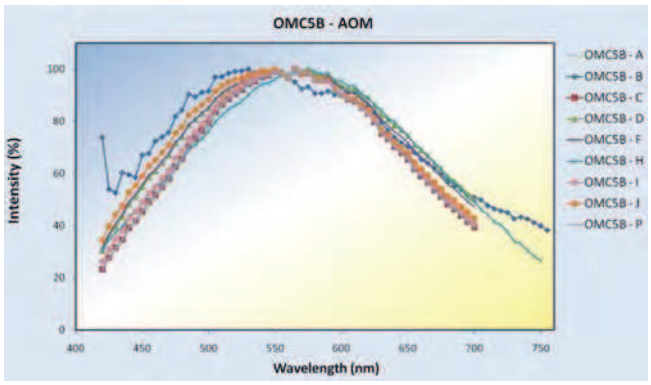


Figure 11: Spectral curves for AOM of samples OMC5A and OMC5B.

Table 7: Correlation between SF and Rr% (Equivalent and Measured) parameters for Green River Fm. (OMC5).

OMC5A (AOM) λ (nm)	Equivalent R_r	Measured R_r	OMC5B (AOM) λ (nm)	Equivalent R_r	Measured R_r
565	0.57		565	0.57	
610	0.88		530	0.43	
540	0.50		565	0.57	
565	0.57	Mean = 0.38	565	0.57	Mean = 0.37
565	0.57		565	0.57	
590	0.70	SD = 0.08	575	0.61	SD = 0.08
540	0.50		565	0.57	
540	0.50		550	0.53	
540	0.50		540	0.50	
Mean	**0.59**		**Mean**	**0.55**	
SD	**0.13**		**SD**	**0.05**	

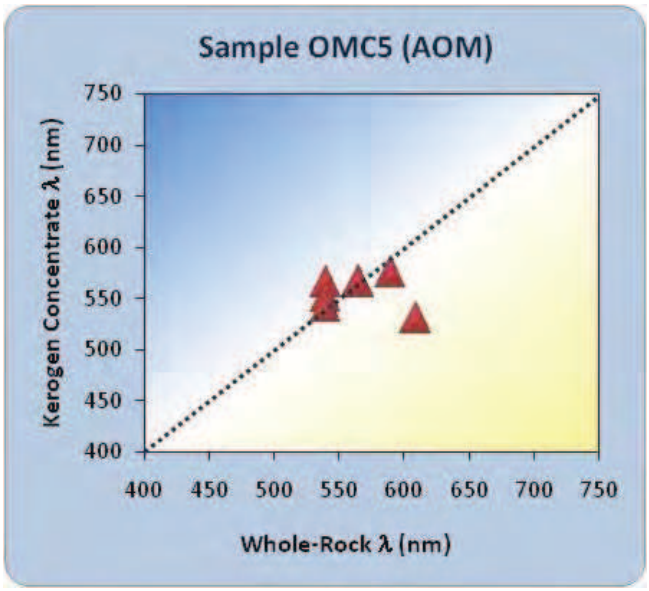


Figure 12: Comparison of λ_{max} values between OMC5A (WR) and OMC5B (KC).

For Tremembé Fm. sample OMC6 the results are represented in Table 8. Plate 2 shows examples of telalginite (*Botryococcus* algae).

The λ_{max} results for sample OMC6 confirm the low rank of Tremembé Fm. Some participants provided results from telalginite (Figure 13) and lamalginite (Figure 15). In general, a shift of max to higher values for sample OMC6B was observed. The difference in values is more apparent when the fluorescence spectra are measured in the lamalginite component (Table 8). These results suggest that the preparation procedures affect fluorescence properties (Table 8) as observed by Mendonça Filho *et al.* (2009, 2010).

Table 8: Spectral fluorescence parameters obtained for Tremembé Fm. (OMC6).

