70th Annual ICCP Meeting

International Committee for Coal and Organic Petrology

September 23rd – 29th 2018 | Royal on the Park | Brisbane, Australia

Conference Program

Geological Society of Australia

Hilgers Technisches Buero

FOSSIL Reflectance Measurement

ERC Energy Resources Consulting Pty Ltd

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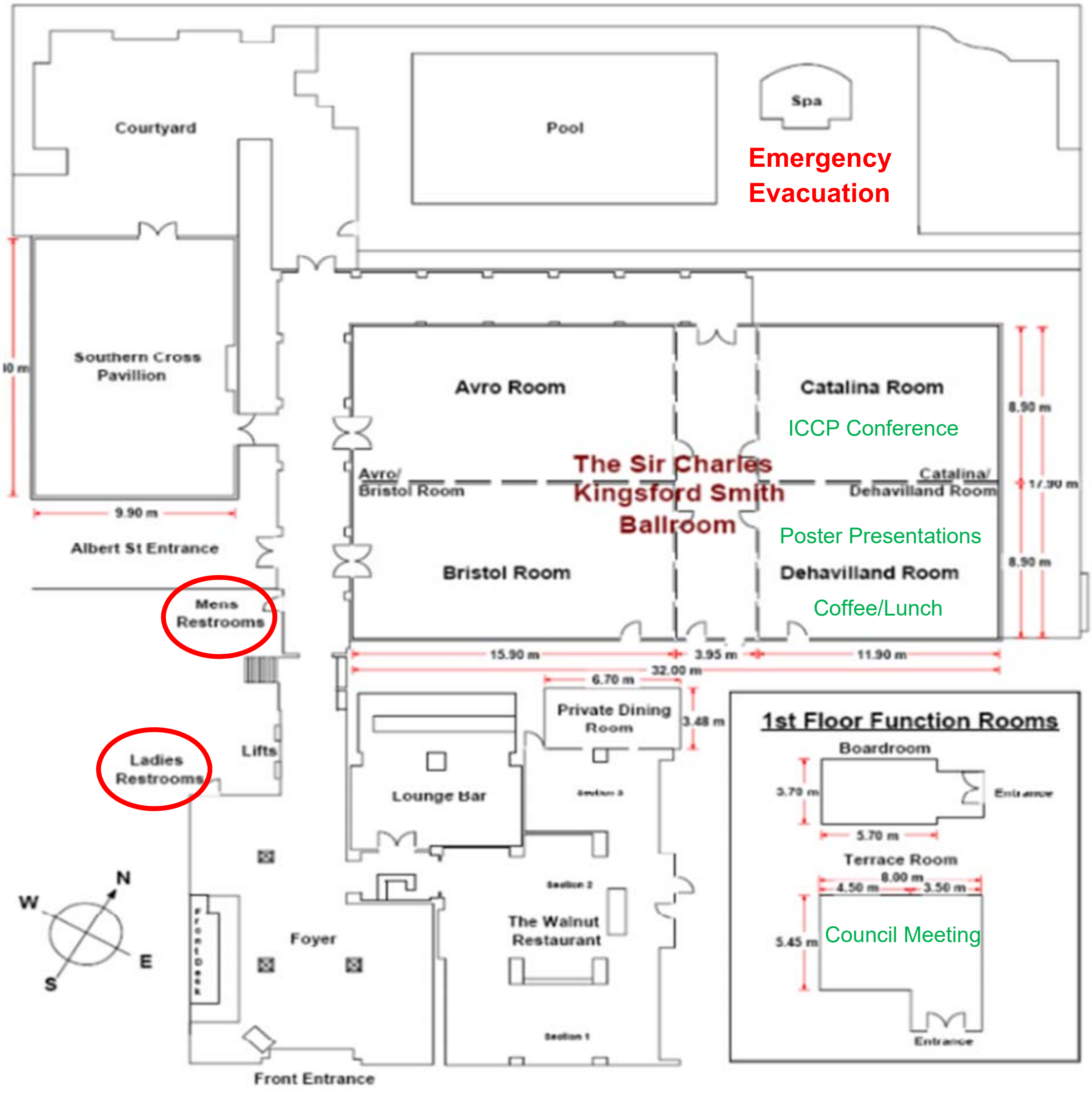
Courtyard
Pool
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Emergency Evacuation
Southern Cross Pavillion
0 m
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Albert St Entrance
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Avro/ Bristol Room
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1st Floor Function Rooms
Boardroom
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5.70 m
Terrace Room
8.00 m
4.50 m
3.50 m
5.45 m
Council Meeting
Entrance

Welcome Message

The ICCP is the premier international body for standardizing organic petrology, verifying the applications of these standards, certifying the competence of organic petrologists and investigating new methods and techniques. Results from this optical microscopy work is used by fossil fuel exploration and production companies, the steel, power and cement industries, universities, environmental agencies and government geological surveys.

The last time that the ICCP met in Australia was at Wollongong in 1990. Much has happened in the Australian (and global) context since that time. While record production and export volumes continue, there is a broad community perception that coal is in decline. Once new coal mine developments were welcomed for jobs and growth. Now they are actively campaigned against as being environmentally irresponsible. There is growing momentum to move electricity production away from fossil fuels into renewables.

It is against this background that the ICCP again comes to Australia. And not to just anywhere in Australia, but to Brisbane; the capital of Queensland and the centre for much of our coal production and coal seam gas activity.

The ICCP is arranged into 3 Commissions: Commission I on Nomenclature and Classification; Commission II on Geological Applications of Organic Petrology and; Commission III on Industrial Applications of Organic Petrology. Each commission is comprised of Working Groups. During the course of the year, Working Group members contribute to activities.

Those who have never attended an ICCP meeting find them unusual in many respects. At the annual meeting, it is the activities and results of the working groups that is reported and discussed rather than individual research results. In this way the views of multiple scientists can be compared and contrasted rather than just the opinion of an individual. This occurs over the rest four days.

In addition to the Commission Meetings, a one day symposium is incorporated into the programme. This year's theme is "Organic Petrology in the 21st Century" is designed to reflect the many challenges facing this community in coming years.

Back in 1990, Brisbane was best described as a large country town. Now it is a vibrant, cosmopolitan city that operates almost around the clock. Looking for a good coffee at 6am? - No problem. Looking for a good coffee at midnight? No problem. But be warned, Australia has a peculiar coffee language; a smalldoubleshotskinnycap (one word) is what I normally order.

The timing of the ICCP in late September is also co-incident with the major cultural event of the Brisbane Festival which has been occurring from the 8th of September and will conclude this Saturday on the 29th of September. Be warned, the last Saturday of the Festival and the ICCP Meeting, is programmed for a major re-works display "Riverfire" which sees 10's of thousands of people congregate in the city.

So, on behalf of the organizing committee, I warmly invite you to attend the fireworks of the 70th ICCP Meeting - either during or after the formal program.

Dr Peter Crosdale

Chair Organising Committee

Vice-President, ICCP

Key Note Speakers

Dr James William Beeston

James (Jim) studied at the University of Queensland St Lucia receiving a BSc degree in geology in 1972, an MSc degree in geology in 1981 and a PhD in coal geology in 1988. Jim started employment with the Geological Survey of Queensland in 1968 and joined the Coal Group based at Redbank in 1972. He joined a group of geologists carrying out major exploration and scientific studies on coal in the poorly explored Bowen, Galilee and Surat Basins. His major contribution was through coal petrology, including petrographic analysis and vitrinite reflectance measurement, the latter which led to rank maps for the above basins and others. Jim started the Coal Geology Group (Specialist Group) in the Geological Society of Australia Inc in 1976, officially starting operations in 1978 and still flourishing. Through the journal Coal Geology (later Australian Coal Geology) and support of numerous coal symposia, the group has initiated hundreds of papers on coal in Australia (and some overseas). For his work in the Coal Geology Group Jim was made an Honorary Member of the GSA. In 2011 he received The Leichhardt Award from the Bowen Basin Geologist Group in 'recognition of original contribution to the advancement of coal geology or related disciplines in Queensland'. Jim is still active in retirement acting as the CGG's Honorary Editor.

Dr Joan Esterle | Chair of the Vale-UQ Coal Geoscience Program

Professor Joan Esterle is the Chair of the Vale-UQ Coal Geoscience Program. She received her PhD from The University of Kentucky, USA, in 1990. She worked for 17 years with CSIRO, followed by GeoGAS-Runge Group before joining the UQ full time in 2010. Her research interests are varied but focused on how geological history impacts on coal measures behaviour during mining, processing and utilization. In addition to working with Vale and other industry partners, she conducts multi-client studies through the Australian Coal Research Program (ACARP), The Australian National Low Emissions Coal Research (ANLECRD), and the UQ Centre for Coal Seam Gas (CCSG).

Hon. Ian Macfarlane | Queensland Resources Council

Hon. Ian Macfarlane was elected to Federal Parliament in 1998 and became Australia's longest serving Federal Resources and Energy Minister and the Coalition's longest serving Federal Industry and Innovation Minister with over 14 years of experience in both Cabinet and shadow ministerial positions before retiring from parliament in May 2016. Ian became Chief Executive of the Queensland Resources Council in 2016 and is a non-executive director of Woodside Petroleum and the Chairman of the Innovative Manufacturing Co-operative Research Centre.

Conference Agenda - Overview

Time	Sunday Sept 23	Monday Sept 24	Tuesday Sept 25	Wednesday Sept 26	Thursday Sept 27	Friday Sept 28	Saturday Sept 29
07.00-07.30				Field Trip 1 - Jeebropilly			
07.30-08.00							
08.00-08.30		Registration	Coffee		Coffee	Coffee	
08.30-09.00			Commission 1				
09.00-09.30		Welcome			Commission 2	Symposium	Field trip 2 - Lone Pine
09.30-10.00							
10.00-10.30							
10.30-11.00		Coffee	Coffee		Coffee	Coffee	
11.00-11.30		General Assembly	Commission 3		Commission 2	Symposium	
11.30-12.00							
12.00-12.30							
12.30-13.00		Lunch	Lunch		Lunch	Lunch	Lunch
13.00-13.30							
13.30-14.00		Commission 1	Commission 3	Lunch	Commission 2	Symposium	Brisbane Festival River Fire Event in the City - Entertainment from 4pm - Fireworks from 7pm
14.00-14.30	Council Meeting			Commission 3			
14.30-15.00							
15.00-15.30		Coffee	Coffee		Coffee	Coffee	
15.30-16.00		Commission 1	Microscope	Coffee	General Assembly	Symposium	
16.00-16.30				Commission 3			
16.30-17.00							
17.30-18.00	Ice-breaker		ICCP Council Meeting				
18.00-18.30					Conference dinner		
18.30-19.00							
19.00-19.30							
19.30-20.00							
20.00-20.30							
20.30-21.00							
21.00-21.30							
21.30-22.00							
22.00-22.30							

Conference Agenda – Detailed

Monday, September 24th 2018

Venue: Royal on the Park Hotel

08:00 - 09:00	**Registration**
09:00 - 09:10	**Welcome to the 70th ICCP Meeting** *Dr. Peter Crosdale*
09:10 – 09:40	**Formal Opening** *Dr Jim Beeston*
09:40 – 10:10	**Keynote: Back to Basics** *Dr Joan Esterle*
10:10 - 10:30	Group Photo at the Botanical Gardens
10:30 - 11:00	**Coffee Break**
11:00 - 11:30	**Keynote: How industry sees the incentives for exploration in Queensland** *Hon. Ian Macfarlane*
11:30 -12:30	**Opening Plenary Session of the General Assembly** **Chair: Dr Angeles Borrego, President ICCP**
12:30 - 13:30	**Lunch Break**
13:30 - 13:45	**Meeting of Commission I** **Chair: Dr Stavros Kalaitzidis** **Secretary: Dr Dragana Životić** Opening remarks and schedule of Commission I
14:45 - 14:45	**SCAP – Single Coal Accreditation Program** Prof. Dr Kimon Christanis
14:45 - 15:00	**ISO Standard** Dr Walter Pickel
15:00 - 15:30	**Coffee Break**
15:30 - 16:00	Continuation of Commission I **Xylite-rich Lithotype Classification WG** Dr Yiannis Oikonomopoulos
16:00 - 17:00	**Standardization WG** Dr Walter Pickel