Participant	OMC6A *Telalginite Botryococcus* λ (nm)	OMC6A *Lamalginite* λ (nm)	OMC6B *Telalginite Botryococcus* λ (nm)	OMC6B *Lamalginite* λ (nm)
A	510		515	
B		505		610
C	530		530	
D	505		520	
F	510		520	
H	515	515	510	575
I		520		565
J	510		530	
P	530		530	

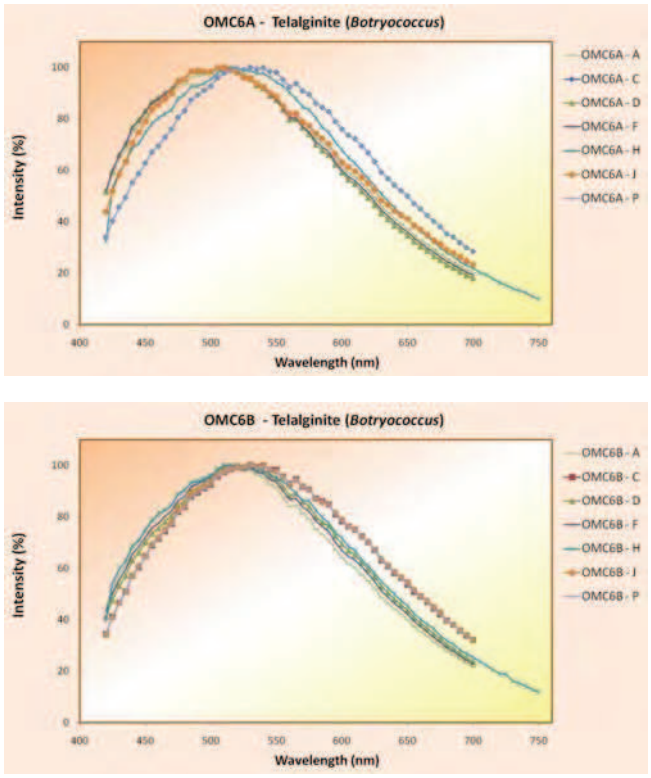


Figure 13: Spectral curves for telalginite of samples OMC6A and OMC6B.

For the single participant measuring both lamalginite and telalginite (participant H) the spectra of lamalginite had a similar λ_{max} value to that of telalginite (Figures 13 and 15 and Table 8). In addition those participants reporting only spectra on lamalginite (participants B and I) measured spectra similar to that of participant H and within the range of the spectra obtained for telalginite by the rest of the participants. Accordingly, the estimated vitrinite reflectance using both telalginite and lamalginite for the OMC6A sample was similar (Tables 9 and 10). In the case of OMC6B a different situation is observed. The telalginite spectra were slightly shifted to higher wavelengths compared to OMC6A telalginite, whereas the lamalginite spectra were strongly shifted to the red compared to OMC6A lamalginite spectra

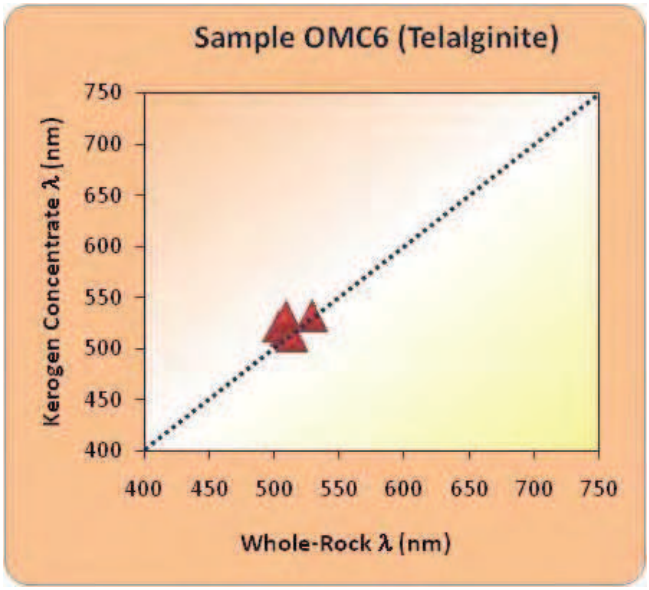


Figure 14: Comparison of λ_{max} values between OMC6A (WR) and OMC6B (KC) samples using Telalginite as object of measured

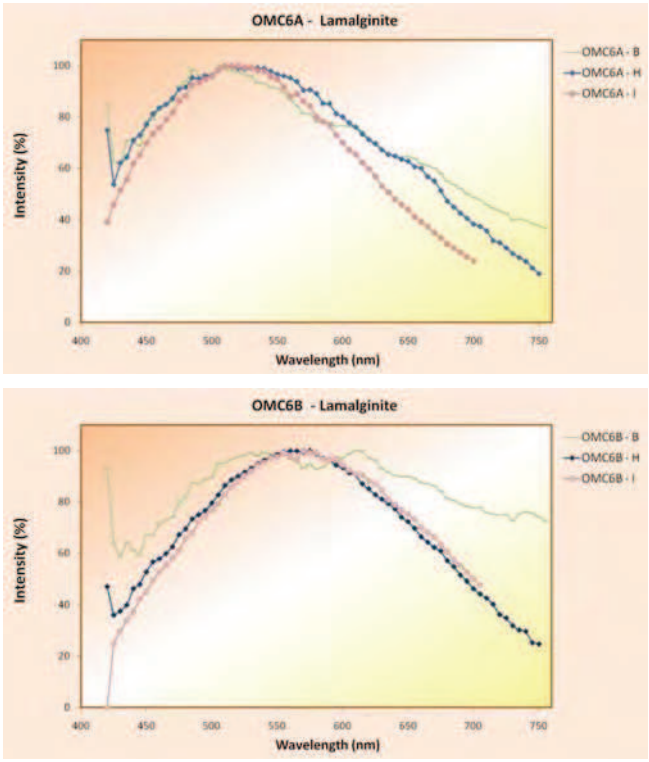


Figure 15: Spectral curves for lamalginite of samples OMC6A and OMC6B.

Figures 14 and 16 show the comparison of λ_{max} values between OMC6A (WR) and OMC6B (KC)

using telalginite and lamalginite as the object of measurement, respectively. For telalginite (Figure 14), all of the points are on the median, indicating that λ_{max} values were practically the same in the KC and WR. For lamalginite (Figure 16), all points are above the median, indicating that λ_{max} values were higher in the KC than in WR.

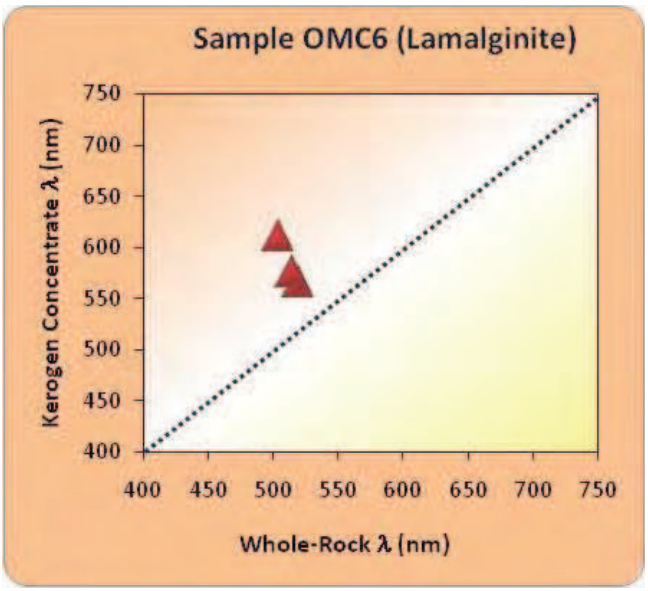


Figure 16: Comparison of λ_{max} values between OMC6A (WR) and OMC6B (KC) samples using lamalginite as the object of measurement.

Figure 17 indicates that both telalginite and lamalginite in sample OMC6 were affected by the isolation procedure, although telalginite showed only a slight red shift in the spectra and a slight increase in estimated vitrinite reflectance, whereas the effect was much stronger in lamalginite. If the measured data for vitrinite reflectance are compared to those calculated from λ_{max} of the fluorescence spectra the following observations are derived:

- The equivalent vitrinite reflectance for sample OMC6, using telalginite as the object of measurement (Table 9), presents a reasonable correlation with the average measured vitrinite reflectance, although the calculated values tended to be slightly higher (Figure 17);
- For sample OMC6B the misfit between equivalent vitrinite reflectance and average measured vitrinite reflectance was higher and calculated values were higher than measured ones (Figure 17), especially when lamalginite was used to obtain the spectra;
- For samples from Green River Fm. a similar result was obtained. Calculated values were higher than measured values for each participant. This can be seen in Figure 18. The effect would have been even higher if reflectance values of all participants could have been considered because those participants measuring spectra reported the highest values for vitrinite reflectance in Table 2.