Tuesday, September 25th 2018

Venue: Royal on the Park Hotel

08:30 - 08:45	**Coffee**
08:45 - 09:00	Meeting of Commission I cont.… **Suberinite WG** Dr Peter Crosdale
09:00 - 09:30	**Distinguishing Features of macerals WG** Dr Walter Pickel
09:30 - 09:50	**Petrographic Image Database** Dr Petra David
09:50 - 10:10	**New Handbook** Dr Petra David
10:10 - 10:30	*Closing remarks of Commission I – Chair of Commission*
10:30 - 11:00	**Coffee Break**
11:00 - 11:20	**Meeting of Commission 3:** **Chair: Dr. Magdalena Misz-Kennan** **Secretary: Dr Sandra Rodrigues** Opening remarks and schedule of Commission III
11:20 - 11:30	**Liquefaction Residues WG** Dr Henny Gerschel - presentation delivered by the chair.
11:30 - 12:30	**Fly Ashes WG** Dr Isabel Suárez-Ruiz
12:30 - 13:30	**Lunch Break**
ey, Kalaitzidis13:30 - 14:00	Meeting of Commission 3 cont… **Gasification Products WG** Dr Nikki Wagner
14:00 - 15:00	**Self-heating WG** Dr Magdalena Misz-Kennan
15:00 - 15:30	**Coffee Break**
15:30 - 17:00	**Microscope Session** Samples by Hackley; Kalaitzidis; Borrego; Pickel; Crosdale

Wednesday, September 26th 2018

Venue: Royal on the Park Hotel

07:00 - 13:30	**Field Trip 1** 07:00 Depart Royal on the Park (if you are late you will be left behind) 07:20 Arrive Mt Coot-tha Lookout for view of Brisbane City 07:50 Depart Mt Coot-tha 08:20 Arrive Brisbane Correctional Centre (Gaol), Wacol – wildlife (kangaroos) viewing. 08:50 Depart 09:20 Arrive Jeebropilly Coal Mine 11:30 Depart Jeebropilly Coal Mine 11:50 Arrive Dinmore Plant Beds – fossil collecting 12:45 Depart Dinmore Plant Beds 13:30 Arrive Royal on the Park for lunch
13:30 -14:00	**Lunch Break**
14:00 - 14:20	Meeting of Commission III cont… **Organic Matter Oxidation WG** Dr Isabel Suárez-Ruiz
14:20 - 14:40	**Environmental Applications WG** Dr. George Siavalas Dr Stavros Kalaitzidis
14:40 - 15:00	**Optimization of the reflectance measurements on complex blends** Dr Ashok Singh
15:00 - 15:30	**Coffee Break**
15:30 - 16:10	**Coke Petrography WG** Dr Małgorzata Wojtaszek– Presentation by *skype*
16:10 - 16:45	*Closing remarks of Commission III – Chair of Commission*

Thursday, September 27th 2018

Venue: Royal on the Park Hotel

08:30 - 09:00	**Coffee**
09:00 - 09:15	**Meeting of Commission II:** **Chair: Dr. Paul C. Hackley** **Secretary: Dr. Jolanta Kus** Opening remarks and schedule of Commission II
09:15 - 09:50	**Gas Shale Studies** Dr Lila Gurba
09:50- 10:30	**ICCP-TSOP DOM Atlas** Dr Isabel Suárez-Ruiz
10:30 - 11:00	**Coffee Break**
11:00 - 11:30	**Confocal Laser Scanning Microscopy WG** Dr Paul Hackley
11:30 - 12:00	**CBM-CO_2 WG** Dr Lila Gurba
12:00- 12:30	**Identification of Primary Vitrinite WG** Dr Paul Hackley
12:30 - 13:30	**Lunch Break**
13:30 - 14:30	Meeting of Commission II cont… **DOMVR lessons learned** Dr Angeles Borrego
14:30 - 14:45	**2018-2019 DOMVR Accreditation Program** Dr João Graciano Mendonça Filho
14:45 - 15:00	*Closing remarks of Commission II - Chair of Commission*
15:00 - 15:30	**Coffee Break**
15:30 - 17:00	General Assembly
18:00 - late	**Conference Dinner at Blackbird Bar and Grill (Restaurant downstairs)** **Riverside Centre, 123 Eagle St, Brisbane City**

Friday, September 28th 2018

Symposium
“Organic Petrology in the 21st Century”

Venue: Royal on the Park Hotel

08:00-09:00	**Coffee**
09:00-09:10	**Session 1 - Thermal Maturity** *Convenor - Dr Angeles Borrego*
09:10-09:35	**Impacts of thermal history and maceral composition on the Walloons coal seam gas system** *Dr Mohinudeen Faiz*
09:35-10:00	**Assessing Hydrocarbon Potential of Old Rocks Using Programmed Pyrolysis Techniques (Rock Eval)** *Peter J Crosdale and S. P. Ranasinghe*
10:00-10:25	**Organic Petrography and Diagenesis of Wolfcamp Mudrocks and Carbonates, Midland Basin.** *Paul C. Hackley, Tongwei Zhang, Brett J. Valentine, Frank T. Dulong, Javin J. Hatcherian, and Aaron M. Jubb*
10:25-10:30	Closing Remarks
10:30-11:00	**Coffee**
11:00-11:10	**Session 2 - Carbons** *Convenor - Dr Isabel Suárez-Ruiz*
11:10-11:35	**Fifty shades of grey: The extraction of char from a variety of coal ash for consideration as synthetic graphite** *C. Badenhorst; N. Wagner; B. Valentim; F. Viljoen*
11:35-12:00	**Optical characterization of carbon deposits formed during targeted methane decomposition and their incorporation in the coke structure.** *H. Liszio, M. Schulten, V. Stiskala, P. Liszio*
12:00-12:25	**Structural characterization of selected Indian coals by wide angle X-ray scattering and its correlation with Petrographic Rank Parameter** *Prabal Boral, Sudip Maity, Ashok K. Singh, Pradeep K. Singh and Atul K. Varma*
12:25-12:30	Closing Remarks
12:30-13:30	**Lunch**

13:30-13:40	Session 3 - Coal Characterisation **Convenor - Dr Paul Hackley**
13:40-14:05	**Applications of Coal Grain Analysis (CGA).** *K. Warren, G. O'Brien, P. Hapugoda and S. Koval*
14:05-14:30	**Organic petrology and geochemistry of hydrocarbon inclusions in the Pennsylvanian carbonates of Picos de Europa mountains (Cantabrian Zone, N of Spain)** *A. López García, O. Merino Tomé, J.R. Bahamond and A.G. Borrego*
14:30-14:55	**Development of Very Thick Permian Coal Seams in the Cooper Basin, Australia** *E. Alganaeva and G.C.Smith*
15:25-15:00	Closing Remarks
15:00-15:30	**Coffee**
15:30-15:40	**Session 4 – University Research** *Convenor – Dr Joan Esterle*
15:40-15:55	**An appetite for apatite: A micro-analysis review** *B. Davis and A. Duxbury*
15:55-16:10	**Magnetic susceptibility in coal: Is it a possibility?** *E. Williamson*
16:10-16:25	**Petrographic characterization of DOM in Proterozoic source rocks, McArthur Basin, Australia** *S. Rodrigues, S. Golding, S. Hamilton*
16:25-16:30	Closing Remarks

Saturday, September 29th 2018

08:30 - late	**Field Trip 2** 08:30 Depart Royal on the Park 09:00 Arrive Lone Pine Koala Sanctuary http://www.koala.net/en-au/ Entry fee and transport only are provided. Kangaroos and emus can be patted for free or food purchased for a small sum (about $2) for feeding; photographs with Koalas etc. are available at your own cost. Check Sanctuary schedule for free snake / crocodile shows – you may be able to pat the snakes but crocodile patting is discouraged. 11:30 Depart Lone Pine Koala Sanctuary 12:00 Lunch Arrive Fritzenberger Brewery. 52 Petrie Terrace, Milton Some time later…. Depart Fritzenberger and make own way to city (one train stop or 20 minute walk) to enjoy the River Fire Festivities of the Brisbane Festival

ICCP Symposium
“Organic Petrology in the 21 Century”

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Oral Presentations – in presentation order

Poster Presentations – in alphabetical order by title

Abstracts
For Oral presentations

Impacts of thermal history and maceral composition on the Walloons coal seam gas system

Mohinudeen Faiz

Source to Reservoir Solutions

e-mail: source2reservoir@gmail.com

Abstract

The Middle Jurassic Walloon coals are enriched in liptinite and in particular suberinite derived from bark tissues (Fig. 1). The composition of many Walloon coal seams approach that of sapropelic coal (i.e. elevated the H/C ratios) and is grossly different to typical humic coals encountered in San Juan (USA), Powder River (USA), Bowen (Australia) and Western Canada basins (Fig. 2). Therefore, the behaviour of coal seam gas reservoirs in these basins cannot be directly used as an analogue to model the Walloon coal seam gas system. Liptinite-enriched coals are dull in nature, tend to be ductile and when immature contain a low frequency of cleat. However, with increasing thermal maturity these coals are expected to develop more cleat and become increasingly brittle.

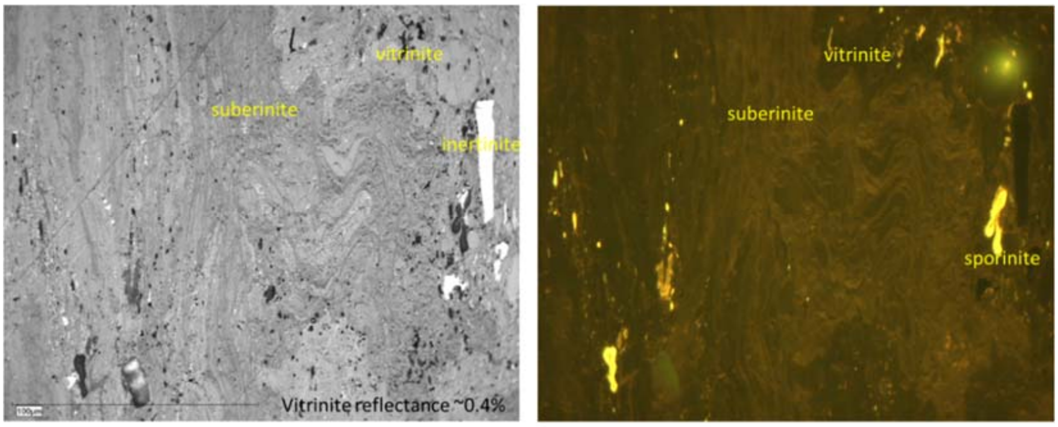

Figure 1. Suberinite enriched coal, Walloon Coal Measures, Surat Basin.

The majority of gas in Walloon coals is interpreted to be biogenic in origin. Biogenic gas can be either primary or secondary with respect to burial history of the basin. Primary biogenic gas was generated during the Jurassic soon after coal (peat) was deposited and continued to generate throughout early stages of basin subsidence. Early thermogenic hydrocarbons were generated within deeper parts of the Mimosa Syncline as well as in specific structural domains where the pre-Jurassic section is thin and the basal heat-flux is elevated. Subsequently, when the coals were uplifted to present day depths secondary biogenic gas was generated through metabolism of pre-existing thermogenic hydrocarbons, carbon dioxide and organic components of the coal. Secondary biogenic gas generation within the uplifted coals requires the influx of appropriate micro-organisms together with nutrients to promote organic metabolism. The present-day gas content and methane saturation levels are largely a function of thermal history, microbial ecology, permeability and hydrodynamics of the system. A large part of the gas in the Walloons is likely to be primary biogenic gas, however, secondary biogenic gas is a significant component in the highly productive "sweet spots".

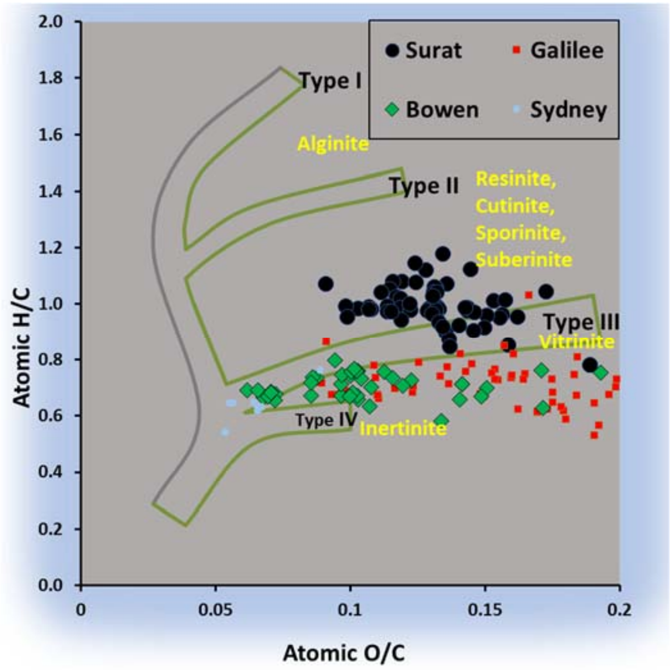

Figure 2. van Krevelen diagram showing perhydrous nature of Surat Basin coals. Gas reservoir properties of Surat Basin coals are affected by the high aliphatics content.