Table 9: Correlation between SF and R_r% (Equivalent and Measured) parameters for Tremembé Fm. (OMC6) (Telalginite).

OMC6A Telalginite λ(nm)	Equiv-alent R_r	Meas-ured R_r	OMC6B Telalginite λ(nm)	Equiv-alent R_r	Meas-ured R_r
510	0.33		515	0.35	
530	0.43		530	0.43	
505	0.31		520	0.38	
510	0.33	Mean = 0.38	520	0.38	Mean = 0.37
515	0.35		510	0.33	
510	0.33	SD = 0.08	530	0.43	SD = 0.08
530	0.43		530	0.43	
Mean	**0.36**		**Mean**	**0.39**	
SD	**0.05**		**SD**	**0.05**	

Table 10: Correlation between SF and Rr% (Equivalent and Measured) parameters for Tremembé Fm. (OMC6) (Lamalginite).

OMC6A Lamalginite λ(nm)	Equi v-alent R_r	Meas-ured R_r	OMC6B Lamalginite λ(nm)	Equi v-alent R_r	Meas-ured R_r
505	0.31	Mean = 0.30	610	0.88	Mean = 0.28
515	0.35	SD = 0.07	575	0.61	SD = 0.07
520	0.38		565	0.57	
Mean	**0.35**		**Mean**	**0.69**	
SD	**0.04**		**SD**	**0.17**	

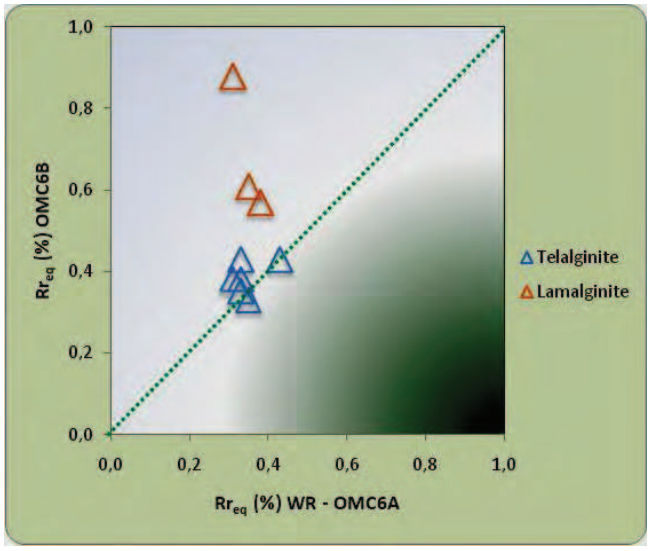
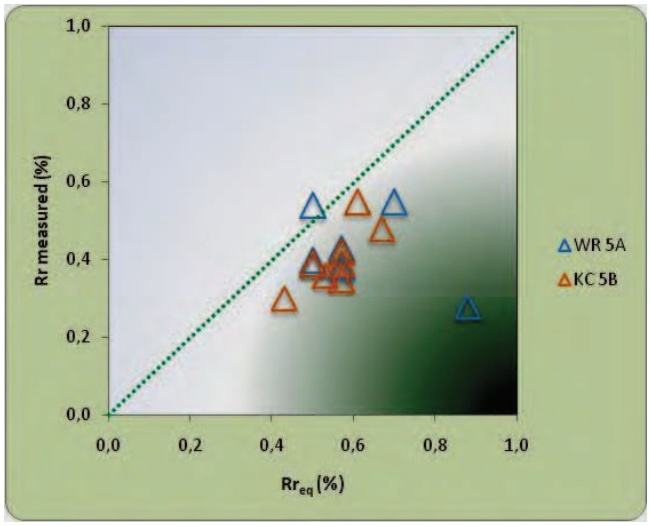


Figure 17: Comparison of equivalent R_r estimated from telalginite and lamalginite for both the whole rock and the kerogen concentrate in sample OMC6.