Basin modelling suggest that maximum burial depth, heat flow and the extent of post-Cretaceous exhumation vary across the Surat Basin and these impact coal seam gas reservoir properties. Highly permeable coals are encountered in areas where the sedimentary sequence has been excessively uplifted since the Cretaceous thermal maturation (Fig 3).

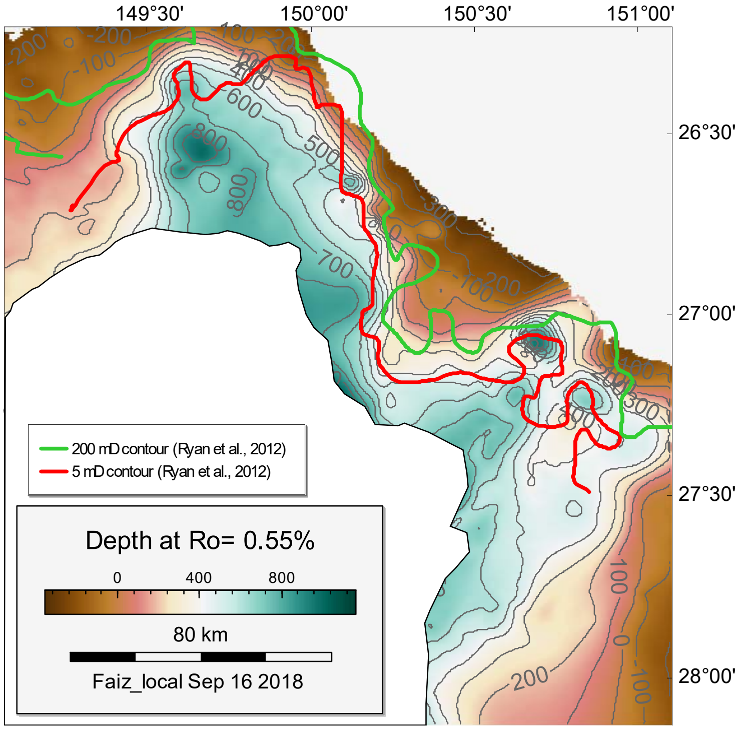

Figure 3. Iso-maturity depth map reflecting the extent of post maturation uplift in the Surat Basin. Also shown are 5 mD and 200 mD permeability contours for the Walloons CSG reservoir.

References

Ryan, D.J., Hall, A., Erriah, L. and Wilson, P.B. 2012. The Walloons Coal Seam Gas play, Surat Basin, Queensland. APPEA Journal 2012, 273-290.

Assessing Hydrocarbon Potential of Old Rocks Using Programmed Pyrolysis Techniques (Rock Eval)

P. J. Crosdale and S. P. Ranasinghe

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e-mail: peter.crosdale@energyrc.com.au

Abstract:

Precambrian rocks are locally important as oil and gas plays e.g. Beetaloo Basin, Northern Territory. They can be organic rich and have demonstrably generated hydrocarbons. Interpretation of the geochemical characteristics of these rocks is difficult due to the different nature of the initial organic matter. Kerogen in such old rocks is relatively poor in hydrogen. Proterozoic kerogens commonly have $H/C < 0.2$ compared to modern plankton starting at $H/C = 1.6$, which decreases during maturation (Hunt, 1996). While low H/C ratios may be a function of thermal history, there is a suggestion that Proterozoic organisms may have had fundamentally different organic chemistry, which affects interpretation of thermal maturation by techniques such as programmed pyrolysis (RockEval).

A number of wells from the Beetaloo Basin have been geochemically characterised using a HAWK instrument for programmed pyrolysis parameters (e.g. T_{max}). The same samples have also been characterised by optical microscopy for organic matter typing and reflectance determination.

Optical microscopy confirms that the primary organic matters present are dominantly algal. Reflectance of the algae increases in a regular manner with depth, as would be expected as a normal thermal maturation trend. Bitumen is also readily identified in many samples. It too shows an increasing reflectance trend with depth. However, multiple bitumen populations may be present and care must be taken to measure only one of them. In addition, there is a clear association between the appearance of bitumen in large quantities at algal reflectances of about 0.8 to 0.9%. This appearance of the bitumens is associated with an apparent decrease algal abundance. It is suggested that the generation of large quantities of bitumen from the algae cause the algae to lose large amounts of mass, allowing them to substantially compress and thereby making their identification much more difficult. Such a mechanism would be at odds with the notion that the original algae had low hydrogen content but would be consistent with a low H/C ratio for the subsequent kerogens.

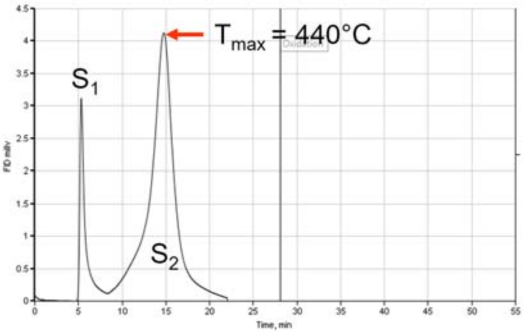
Organic matter typing on a pseudo van Krevelen plot using programmed pyrolysis data gives a confusing picture. Many samples plot on the Kerogen Type I/II trend consistent with an algal affinity as confirmed by petrographic observation. However, very many samples also plot on the Kerogen Type III trend. Kerogen type III are classically related to terrestrially derived vitrinites, which did not exist at this time. Grouping the data into 0.5% TOC classes shows that samples with a TOC > 2.0% plot in the expected Type I / II kerogen trend. When TOC < 2.0%, a type III kerogen trend is developed. This suggests that low amounts of CO and CO2 are evolved with low TOC contents are possible within detection limits, leading to spuriously high OI values.

The classical rank parameter of pyrolysis is T_{max}. This parameter follows the normal trend of increasing T_{max} with increasing organic matter (alginite) reflectance for lower maturations. There seems to be a maturation jump at the point where alginite fluorescence disappears and large amounts of bitumen are generated. At higher levels of maturation, T_{max} becomes extremely problematic. The pyrolysis FID trace suddenly becomes weak and multi-modal, with the most prominent mode ($=T_{max}$) often at extremely low values, as low as about 320°C in

many cases. Careful analysis and interpretation of the FID trace in this region leads to rejection of the machine determined T_{max} value in favour of one at much higher temperature. This new value ($T_{max\text{-}mat}$) is more representative of the thermal maturation (and hence $T_{max\text{-}mat}$) and provides a better correlation with primary organic matter reflectance.

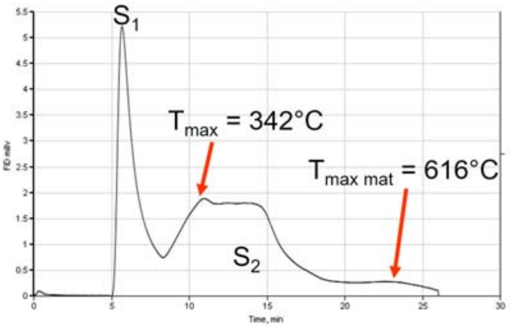
References:

Hunt, J.M. (1996) Petroleum Geochemistry and Geology. 2nd ed. W.H. Freeman and Company, New York. 743pp.



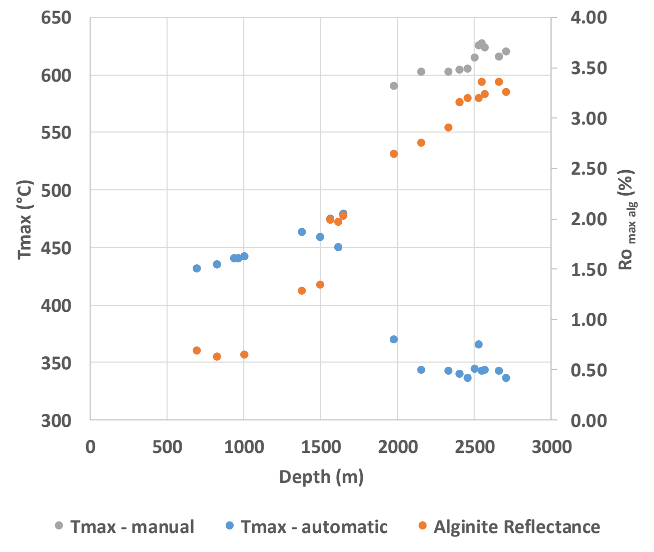
Normal, low maturity FID Trace

- Single, well defined peak
- Strong signal
- T_{max} good indicator of maturation
- TOC of sample =0.68%



Typical, high maturity FID Trace

- Multiple peaks
- Weak signal
- T_{max} does not represent maturation
- $T_{max\text{-}mat}$ parameter preferred to represent maturation
- TOC of sample =0.75%



- Reflectance of alginite increases with depth as expected for a normal maturation trend
- Instrument determined T_{max} increases with depth up to about T_{max} = 475°C, beyond which this measurement does not follow expected maturation trends and yields values that are demonstrably too low
- Manually determined $T_{max\text{-}mat}$ is a better indicator at higher levels of maturation

Organic Petrography and Diagenesis of Wolfcamp Mudrocks and Carbonates, Midland Basin

Paul C. Hackley[1,*], Tongwei Zhang[2], Brett J. Valentine[1], Frank T. Dulong[1], Javin J. Hatcherian[1], and Aaron M. Jubb[1]

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[2] Bureau of Economic Geology, Jackson School of Geosciences, The University of Texas at Austin, J.J. Pickel Research Campus, 10611 Exploration Way, Austin, TX, 78758, USA
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Abstract

As part of a larger investigation, this study used an organic petrology approach to evaluate the occurrence of source and reservoir facies in Leonardian Wolfcamp A core samples from the R. Ricker #1 well in northern Reagan County in the Midland Basin, Texas, USA. The study was designed to address the following research questions: 1. is free oil in the Wolfcamp A associated with a specific organic matter type, and/or is it related to petroleum expulsion efficiency as a function of lithofacies?, and 2. has petroleum expulsion from organic-rich facies to organic-lean facies occurred in repeated meter-scale lithofacies cycles in the Wolfcamp A? The answers to these questions potentially can be used to develop a practical oil storage and saturation model for the Wolfcamp Formation in the Permian Basin, which presently produces about 3 million barrels of oil per day and is forecast to produce about 30% of total US oil production in 2018.

Previous workers identified organic-rich siliceous and calcareous mudrock and organic-lean carbonate lithofacies in the Wolfcamp A. These lithofacies were defined by sedimentary features including grain size and relative quartz and calcite abundances. Organic-rich siliceous and calcareous mudrocks represent predominantly hemipelagic basinal sediments deposited below storm wave base, whereas the fine-grained, organic-lean carbonate lithofacies represent turbidite deposition (also deep water) of sediment transported from adjacent shelf areas.

Fifty-three core samples in total were selected from the R. Ricker #1 well for this study at depths ranging from about 7907 to 8016 ft below surface and analyzed via programmed pyrolysis (Hawk), LECO total organic carbon (TOC), and X-ray diffraction (XRD) mineralogy. Of this overall sample set, sixteen samples were selected for organic petrography and three samples from this subset were examined via scanning electron microscopy.

Petrographic observations of siliceous mudrocks (n=6) (solid bitumen, quartz+clay-rich matrix, pyrite framboids, micrinite, phosphatic cements) are consistent with an original Type II organic matter comprised of planktic algal material from the photic zone and its derivatives: amorphous organic matter and bacterial biomass. This organic matter would have been incorporated into the hemipelagic sediment, deposited as a marine snow and preserved in anoxic conditions. A moderate correlation (r^2 0.75) between loss-on-ignition (LOI) and total clays may suggest original organic carbon was adsorbed to clay surfaces for transport and burial preservation, although this relationship has been subsequently modified by kerogen conversion and hydrocarbon expulsion during thermal maturation. Summarizing, organic matter in the source lithofacies is dominated by solid bitumen interpreted as a polar molecule-rich residue of an original oil-prone Type II kerogen which has converted to petroleum.

The fine-grained carbonate reservoir lithofacies (n=4) contain abundant calcareous microfossils, e.g., calcispheres, minor pyrite framboids (locally some white-colored sulfide), and patches of interstitial granular solid bitumen occurring in a microcrystalline calcium

phosphate cement. As there is no petrographic evidence for the presence of an original oil-prone kerogen in the four fine-grained carbonate reservoir facies samples, the solid bitumen present is interpreted as a migrated phase from the adjacent organic-rich siliceous mudrock and calcareous mudrock facies. The presence of granular solid bitumen within interstices of microcrystalline calcium phosphate cement suggests kerogen conversion was coeval with or occurred in the later stages of phosphatic cementation. The occurrence of microcrystalline calcium phosphate cements within both source and reservoir lithofacies suggests pervasive vertical migration of cementing fluids throughout the sediment stack, i.e., phosphatic cements are not restricted to a single lithofacies and occur throughout the samples. Clay minerals were not observed in reservoir facies via optical microscopy and were not detected in the XRD patterns from low temperature ash residues, suggesting absence of a marine snow depositional mechanism for organic carbon transport and burial preservation. This lithofacies is interpreted as turbidite and debris flow deposition during high carbonate production at the shelf margin.