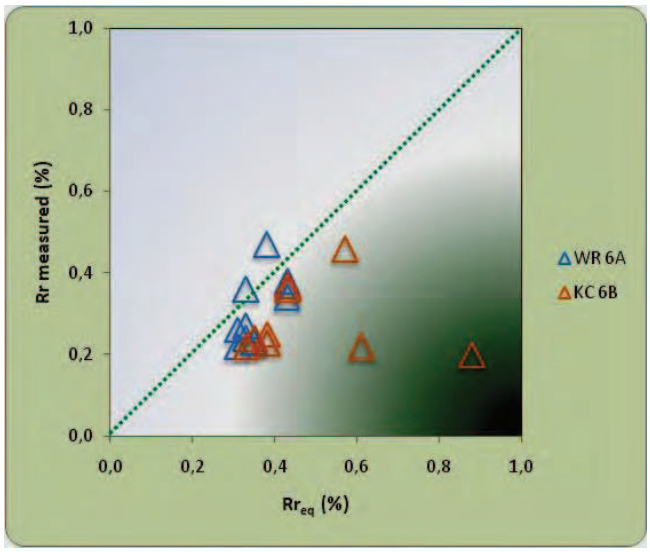
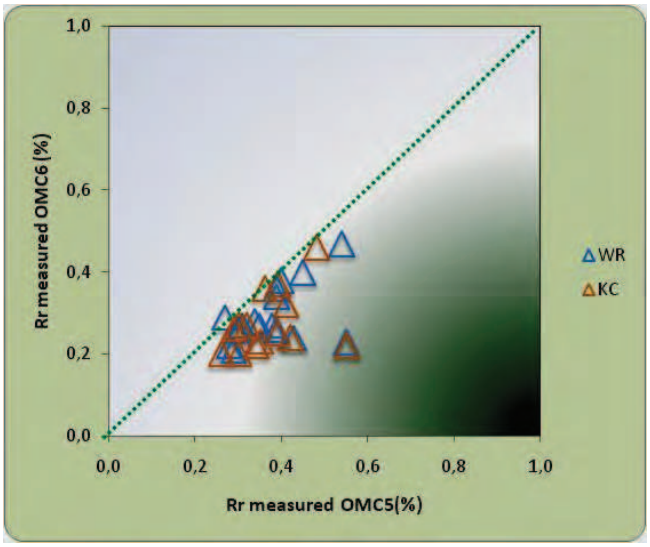


Figure 18: Comparison of equivalent R_r estimated from alginite and the measured values for both the whole rock and the kerogen concentrate in samples OMC5 and OMC6. Only the values for those reporting spectra are plotted.

6. Differences in maturity between samples as derived from optical parameters.

The two samples analyzed were low rank Type I organic matter samples. The results obtained from both samples for the different optical parameters overlap to some extent. To check which sample is more mature Figure 19 plots measured vitrinite reflectance and calculated reflectance from spectral data. In both cases, values were higher in the Green River sample (OMC5) than in the Tremembé sample (OMC6), indicating a higher maturity for the former according to the averaged values reported in Table 2.



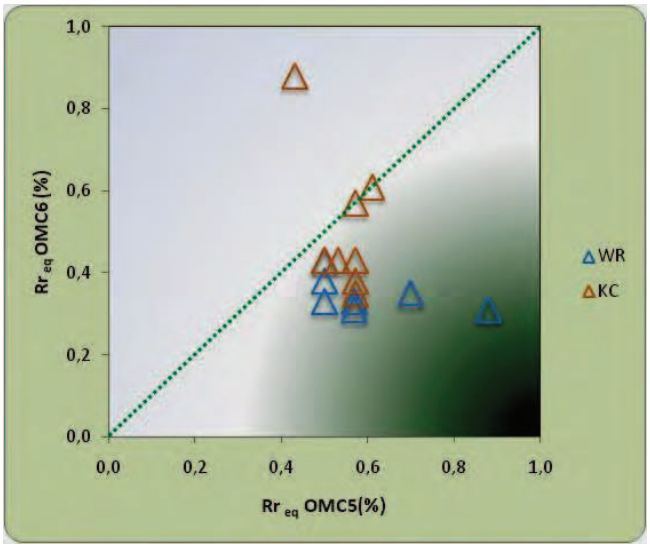


Figure 19: Comparison of the maturity of the two samples analysed based on the measured vitrinite reflectance and on the calculated vitrinite reflectance using spectral data.