The fine-grained carbonate reservoir lithofacies transitions upwards to siliceous mudrock source lithofacies through decreasing carbonate abundance, with matrix-supported detrital (largely biogenic, e.g., calcispheres) and authigenic carbonate occurring in a quartz+clay matrix. The petrography and XRD results indicate these samples represent a transitional calcareous mudrock source lithofacies (n=6). This transitional lithofacies is interpreted as occurring due to resumption of hemipelagic sedimentation and landward stepping of the carbonate factory. The presence of significant quartz+clay content (39 to 60 wt.%) and moderate LOI values is consistent with a ‘marine snow’ mechanism for accumulation of original oil-prone organic matter in the transitional lithofacies.

Measured values of mean solid bitumen reflectance (BR_o) range from 0.55 to 0.86% BR_o. The solid bitumen reflectance values are consistent with the R. Ricker #1 samples being in the early to middle oil window. The absence of any conclusive organic fluorescence in the source lithofacies samples also is consistent with mid-oil window thermal maturity and is consistent with prior studies which suggested similar thermal maturity.

In summary, the absence of definitive oil-prone kerogen and presence of abundant solid bitumen with reflectance of 0.55 to 0.86% suggests complete or near-complete conversion of oil-prone kerogen to petroleum in the Wolfcamp A study location. Organic-rich siliceous and calcareous mudrock source lithofacies contains solid bitumen interpreted as polar-rich petroleum that is residual from the conversion of original Type II kerogen during thermal maturation. The original Type II kerogen consisted of algal matter from a high productivity photic zone potentially influenced by nutrient upwelling (as suggested by presence of phosphatic cements). Algal organic matter and its derivatives were likely deposited as marine snow associated with siliciclastic sedimentation and preserved in anoxic bottom sediments. The organic-lean fine-grained carbonate reservoir lithofacies contains migrated hydrocarbons sourced from nearby siliceous and calcareous mudrock lithofacies. Kerogen conversion and petroleum migration occurred coeval with or in the latter stages of phosphatic cementation. The observations suggest high free oil contents (high oil saturation index values) observed in the Wolfcamp A are due to primary migration of petroleum from siliceous and calcareous mudrock source lithofacies into adjacent fine-grained reservoir lithofacies, and that the repeated meter-scale lithofacies cycles are favorable for this primary migration.

Fifty shades of grey: The extraction of char from a variety of coal ash for consideration as synthetic graphite

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Abstract

Graphite is at the forefront of technological development with Li-ion batteries, fuel cells and pebble-bed nuclear reactors all relying on graphite to function successfully (Desjardins, 2012). However, a shortage in natural graphite is problematic for the above-mentioned end-markets and alternative sources, such as synthetic graphite, are highly sought after (European Commission, 2010). In this study, the char fraction found in coal combustion ash (also known as unburned carbon) is evaluated as a possible precursor for synthetic graphite. These char particles have a high degree of turbostratic order which is an essential requirement for a material to be graphitizable (Hurt *et al.,* 1995). As a secondary objective, removing the char will also result in a cleaner ash product more suitable for cement and concrete applications (Hower *et al.,* 2017).

The research forms part of an ERA-MIN collaboration between Portugal, Romania, Argentina, Poland, and South Africa. The end-point is to obtain a 90% carbon product for graphitisation trials. Coal ash samples were obtained and characterised. Chemical and mineralogical analyses revealed that the ash samples consisted mainly of aluminum-silicon oxides such as glass, quartz, mullite, and anorthite, and in lesser amounts hematite, magnetite, and char. Carbon percentages ranged between 1 and 8 wt. % and detailed analyses showed that ~80% of the carbon is of elemental (graphitic) form whilst the rest is of organic and inorganic carbon forms (non-graphitic). Petrographic analyses indicated that the char particles have mainly anisotropic and inertinitic textures, and some chars contained included mineral matter.

The char particles were separated from the ash using a variety of dry beneficiation techniques. Size, density, froth flotation, electrostatic, and magnetic separation were all used on a “trial and error” basis and loss of ignition (LOI) (inferring total organic carbon) was determined on each product. Combining sizing, electrostatic, and magnetic separation stages gave the best results for the South African ash samples (Table 1). One sample was submitted for dense medium separation in Poland, where 79 wt % carbon was achieved. Yields are in the order of 40%. The char particles were characterised in detail petrographically to understand if further liberation was possible (Table 2; Figures 1 – 2).

The chars will be assessed by x-ray diffraction (XRD) and Raman to determine the organic structure, which will be compared to natural graphite from the region. This information is important to consider the graphitizability of the prepared chars.

Acknowledgements:

The funding for this collaborative ERA-MIN project was obtained from the DST-NRF in South Africa, with support funding from CIMERA (DST-NRF Centre of Excellence in Earth Mineral Resource Analyses). This work is based on research supported wholly / in part by the National Research Foundation of South Africa (NRF) (Grant number 103466). 3rd ERA-MIN Joint Call (2015), project CHARPHITE: Fundação Ciência e Tecnologia (ERA-MIN/0005/2015); Institute of Earth Sciences (ICT; FCT UID/GEO/04683/2013); COMPETE POCI-01-0145-FEDER-007690.

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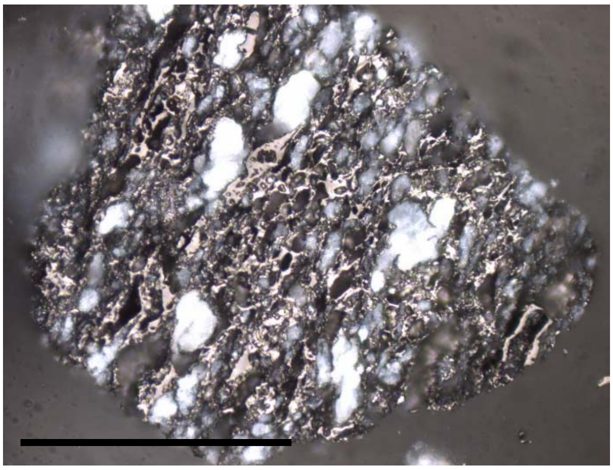
Table 1: Carbon concentration in four ash products from South Africa using a variety of techniques. FA = fly ash; PS = power station; BA = bottom ash; GA = gasification ash **(wt. % C)**.

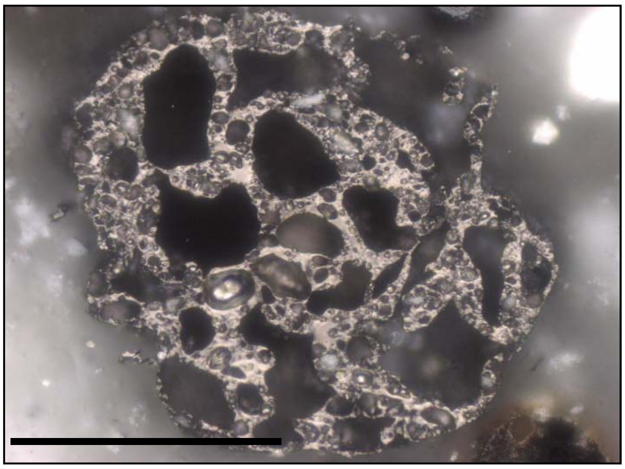
	Initial grade	**Grade after size separation**	**Grade after electrostatic separation**	**Grade after magnetic separation**	**Grade after dense medium separation**
FA PS1	7.04	12.56	44.30	65.74	78.99
FA PS2	4.01	8.44	36.70	56.55	NA
BA PS1	5.47	NA	45.17	53.21	NA
GA East	6.90	NA	37.12	45.10	NA

Table 2: Char composition of FA PS1 after sizing, electrostatic, and magnetic separation (vol %).

Tenuisphere	**Crassisphere**	**Tenuinetwork**	**Crassinetwork**	**Mixed porous**	**Mixed dense**	**Inertoid**	**Fusinoid/Solid**	**Mineroid**
0	0	4.9	13.4	17.4	24.7	11.8	11.6	16.2



a



b

Fig 1a: Char particle showing included minerals; it is difficult to remove these minerals. (BA PS1)
Fig 1b: Char particle showing a relatively ash-free char particle (FA PS1)
(Reflected light microcope, oil immersion, X500; scale bar = 100 microns)

Optical characterization of carbon deposits formed during targeted methane decomposition and their incorporation in the coke structure

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Abstract

A research consortium tried to develop a green and competitive basis for a large-scale use of the greenhouse gas methane. The project target was an alternative production of H2 with a low CO2-footprint as a feedstock for synthesis gas. Conventional processes used for synthesis gas production are typically employing the process of reforming in which the feedstock such as natural gas or petroleum reacts with steam or oxygen under a large energy supply.

Within the framework of the project, an alternative approach has been taken to produce H2 without simultaneous production of CO2. Besides hydrogen, the new process generates elementary carbon. The pyrolysis of natural gas / methane delivers hydrogen yield of 25% together with 75% yield of carbon which can be used in several industrial processes. The utilization of such carbon feedstock in coking coal blends is one of many possibilities. This kind of pyrolytic carbon can be used as an additive or as a substitute for other fossil carbon resources.

In the presented work, the influence of pyrolytic carbon as well as other carbon-based products was tested in several test campaigns. Both laboratory scale tests of coal dilatation and pilot scale carbonization tests were carried out with up to 20% of the additives in the coal blend. Coke properties such as cold mechanical strength, reactivity to CO2 and strength after reaction (CSR) were determined from the produced cokes. Furthermore, the optical character of the additives was analyzed. Various types of additives showed various capabilities to get incorporated in the coke structure. The incorporation of these components has a huge influence of the quality of produced coke.

Structural characterization of selected Indian coals by wide angle X-ray scattering and its correlation with Petrographic Rank Parameter

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Abstract

In order to characterize the evolution of organic matter during coalification, ten coal samples were collected from different basins of India. These coals were characterized chemically and dry mineral free Carbon (C_{dmf}) content of the coals varied between 76.51 – 90.88 %. The mean random reflectance of vitrinite (*VRr*) of coals were determined for each sample and varied between 0.44 – 1.24 %. All the coals were demineralized using standard HCl + HF treatment to overcome the interference effect of mineral matter during X-ray analysis of coal. Structural parameters of the demineralized coals were determined by Wide Angle X-ray Scattering (WAXS).

WAXS patterns of the samples were recorded using a slow step scan (0.02°/step) over an angular range 2θ ~ 10 – 90°. TOPAS 3.0 (M/s BRUKER AXS, Germany) software was used to determine the structural parameters like inter layer spacing (d_{002}), aromaticity (f_a) and ratio of intensity of π- and γ-band (I_{26}/I_{20}) which were determined by fitting the hump in WAXS graph at broad peak position of around 26° and 20° known as π- and γ- band respectively. The I_{26}/I_{20} values of X-ray are also called as X-ray rank parameter.

It was observed that d_{002} values of coal decreased with increase in maturity and ranging from 3.521 – 3.451 Å. Whereas, the f_a and I_{26}/I_{20} values increased with maturity and varied from 0.30 – 0.53 and 1.09 – 2.54 respectively. The d_{002}, f_a and I_{26}/I_{20} values were plotted against *VRr* and shown in Fig. 1 – 3 with high coefficient of determinations. Based on the above studies, it was inferred that the X-ray parameters of coals are petrographic rank dependent.

Key words: Coal, WAXS, petrography, reflectance, rank.

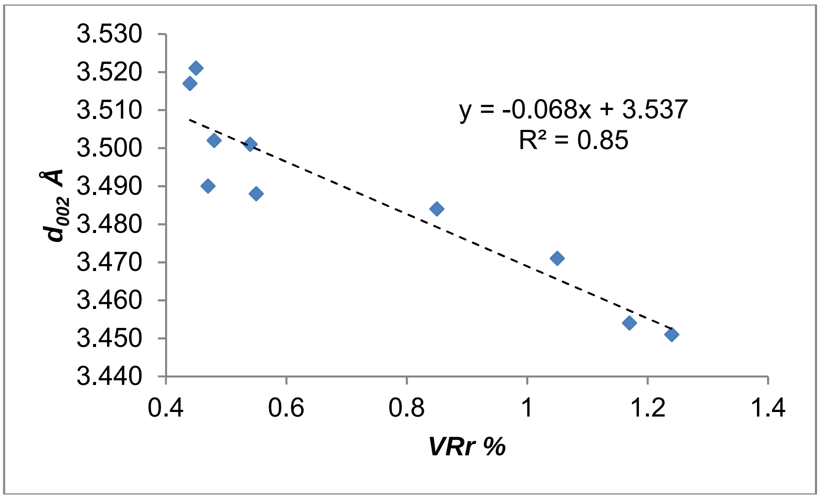

Fig. 1: Relationship d_{002} and *VR_r*.