7. Conclusions:

Based on the proposed objectives and results obtained in the studied samples for OMCWG 2010, it is concluded that Type I kerogen samples yield a lower amount of vitrinite than Type II and Type III kerogen samples (OMCWG 2009 and 2008, respectively).

Samples OMC5A and OMC5B (Green River Fm.) and OMC6A and OMC6B (Tremembé Fm.) showed a low to moderate amount of measurable vitrinite particles. This resulted in a large variation in the number of readings by each participant in both samples. The reported values were considered robust enough for the assessment of the effect of the isolation procedure on vitrinite reflectance of the samples.

In general, the Standard Deviation (SD) values in both samples were low and the scatter of the readings was small. However, the results for the Green River sample (OMC5) indicated that participants were measuring different vitrinite populations. This suggests greater difficulty to identify vitrinite particles in sample OMC5 than in sample OMC6. Specific instructions to select the vitrinite population in this sample are required. In the Tremembé sample (OMC6) the presence of two vitrinite populations was not evident from the data, although some scatter is also observed.

Spectral fluorescence results showed that λ_{max} values for sample OMC5 and OMC6 confirm a low rank for both samples. In general, the calculated vitrinite reflectance values using Mukhopadhyay (1994) were higher than the measured values for those participants providing both reflectance and fluorescence results. This could indicate suppression of vitrinite reflectance commonly reported in organic rich shales. The suppression would be higher for sample OMC5 if all measured values are taken into account because many of the lowest reflecting values corresponded to participants not reporting spectral data.

The spectral parameters of the kerogen concentrate and the whole rock measured on unstructured fluorescing organic matter (AOM) for the Green River sample (OMC5) showed significant scatter and no clear trend. On the contrary, spectral parameters of the kerogen concentrate measured on both telalginite and lamalginite in the Tremembé sample (OMC6) indicated a higher maturity for the kerogen concentrate. This result indicates that both components in this sample are sensitive to the isolation procedure. These results are interpreted to indicate that the preparation procedures affect fluorescence properties as observed by Mendonça Filho *et al.* (2009, 2010).

The equivalent vitrinite reflectance for sample OMC6A, using telalginite as the object of measurement, presents an excellent correlation with average measured vitrinite reflectance. However, in the equivalent vitrinite reflectance results for sample OMC6A, using lamalginite as the object of measurement, correlation with average measured vitrinite reflectance is poorer. For sample OMC6A, misfit between equivalent vitrinite reflectance and average measured vitrinite reflectance is high, and in sample OMC6B the misfit is very high. In this case, these results may suggest that those organic particles are inappropriate for this analysis, in addition to reflecting a more accentuated influence of the preparation procedures on the fluorescence properties of these macerals.

In summary, following the criteria and parameters described in the statistical evaluation system of ICCP (www.iccop.org), excellent results were obtained in this exercise and the selected samples allowed an accurate study on the effect of the isolation procedure on organic matter optical parameters in Type I kerogen.

8. References:

1. Barker, C.E., Pawlewicz, M.J., 1993. An empirical determination of the minimum number of measurements needed to estimate the mean random vitrinite reflectance of disseminated organic matter. Organic Geochemistry 20, 643-651.
2. Borrego, A.G., Araujo, C.V., Balke, A., Cardott, B., Cook, A.C., David, P., Flores, D., Hámor-Vidó, M., Hiltmann, W., Kalkreuth, W., Koch, J., Kommeren, K., Kus, J., Ligouis, B., Marques, M., Mendonça Filho, J.G., Misz, M., Oliveira, L., Pickel, W., Reimer, K., Ranasinghe, P., Suárez-Ruiz, I., Vieth, A., 2006. Influence of particle and surface quality on the vitrinite reflectance of dispersed organic matter: Comparative exercise using data from the qualifying system for reflectance analysis working group of ICCP. International Journal of Coal Geology, 68, p.151 - 170.
3. Espitalié, J., Laporte, J.L., Madec, M., Marquis, F., Leplat, P., Paulet, J., 1977. Méthode rapide de caractérisation des rochès, de leur potential pétrolier et de leur degree d'évolution. Revue de L'Institut Français du Pétrole 32, 23-43.
4. Mendonça Filho, J. G.; Araujo, C. V.; Borrego, A. G.; Cook, A.; Flores, D.; Hackley, P.; Hower, J.; Kern, M. L.; Kommeren, K.; Mendonça, J. O.; Menezes, T. R.; Newman, J.; Ranasinghe, P.; Souza, I. V. A. F.; Suarez-Ruiz, I.; Ujiié, Y. (2008). Report on Organic Matter Concentration Working Group (OMCWG 2008).

ICCP News, N°45, p. 43-51.
5. Mendonça Filho, J. G.; Araujo, C. V.; Borrego, A. G.; Cook, A.; Flores, D.; Hackley, P.; Hower, J.; Kern, M. L.; Kus, J.; Mastalerz, M.; Mendonça, J. O.; Menezes, T. R.; Souza, I. V. A. F.; Suarez-Ruiz, I (2009). Report on Organic Matter Concentration Working Group (OMCWG 2009). ICCP News, N°49, 2010, p. 19-28.
6. Mendonça Filho, J. G.; Araujo, C. V.; Borrego, A. G.; Cook, A.; Flores, D.; Hackley, P.; Hower, J.; Kern, M. L.; Kommeren, K.; Kus, J.; Mastalerz, M.; Mendonça, J. O.; Menezes, T. R.; Newman, J.; Ranasinghe, P.; Souza, I. V. A. F.; Suarez-Ruiz, I.; Ujiié, Y. (2010). Effect of concentration of dispersed organic matter on optical maturity parameters: Interlaboratory results of the organic matter concentration working group of the ICCP, International Journal of Coal Geology (doi 10.1016/j.coal.2010.07.005).
7. Mukhopadhyay, P.K. (1994). Vitrinite Reflectance as Maturity Parameter. Petrographic and Molecular Characterization and its Applications to Basin Modelling. In: Mukhopadhyay and Dow (eds.) Vitrinite Reflectance as a Maturity Parameter. Applications and Limitations. American Chemical Society, Washington. p. 1-24.

9. Acknowledgments:

¤ The sample from Green River Formation was provided by Paul Hackley (USGS).
¤ The sample from Tremembé Formation was provided by André L. D. Spigolon (CENPES/PETROBRAS).
¤ The effort of Alexandre J. Sant'Anna (CENPES/PETROBRAS) and Antonio D. de Oliveira (Palynofacies and Organic Facies Laboratory/IGEO/UFRJ) for sample preparation is gratefully acknowledged.
¤ Petrobras Research Development Center and Palynofacies and Organic Facies Laboratory/IGEO/UFRJ provided organic geochemistry analysis.
¤ Special thanks to Taíssa R. Menezes and Angeles G. Borrego for their help with statistical treatment and data interpretation.

DEADLINE FOR NEXT ICCP NEWS : 4TH NOVEMBER 2013

Membership Matters

please keep your email contact current - or you will miss out on important information

Contact Updates

Dr. Mária Hámor Vidó
Deputy Director
Geological and Geophysical Institute of Hungary
Stefania st. 14
Budapest, 1143
Hungary
Tel: +36 1 36 37 270
Tel: +36 1 36 37 840
Mobil 1:+36 30 84 14 053
Mobil 2:+36 20 37 46 796
Fax: +36 1 36 37 256
mailto: vido.maria@mfgi.hu
mailto:hamorvido@gmail.com
http://www.mfgi.hu

Wayne Knowles
Organic Petrologist
Geochemical Services Group
Weatherford Laboratories
Torridge House
4 Buttgarden St.
Bideford
Devon
EX39 2AU
UK
Direct: +44.(0)560.331.5333
Fax: +1.281.681.0326
mailto:Wayne.Knowles@weatherfordlabs.com | www.weatherfordlabs.com

Jolanta Kus now works in the Section "Geochemistry of Energy Resources and Gas Monitoring" with other details unchanged.

Joachim Koch has a new email address: joachim.koch002@kabelmail.de