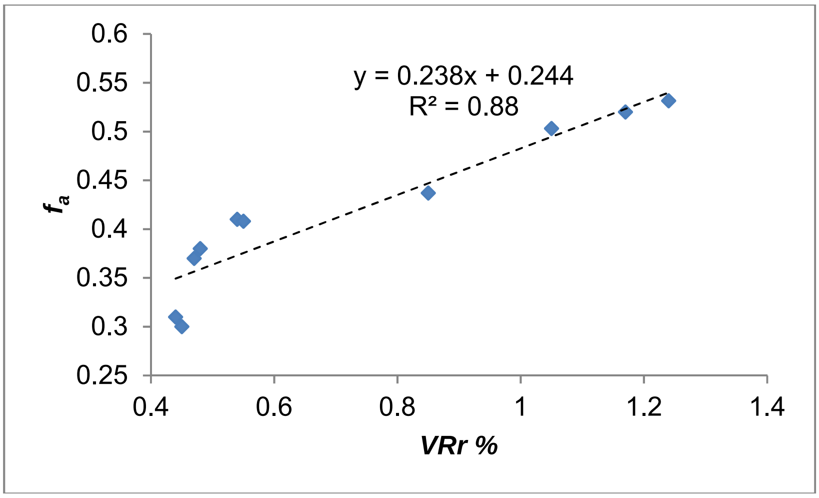

Fig. 2: Relationship between f_a and *VRr*.

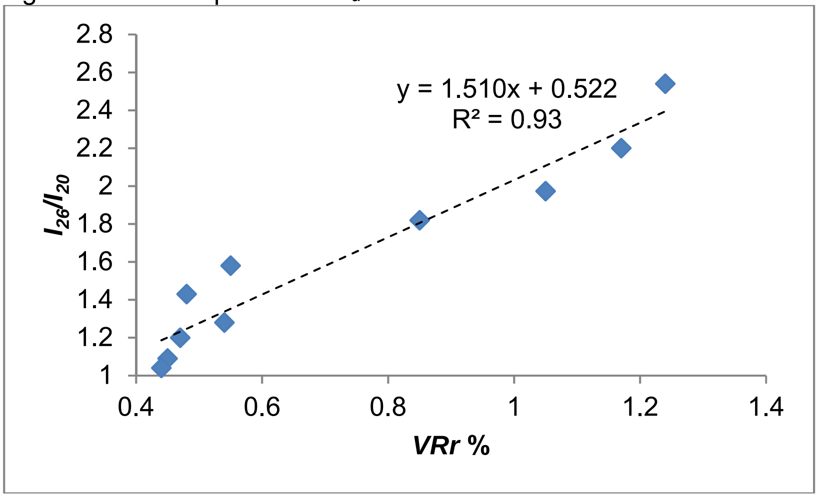

Fig. 3: Relationship between I_{26}/I_{20} and *VRr*.

Applications of Coal Grain Analysis (CGA)

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Abstract

Coal is mined, processed and used as a particulate material, therefore quantitative information about the composition of each grain is required to understand how it will respond to an external process. Reliable characterisation of the maceral and mineral associations within coal particles is fundamental for resource evaluation and for optimising recovery and usage. CSIRO, with ACARP (Australian Coal Association Research Program) support, have developed the Coal Grain Analysis (CGA) method, an optical reflected light imaging and analysis system that provides reflectance and composition information on individual coal particles.

To do so, a petrographic grain mount is prepared and calibrated high resolution images are collected of the settling plane and mosaicked together. Size and compositional information (the amount of vitrinite, inertinite, liptinite and mineral) is obtained for each individual particle and this information is used to estimate the mass, density and ash value of each particle. The particles are classified as pure components (>95% of a single component) or as dominant (<95% and >65% of a single component) or composites (mixed grains classified by the most abundant component). The system routinely analyses typical coal petrography samples, but can also provide detailed information on particle sizes up to 4mm in size. The system can also be used with a higher powered objective which enables analysis of particles down to approximately 1 micron in size. This higher resolution is required for the analysis of fine coal processing and environmental samples.

Resource Evaluation

CGA on exploration samples are typically performed at standard petrographic size (1mm topsize). This enables the washability of the sample to be estimated and benchmarks the washability characteristics (yield Vs ash%) of the exploration samples at this topsize. Ash% Vs cumulative yield curves are availble for the whole sample and for the individual grain types.Estimates of the expected yield and petrographic composition of the product coal at different target product ash values are also able to be made from the analysis of the raw coal samples. Other work has demonstrated that it is possible to predict washability by size, e.g. for up to 50mm topsize, from slimcore samples (Atkinson, 2016).

Coal Processing

CGA information enables predicted yield Vs ash curves for matched samples of flotation feed, concentrate and tailings to be constructed. Estimation of expected yield, mineral entrainment and coal loss can be made for a desired target ash value (Figure 1)

Atkinson et al. 2017, used this method for evaluating flotation performance at a Bowen Basin (Queensland, Australia) coal mine. The feed composition and recovery by coal component information was found to be invaluable in interpreting full-scale flotation. This and other studies (eg. Ofori et al., 2013) have found that CGA can facilitate real-life flotation rate constants to be applied for the purpose of modelling flotation performance based on the actual coal maceral components.

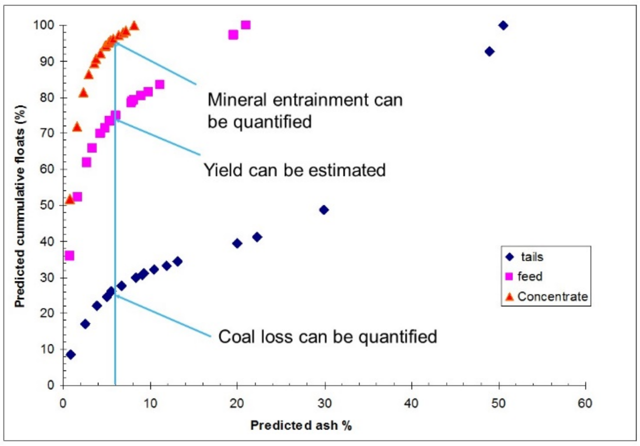

Figure 1: Predicted yield Vs ash curves constructed from CGA data for matched flotation feed, concentrate and tailings samples.

Coal Utilisation

CGA has been used to obtain information on the coal at the size it is charged into coke ovens. This requires four size fractions (+2mm, -2+1mm, -1+0.5mm and -0.5mm) to be analysed for each coke oven feed coal. The size of the coal in the coke oven feed affects physical properties of the coke, its ability to react and its strength. Mahoney et al., 2013 used CGA to determine the size distribution of single component and composite grains in coke oven feed and to link these results to coke quality. More recent developments (Warren et al., 2015) have focussed on determining the amount of fusible and infusible inertinite in coking coals.

Environmental monitoring

Coal dust is of community concern, particularly associated with the handling of coal on railways and ports in urban environments. For dust samples, size information (area, particle length, and width) and a reflectance fingerprint is obtained for each individual particle in the image. These reflectance fingerprints are then used to classify each particle and the size information obtained for each particle can be used to determine the amount of coal and non-coal material in specific size classes of (1-2.5µm, 2.5-10µm, 10-30µm and +30µm) (O'Brien et al. 2017).

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The case studies highlighted were undertaken as industry funded projects with significant input from Bruce Firth and Philip Ofori (CSIRO), Bruce Atkinson (Basacon) and Merrick Mahoney (University of Newcastle). The authors sincerely acknowledge the support provided by CSIRO and ACARP during the development and commercialisation of the CGA technology.

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Organic petrology and geochemistry of hydrocarbon inclusions in the Pennsylvanian carbonates of Picos de Europa mountains (Cantabrian Zone, N of Spain)

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Abstract

The Cantabrian Zone (CZ) of the Iberian Massif in the North of Spain comprises a thick stratigraphic succession extending from Cambrian to Carboniferous affected by thin-skinned tectonics during the Variscan Orogeny, extensional tectonics during the Mesozoic and finally Alpine compression during the Cenozoic. Although potential source and reservoir rocks are present in the succession, the lack of commercial discoveries of hydrocarbons has caused the abandonment of prospects for conventional or unconventional resources. Recent studies of the Carboniferous successions had dealt with the hydrocarbon potential of Upper Tournaisian black shales (Vegamian Formation), which showed TOC values in the range 2.8-7.4% (Borrego et al., 2018) and abundant layered bitumen, as well as a thick succession of Serpukhovian-Lower Bashkirian dark-laminated limestones (Barcaliente Formation), in which TOC values of 0.2-2.8% and oil inclusions have been detected (Borrego et al., 2016), demonstrating that hydrocarbons were generated in the area. In some areas of the CZ, thick Bashkirian–Kasimovian carbonate platform successions, which can reach more than 1000 m in thickness, stratigraphically overly the aforementioned source rocks and could had acted as potential reservoirs hosting migrated hydrocarbons. The organic petrography and geochemistry of those hydrocarbons had been investigated in the southern margin of the Picos de Europa imbricated thrust-nappe system in two sampling sites: Las Colladinas and Ándara area (López-García, 2018).

The samples collected correspond to platform-top strata of the Picos de Europa Formation and and Llacerias Formation (Bahamonde et al., 2007) and comprises ooidal to bioclastic grainstone, microbial boundstone and bioclastic wacke- to packstones deposits. Hand samples show frequent dark-colored staining, frequently concentrated along fractures and stylotites that was thought to be an indication of the bitumen content. The only type of hydrocarbon recognized is solid bitumen, found in: 1) inter-crystalline micro-porosities in the microesparite matrix; 2) intra-particle micro-porosities in ooids and bioclasts; 3) inter-crystalline porosities in sparite cements filling inter-particle, dissolution and fracture porosities and remaining open spaces, partially occluded by cements (angular bitumen); and 4) stylolites. The studied samples show similar microfacies than those of the Carboniferous platform-top reservoir rocks in Tengiz oilfield (Caspian Sea, Kazakstan; Kenter et al., 2003), nevertheless the amount of open porosity hosting migrated hydrocarbons is much lower as also shown by measured TOC values (0.02%< TOC< y 0.12 %). The hydrocarbon migration and emplacement took place during the deep late burial diagenesis of the studied samples, after most of the phases of cementation that almost completely occluded the rock porosity, and was postdated by dolomitization processes with potential Permian to Early Cretaceous age.

Three populations of bitumen have been identified in the studied samples: a lowest reflecting one showing cokefied domains which was more common in Ándara (BRr~1.1%), an intermediate reflecting population mostly found in Ándara, but also in Las Colladinas (BRr~1.8%) and a high reflectance population (BRr~2.8%) mostly found in Las Colladinas, the later also showing cokefied mosaic texture. The maturity parameters based on aromatic

compounds indicate a lower maturity for Ándara than for Las Colladinas, which is in agreement with the measured bitumen reflectance and available CAI data (Blanco-Ferrera et al., 2017).

The GC/MS (gas chromatography/mass spectrometry) study of biomarkers show evidences of biodegradation in Las Colladinas and point to an oxyc-disoxyc environment for the deposition of the parent organic matter, more oxic for Las Colladinas than for Ándara samples. Factorial and cluster statistical analyses carried out on *n*-alkane distribution using these samples plus the potential source rocks from Barcaliente and Vegamian Fms. Indicate that Ándara samples are more related to these source rocks than Las Colladinas samples.

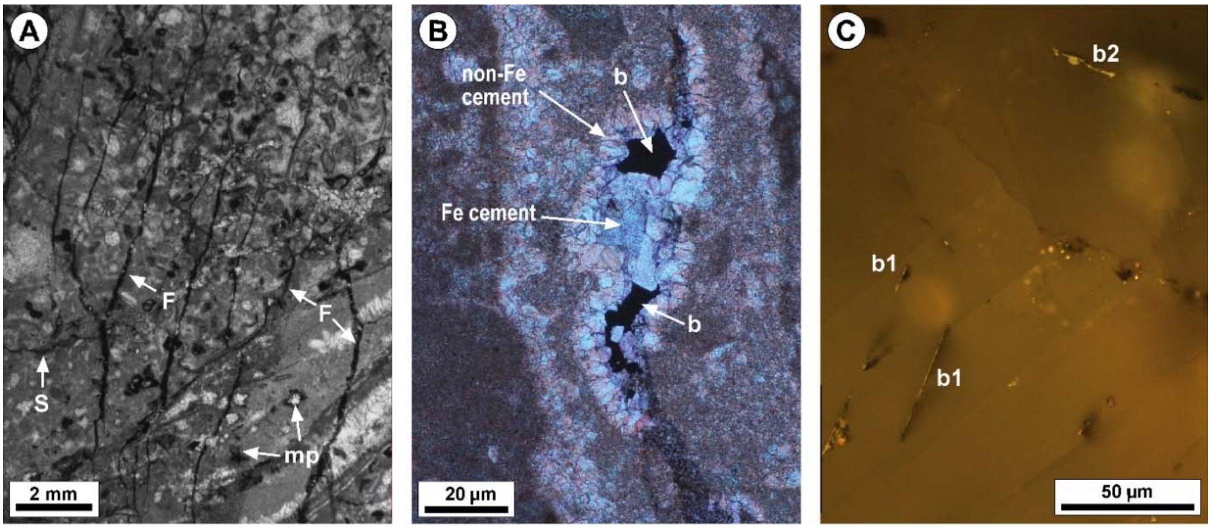

Fig. 1. Appearance of the samples through the optical microscope: A) Thin section showing bitumen inclusions in fracture systems (F), stylolites (S) and biomoldic porosities. B) Stained thin section showing bitumen inclusions (b) occluding available pore space postdating older non-ferroan calcite (pink stain) and ferroan sparitic calcite cement (pale blue stain). C) reflected white light oil immersion (b1 and b2 refer to different bitumen populations).

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Development of Very Thick Permian Coal Seams in the Cooper Basin, Australia

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Abstract

Some very thick Permian hard coal seams (30-40m) were discovered recently by deep exploration drilling for coal bed methane in the SW part of the Cooper Basin, South Australia (Figure 1). The characteristics of these thick coal seams and their tectonic and structural location has several similarities to other examples of very thick Permian coal seams in Australian and to the structural setting for the very thick Tertiary Latrobe Valley coal seams. The Cooper Basin is a petroleum basin with deep seismic data, well logs and extensive core which has been used to study the structural and depositional history of the coals.

The Cooper Basin is an intra-cratonic Late Carboniferous to Triassic basin, in which the large depocentres are dominated by NE-SW structures, with secondary NW-SE to N-S structures (Figure 1). They are strongly influenced by the basement structural trends which controlled the development of a relict glacial palaeotopography in the Late Carboniferous, when southern-central Australia was covered by an ice-sheet. The Early Permian sediments largely filled these glacial valleys with the younger Permian sediments onlapping the adjacent basement highs. In the depocentres the Permian burial was originally controlled by normal faults on their flanks with substantial sedimentation, thermal subsidence and compaction in the main troughs. These faults were overprinted by approximately NW-SE compressional stress in the Late Permian, Triassic, becoming E-W in the Cretaceous-present day, producing monoclinal folds over reverse faults and widespread erosion especially over basement related highs.

Coal measures developed in three main formations: the Patchawarra (early Permian), Epsilon (mid Permian) and Toolachee (late Permian), each separated by shale dominated formations (Figure 1). The palaeocurrent direction was from the SW to the NE along the main troughs, the Patchawarra and Nappamerri, where deposition is dominated by fluvial channel belts and floodplains. The coal seams are typically thin in the order of several metres and make up <5% of the sediment thickness. They vary in rank from sub-bituminous to semi-anthracite in the deeply buried areas. The coals consist of vitrinite with high amounts of inertinite (especially semi-fusinite) and minor liptinite (mainly sporinite and cutinite) resulting from peat development under alternating subaqueous and subaerial water table conditions (Hunt and Smyth, 1989).

Very thick coal seams are found in the Milpera Depression and Weena Trough to the SW of the Cooper Basin (Figure 1) closest to the ice-sheets. The troughs are E-W asymmetric graben in which subsidence and sedimentation was less than in the main basin centres. The net coal isopach relative to sand and shale is much higher with coal making up over 25%. The coals are also high inertinite to vitrinite coals but they have conspicuous amounts of semi-fusinite and alginite (especially *Bottryococcus* sp.). The lower burial has produced sub-bituminous to bituminous coal ranks. The high semi-fusinite content and open cleat development produce high porosity and permeability and the high alginite content boosts wet gas content.

The development of these seams can be tracked through time on the seismic, and shows that they began on the flanks of the troughs but moved out into the troughs in a series of jumps between intervening periods of non-accumulation, becoming more extensive with trough fill. The structural and depositional controls produced a combination of coal rank, thickness, extent and petrographic composition that makes these coals excellent coal-bed-methane producers.

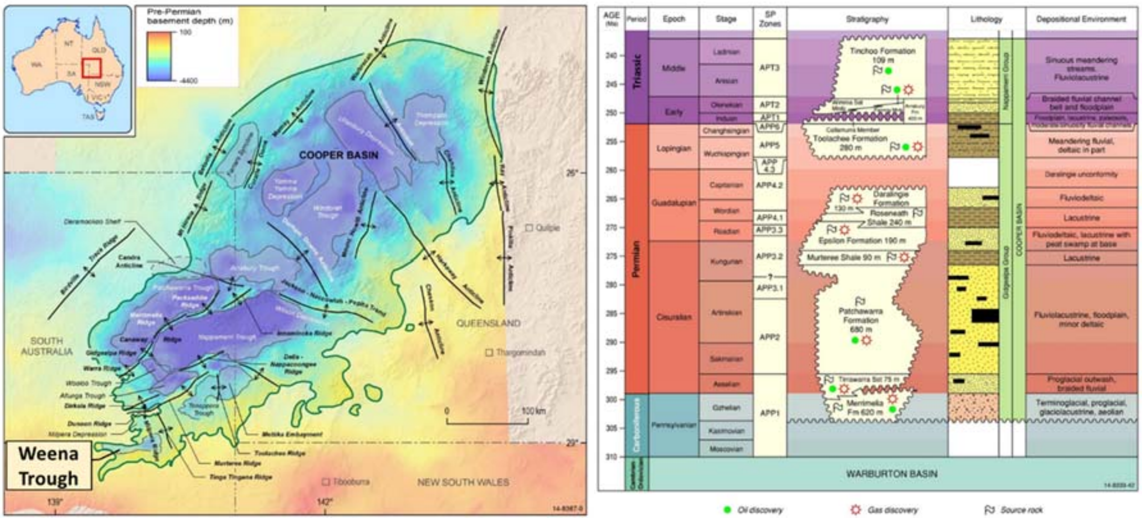

Fig. 1: Cooper Basin SW area around Weena Trough and Lithostratigraphy (Based on Hall et al., 2016)

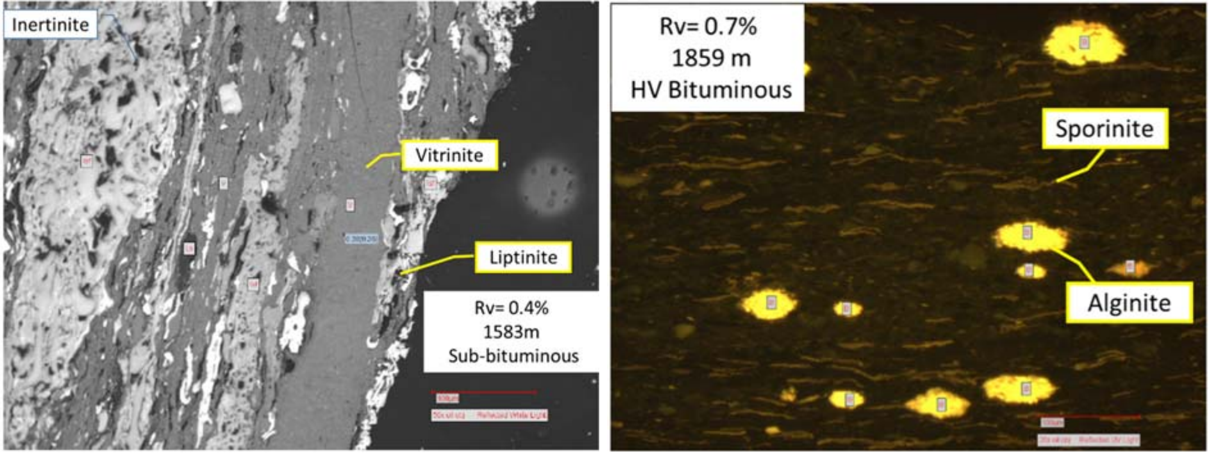

Fig. 2: Coal photomicrographs, reflected white light at 1583m (left) and fluorescent light right at 1859m (right) from Le Chiffre 1 Well Completion Report, Petrographic Report (E. Barcelona, 2014)

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Abstracts
for Poster presentations

An appetite for apatite: A micro-analysis review

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Abstract

Phosphorus (P) and fluorine (F) are undesirable trace elements within coal. Elevated P and F concentrations reduce the economic viability of coal deposits and generate potential environmental issues during utilisation. Increased P contents in coking coal can make hot metal brittle, while the F in thermal coals is readily released during combustion, corroding the boiler and posing a risk to the environment and human health. The minerals that host these elements often occur as small (<20 μ), discrete inorganic minerals such as fluorapatite [Ca5(PO4)3F] within the coal particles as described by Ward (2016) and so can occur across different size fractions and densities (Partridge et al., 1992). Therefore, societal tolerance levels for these elements are reducing, requiring methods to predict and mitigate their occurrence through selective mining or novel beneficiation for sustainable coal utilisation.

A number of potential mechanisms identified as influencing the origin of mineral matter within coal, such as: source-rock, marine influences, hydrothermal fluids, groundwater and volcanic ash will be tested (Dai et al., 2012a; Davis, A. et al., 1984). As part of a larger project, a systematic analysis has been undertaken of the stratigraphic and spatial occurrence of P and F and associated mineral matter in coal in the Bowen Basin, Central Queensland. Samples were collected from (youngest to oldest) Rangal (RCM), Fort Cooper (FCCM) and Moranbah coal measures (MCM) to test for stratigraphic change within the basin.

Through scanning electron microscopy with energy dispersive X-Ray spectrometry (SEM-EDS) multiple phosphate minerals were observed, including: apatite and crandallite [CaAl3(PO4)(PO3OH)OH6]. A stratigraphic variation in mode-of-occurrence between the coal measures is shown in figure 1. The RCM are dominated by cell- and fracture-infilling strontium-fluorapatite grains, while the MCM and FCCM are dominated by detrital-grain-supported chlorine-fluorapatite grains (EDS spectrum not presented here). The cell-infilling apatite is thought to reflect either syngenetic or epigenetic mineralisation; while the fracture-infilling apatite formed later, post coalification (Ward, 2016). This is interpreted to have formed via fluids migrating through permeable and porous pathways created during geological deformation. The mode-of-occurrence of fluorapatite grains hosted within the FCCM tended to be attrital and show similar chemical patterns to fluoapatite isolated from tuffs within the coal measures, inferring a volcanic origin. However, to try to constrain and fingerprint the fluid source, emplacement mechanisms and timing, further work is required.

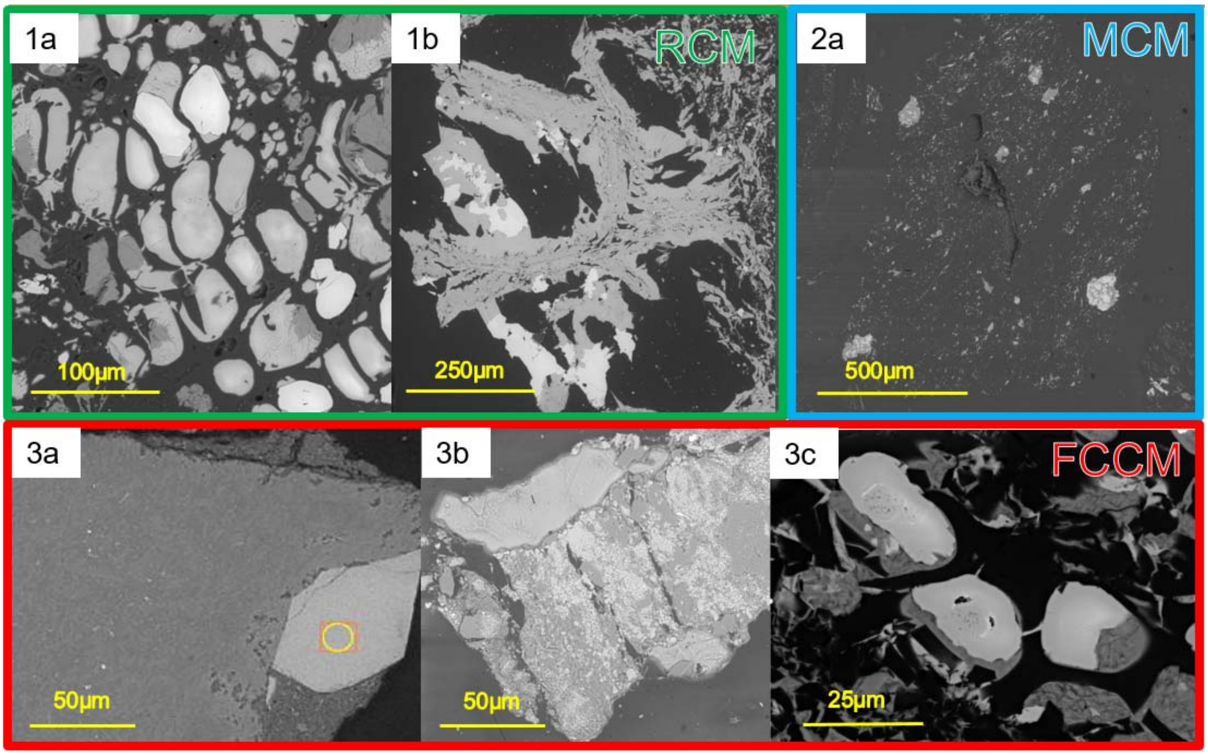

Fig. 1: Scanning electron microscope backscatter micrographs of fluorapatite grains 1a and b Rangal Coal Measures captured using the JEOL6610, figure 2a Moranbah Coal Measures and figures 3a, b and c Fort Cooper Coal Measures captured using the HITATCHI TM3030Plus.

Acknowledgements:
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Analysis of matched coal and coke samples to determine mineral impacts on coke structures

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Abstract

During the coking of coals of suitable rank, the organic material goes through a transformation whereby the vitrinite, liptinite and some of the inertinite material fuses while the remaining inertinite material does not. During this process some of the mineral grains in the parent coal may react and adversely affect the coke matrix surrounding it. To quantify the effect of mineral grains on coke quality during the coking process 20mm diameter cylinders were first prepared from mineral rich coal lumps. The cylinders were then sectioned along the longitudinal axis and one half was coked in a laboratory scale furnace. The matching coal and coke halves were mounted in polyester resin and polished.

The coal half was analysed using CSIRO's Coal Grain Analysis (CGA) optical reflected light microscopy imaging system to determine the reflectance distribution of the coal surface. The coal and coke surfaces were further analysed using a scanning electron microscope (SEM) to identify and obtain size detail for the individual mineral grains in the matched coal and coke halves.

This process has provided valuable information on the mineral transformations that have occurred during the coke making process for individual mineral grains and also allowed mineral/maceral associations in the parent coal to be correlated with their corresponding mineral/matrix associations in the daughter coke.

Methodology

The matching coal and coke halves prepared from 20mm diameter cylinders (mineral rich coal lumps) were mounted in polyester resin and polished using the method described in Warren et al., 2015. The coal half was analysed using CSIRO's Coal Grain Analysis (CGA) optical reflected light microscopy imaging system to determine the reflectance distribution of the coal surface (Figure 1). The coke half was analysed by Pearson Coal Petrography (Pearson's et al. 2012) to produce a map of the fused and non-fused carbon on the surface of the coke lumps (Figure 1(b). The organic structures and the minerals in the coal halves were matched with their corresponding coke matrix structures and minerals in the coke halves (Figure 1(c)).

The coal and coke surfaces were further analysed using a scanning electron microscope (SEM) to identify and obtain size detail for the individual mineral grains in the matched coal and coke halves (Figure 2).

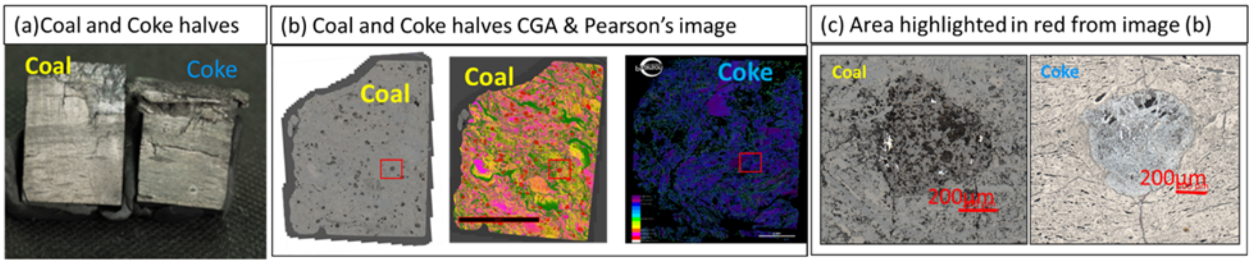

Fig 1: (a) Matched surfaces from Coal and Coke halves (lab scale coal and coke faces),

(b) Original and characterized image of coal & Pearson coke imaging of coke image (C) enlarged area highlighted in red from image (b) showing the progression of a single mineral grain through the coking process.

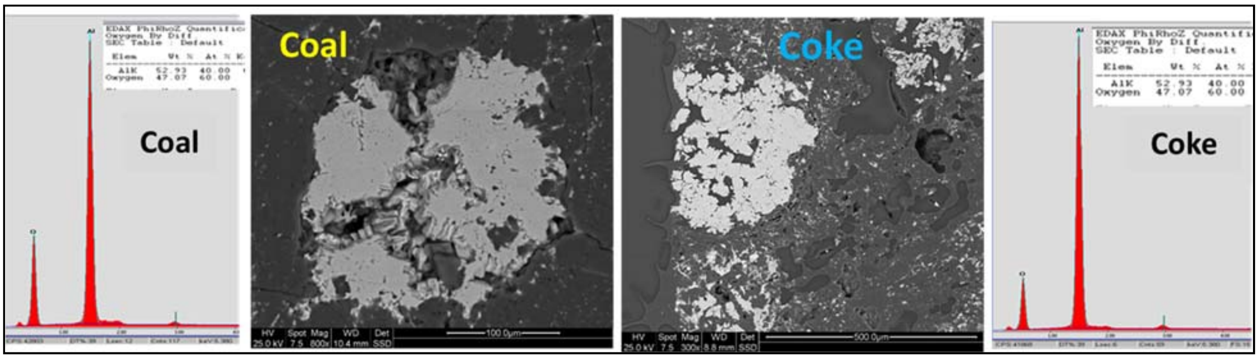

Fig 2: Photomicrographs and spectra of aluminium oxide transformation during coking process collected using SEM.

Results

These results indicated that the size of the mineral grains in the coke matrix and the compositional transformation of minerals during the coking process is dependent on the chemical composition of the original minerals and their associations with the organic material within the parent coal. This process has provided valuable information on mineral transformations that have occurred during the coke making process for individual mineral grains and also allowed mineral/maceral associations in the parent coal to be correlated with their corresponding mineral/matrix associations in the daughter coke.

Acknowledgements:

The authors sincerely acknowledge the support provided by CSIRO and ACARP during the development and commercialization of the CGA technology. They would also like to thank JKMRC, University of Queensland for providing the access to analyse samples using their SEM/MLA system.

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Analyzing the efficacy of using T_{max} values to estimate R_o in the eastern onshore Gulf Coast Basin

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Abstract:

A newly released U.S. Geological Survey (USGS) database has been employed to investigate the relationship of vitrinite reflectance (Ro) and the temperature of maximum pyrolysis output (T_{max}) to generate an Upper Cretaceous Tuscaloosa marine shale (TMS) specific equation for calculating thermal maturity.The dataset was generated in support of the USGS assessment of undiscovered petroleum resources in the Upper Cretaceous Tuscaloosa marine shale, and evaluation of shale gas prospectivity in the Aptian section of the Mississippi Interior Salt Basin. Five hundred sixty-one samples of rock core and cuttings from wells drilled in the region were collected and examined over the course of six years using a variety of geochemical analyses (Enomoto et al., 2018). These analyses included vitrinite reflectance (R_o), programmed pyrolysis, and semi-quantitative X-ray diffraction. The database containing the results of these analyses can be found on the USGS ScienceBase website (https://doi.org/10.5066/F7B27TJ5). In this study, we use data from this repository to investigate the relationship of R_o and the temperature of maximum hydrocarbon output during pyrolysis (T_{max}), and generate a TMS-specific equation to calculate thermal maturity.

Vitrinite reflectance and T_{max} are commonly utilized in assessing the thermal maturity within a play (Tissot and Welte,1984), and can be used in conjunction with other analyses to determine hydrocarbon reserves in a region, or the optimal location and depth for oil and gas extraction in a play. Based on empirical relationships between R_o and T_{max} (Tissot et al., 1987), T_{max} data can be used, in some circumstances, to estimate R_o values (Jarvie et al., 2001; Laughrey, 2014; Wüst et al., 2013). However, as shown in Wüst et al. (2013), R_o-T_{max} regressions are not universally applicable to all shale plays. Factors such as oxidation, non-indigenous vitrinite, and kerogen type can affect the accuracy of the T_{max} vs. R_o curve (Jarvie et al., 2007). Additional analysis of the relationship between R_o and T_{max} in shales containing a wide spectrum of kerogen types, thermal maturity, and levels of TOC is nessessary to more accurately estimate the thermal maturity of shales when utilizing T_{max} values to estimate R_o.

In this study, we compared the R_o vs. T_{max} curve generated from data in Enomoto et al. (2018) to previously developed equations. To determine the linear regression equation for thermal maturity in the TMS, the data in Enomoto et al. (2018) was constrained to values where the remaining hydrocarbon potential (S2) was > 0.5 (Peters, 1986) and the T_{max} data came from a single source (Jarvie, 2007). Unreliable R_o and T_{max} data, as annotated in the sample comment section of the database, and outliers were removed. The resulting equation is $R_o(\%) = 0.014(T_{max}) - 5.4776$, where n = 26 and $R^2 = 0.69$.

Compared with Jarvie's (2001) equation of $R_o = 0.0180(T_{max}) - 7.16$ (n = 179, $R^2 = 0.79$); Laughrey's (2014) equation of $R_o(\%) = (0.01867 \pm 0.00063)(T_{max}) - (7.306 \pm 0.284)$; and Wüst's (2013) equation of $R_o(\%) = 0.0149(T_{max}) - 5.85$ (n = 20, R2 = 0.71); the curve generated by utilizing Enomoto et al.'s (2018) database is closest to Wüst's equation. Wüst's equation used reflectance measurements from solid bitumen (BR_o) in the Devonian Duvernay Formation in western Canada, and converted BR_o to R_o using an equation from Jacob (1989) in which $R_{o\ equivalent} = (BR_o * 0.618) + 0.4$. Jarvie's equation may also be based in part on BR_o judging from the Barnett Shale samples measured, where solid bitumen dominates the organic assemblage. Conversely, our study used measured reflectance of unambiguous vitrinite.

The difference in the Jarvie and Wüst curves appears to coincide with the understood model of vitrinite and solid bitumen reflectance. Reflectance of these macerals increases with increasing T_{max}; however, at lower maturities, vitrinite has a higher reflectance than solid bitumen. As thermal maturity increases through the oil window, solid bitumen reflectance increases faster than vitrinite reflectance. As T_{max} values approach 350 °C, vitrinite reflectance and solid bitumen reflectance measurements begin to converge (Hackley and Lewan, 2018). Reflectance determined from the Jarvie equation ($0.0180*T_{max}$), which we presume is based primarily on solid bitumen reflectance, increases faster than the converted vitrinite reflectance of Wüst's equation ($0.0149*T_{max}$), and the vitrinite-only reflectance of the equation in our study ($0.014*T_{max}$).

This analysis shows that it is critical to understand the maceral type, vitrinite or solid bitumen, used to generate R_o-T_{max} regressions, and reaffirms the observation of Wüst et al. (2013) that an evaluation of the appropriateness of the specific maceral measured is required before such regressions are applied to individual shale formations. To better understand the relationships between R_o and T_{max}, and to continue improving the accuracy of thermal maturity regression equations using these parameters, additional formations will need to be analyzed utilizing petrographic techniques and programmed pyrolysis.

Acknowledgements:

I would like to thank the USGS Gulf Coast Petroleum Systems Project for the production of the aforementioned Mesozoic rock sample database, which facilitated this study. Thanks to Brett Valentine for help conceptualizing the idea for this thermal maturity study.

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Assessing thermal maturation of Lemede Formation (Alcobaça-1 well), Lusitanian Basin, Portugal: vitrinite and solid bitumen reflectance

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Abstract

Vitrinite reflectance is an important petrographic parameter used to determine the maturation of dispersed organic matter (OM) in sedimentary rocks (Tissot and Welte, 1984). In samples lacking vitrinite, the solid bitumen reflectance can also be used as a maturity parameter. In recent years, several studies have been carried out in order to identify and characterize the OM present in Jurassic sediments of the Lusitanian Basin (Portugal) however there are still few studies related to vitrinite and/or solid bitumen reflectance data of this basin. This work was carried out in order to evaluate the origin of the dispersed OM of Lemede Formation (Alcobaça-1 well, Lusitanian Basin, Portugal) and their thermal maturation. Therefore, seven cutting samples (1516 to 1566m depth) were collected and submitted to organic petrography and geochemical studies. The Pleinsbachian to Lower Toarcian of the Lemede Formation consists of alternation of marl (centimeter scale) and micritic limestone (decimeter scale), very rich in belemnites, ammonites, bivalves and brachiopods. The sedimentation occurred in a marine environment under regressive conditions (2nd-order regressive phase) (Duarte et al., 2010).

Total organic carbon (TOC) and Total sulfur analyses were carried out using a LECO© analyzer SC144. Whole rock blocks were prepared according to ASTM D2797-11a standard. Vitrinite and solid bitumen reflectance were measured following ASTM D7708-14 standard using a Leica DM4000 microscope equipped with a Discus-Fossil system using a 50x oil immersion objective. The microscope was calibrated with a YAG standard (0.905% Rr) and an optical black (zero).

Geochemical data indicate low organic carbon content with TOC values varying from 0.47% to 0.61 wt.%. Total sulfur content varies between 0.35 and 0.49 wt.%. Carbonate content range from 59 to 79%, with an increase in the siliciclastic fraction to the top. Microscopic analysis reveals that the OM is composed of the three main maceral groups plus solid bitumen (dominant) and zooclasts. The most represented maceral group is liptinite, mainly bituminite and alginite (mostly lamalginite - dinoflagellate cysts and acritarch). Bituminite and alginite reveal fluorescence (brownish orange and intense yellow, respectively). Terrestrial OM as vitrinite and inertinite (fusinite, semifusinite and macrinite) were also observed. The vitrinite is small in size and shows a mean random reflectance of 0.61% (mean value). Four families of solid bitumen were observed with different petrographic features. The mean random reflectance (Rr) of these bitumens varied between: i) 0.54 to 0.61% for family B1; ii) 0.79 to

0.95% for family B2; iii) 1.29 to 1.65% for family B3 (more abundant); and iv) 2.03 to 2.15% for family B4.

The presence of liptinite and terrestrial material indicative a mixture of type II-III kerogen. Results of vitrinite reflectance suggest that the samples are mature in relation to hydrocarbon generation. The presence of several families of solid bitumens was expected since this trend is

common in the Lusitanian Basin. Based only on %Rr data (Jacob, 1989), the solid bitumens identified in Lemede samples are classified as grahamite and impsonite (epi- and meso-impsonite), respectively. No relation has been established with confidence for the reflectance of bitumens with the reflectance of vitrinite.

Acknowledgements:

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Behaviour of coal matter from the Žacléř Basin during thermal changes in burnt coal waste heap and in laboratory conditions

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Abstract

Spontaneous combustion of coal waste is a source of long-term discussion of the factors that cause them and of the nature of the products that are produced by this complicated process. For these reasons, a diverse spectrum of solid products generated during spontaneous burning processes in coal waste heap was compared with products of experimental thermal alteration of original coal from the seam of the Žacléř Basin. This coal represented high volatile bituminous matter with vitrinite reflectance around 0.70%, and vitrinite macerals prevailing over liptinite and inertinite macerals.

Destructive changes of organic matter are strongly affected by mineralization, humidity, radioactive alteration, burning temperature and mainly by free vs. restricted access of air, as revealed by complex analyses of gaseous, liquid and solid products after experimental heating of the coal under inert and oxidizing atmosphere at temperatures in the range 200-900^{o}C.

At the temperature 200^{o}C, a minimum alteration of organic matter occurred, being accompanied by a release of water from coal and a negligible development of gasses under both atmospheres. Prominent changes in organic matter appeared at 500^{o}C: altered coal and more porous char were found in solid residues, disrupted by fractures and pores during very strong degassing of the liptinite macerals. The effect of atmosphere revealed at this temperature: solid products generated under the oxidizing atmosphere were dominated by char with a reflectance max. 4.5%. Under inert atmosphere altered coal with reflectance of max. 1.5% prevailed over char with the reflectance of 3.5%. In the solvent extracts of the solid products, alkanes (7–100 rel.%) were identified together with aromatic hydrocarbons: phenanthrene and methyl substituted phenanthrenes were dominant in all extracts, together with other polycyclic aromatic compounds containing four, five and six aromatic rings (pyrene, benz(a)anthracene, chrysene, fluoranthene, benzo(c)phenanthrene, triphenylene, benzofluoranthenes). The solid products prepared under oxidizing condition differed in presence of oxygen containing compounds in extracts but their content is not very distinctive. At the temperature 900^{o}C under access of air, the proportion of solid products decreased by about 25%. On the contrary, an increase in the proportion of less porous and fragmented char was observed. The extract contained only n-alkanes in the range n-C15-30.

Generally, the increasing temperature used under both atmospheres was associated with: 1) an increase in average reflectance of coke walls up to 6.05%, 2) an increase in structural ordering of organic matter that was documented by: a) the evolution of a mosaic anisotropic texture and a decrease in the values of the "half width" parameter (FWHM, cm-1) in Raman spectrometry from 280 cm-1 at 200^{o}C to 180 cm-1 at 900^{o}C, b) a decrease in coke extractability, c) a simplification in the compound representation in coke extracts.
The experimental study confirmed that the variability of samples collected from coal wastes is caused by different petrographic and chemical composition of the deposited coals and by variations in oxidative and thermal conditions within coal waste heaps.

In all thermally altered samples collected from coal waste, organic matter was disseminated within the various mineral matter, forming isolated particles of isotropic and anisotropic cokes with reflectance between 4.50 and 8.15%, variable altered coal matter with vitrinite reflectance over 1.10%. Two types of hydrocarbon condensates (tars) were abundant in the surface of burnt material. The solid particles of hydrocarbon condensates with reflectance around 1.05% were found frequently in the surface zone of heap and were much more abundant than the semi-solid particles with reflectance around 0.30%. Porous and massive highly reflecting cokes can be considered solid residues of local high temperature (~1000^{o}C) burning with limited air access. Less reflective cokes and altered coal matter indicate a drop in the temperature and a reduced oxygen supply.

The extracts of samples, collected from the waste heaps, contented various n-alkanes and atypical isoprenoid hydrocarbons, substituted aromatic compounds, and heterocyclic compounds. The compound distributions differed according to the sampling depth and position, reflecting the different burning/smouldering conditions in the heap. Basic fingerprints of the analytical record obtained for laboratory and natural samples were in good agreement. The degree of graphitization in the collected samples, using the “half width” parameter of Raman spectra, revealed the values of FWHM 200 -100 cm-1, and also showed approaching to the values of samples prepared in laboratory conditions.

Acknowledgements:
This study was carried out within the Czech Science Foundation grant project “A model of mobilization and geochemical cycles of potentially hazardous elements and organic compounds in burnt coal heaps” (GACR 15-11674S panel P210). Our thanks to the Operational Program Prague - Competitiveness, project “Centre for Texture Analysis” (No. CZ.2.16/3.1.00/21538), and the long-term conceptual development of the research organization RVO: 67985891.

Changes in organic petrography, stable carbon isotopes, and geochemistry in experimentally-heated bituminous coals of variable rank (Upper Silesian Coal Basin, Poland)

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Abstract

Coals in the Upper Silesian Coal Basin differ in rank and petrographic properties, and in their propensity for self-heating. Coalification increases with stratigraphic depth and towards the western part of the basin. The average vitrinite random reflectance R_r ranges from 0.56-2.00% with depth; the gradient is 0.2%/km.

The main aim of the study was to identify any trends in the petrography, and in the isotope- and organic geochemistry, of typical raw bituminous coals of different rank from seams throughout the Upper Silesian Coal Basin. This study involved samples of different rank from seams representative of all stratigraphic coal-bearing sequences of Carboniferous age in the basin, i.e., from the latest Mississippian mudstone paralic series and Pennsylvanian limnic sandstone- and mudstone series. A further aim was to compare these coals with coals heated to <400°C in open- and closed experimental environments.

Seven samples of bituminous coal differing in ranks were collected *in situ* from four underground coal mines, namely, Marcel, Ziemowit, Wujek, and Piekary. The samples were heated in the laboratory to 400°C in an open system for 24 hours and in a closed system for 2 hours. The heating rate in the closed system was 20°C/min whereas, in the open system, the samples were placed into an oven pre-heated to 400°C. The petrographic and geochemical properties, and the isotope compositions, were determined for both raw- and heated samples.

All three maceral groups are present in the raw samples. The vitrinite maceral group is typically the most common group, followed by inertinite macerals. Only in one sample from the Marcel mine is inertinite the dominant maceral group. Liptinite macerals are the least common. Minerals usually amount to <8% though, in one sample, it is 21.4%. The coal rank varies from 0.56-0.85%.

The heated coal samples show a great variety of morphological forms reflecting the conditions of heating. They are dominated by pale-coloured particles. Irregular cracks are common within the particles and at their edges. The presence of devolatilization pores likely occurring in place of liptinite macerals reflect high experimental heating rates. In many cases, relatively pale oxidation rims characterize the external edges of particles, cracks and pores. Inertinite represented mostly by fusinite, semifusinite, inertodetrinite is typical in the paler particles. In comparison to the fresh coals, the reflectance (R_r) of the altered coals (~2%) is much higher.

Extract yields were variable, from 1.5-3.8 wt% for raw coals, very low ca 0.05 wt% for samples heated in the open system, and higher (< 6.2 wt%), in the case of samples heated in the closed system. In the open system, bitumen present in the coals formed due to the heating and was freely expelled and evaporated whereas, in the closed system, pyrolysate formed which was adsorbed within the rock; the rock extractability was increased.

The GC-MS results on bituminous coals point to the crucial role played by the heating environment (Misz-Kennan and Fabiańska, 2011). Open-system heating, with free access of

oxygen and free evaporation of organic compounds, led to almost complete destruction of bitumen, and very low extract yields. In these conditions, all light-weight compounds evaporated or were oxidized. Residual bitumen where present was composed mostly of heavy PAHs and their oxidized products. Closed-system heating with depleted oxygen availability increased extract yields to values comparable of raw rocks and coals. The closed experimental conditions resulted in the expulsion of bitumen from closed pores, and possibly also partial pyrolysis of organic matter. *n*-Alkane distribution outlines are of Gaussian type without odd-over-even carbon atom predominance. However, typical pyrolytical products such as *n*-alken-1-es were not found in the extracts indicating that temperatures were too low or heating times were too short to cause advanced macromolecule cracking. Most of lighter compounds preserving initial distributions and ratios are still present in the extracts. The increase in *n*-alkane concentrations over pristane, phytane and aromatic hydrocarbons is the most prominent compositional change in these samples.

The content of ^{13}C tends to increase with increasing rank in the raw coals, however, other factors are also controlling it.. The heating experiments revealed almost the same isotope composition of the raw coals and the samples heated in the closed system; differences range from 0.03-0.45‰, as similarly described by Rahman et al. (2017) for natural coals. Significant differences ranging from 0.74-9.79‰ were demonstrated for samples heated in the open system. In these cases, some extracted/desorbed carbon species (methane/carbon dioxide/hydrocarbons) depleted in ^{13}C in comparison to raw coal material probably escaped during the heating procedure and ended up enriching residual thermally changed coals in ^{13}C.

When the system is closed, there are no changes in the isotopic composition as no components are carried away despite the changes in petrographic and geochemical forms. In the open system, the isotopic compositions of all samples significantly shifted towards isotopically heavier values; in the case of two samples, the shift was 10 times greater than in the others.

Acknowledgements:

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Detecting and quantifying urban dust particulates using CGA

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Abstract

In urban environments airborne particulates (dust) must be managed to ensure that industry and community coexist in a mutually beneficial and sustainable manner. Urban dust particulates can come from a wide range of sources and exhibit a range of particle sizes as a function of the particulate source and environmental conditions such as wind source and direction. As Australia is one of the world’s major exporters of metallurgical and thermal coals; coal dust is a significant environmental and social responsibility issue for the coal industry. Whilst coal dust is a significant environmental and social responsibility issue for the coal industry at all parts of the coal chain, it is where mines are in close proximity to towns and in urban and industrial environments, such as along rail corridors and at ports, that public concern with coal dust is greatest.

In general, there is a view by many community members that a significant proportion of inhalable (PM10) and respirable (PM2.5) dust in these environments could be coal. Thus there is a need to have an analytical method that provides a quantitative analysis of the amount and size distribution of the different particulates that can be present in air samples. It is, moreover, important to provide this information in a way that is acceptable to all stake holders: community, government and industry.

Optical reflected microscopy is a well-established manual method for obtaining quantitative information about the abundance of the different particulates in an urban dust sample but it does not generally provide size detail for the individual particulates. CSIRO have developed a semi-automated imaging method, Coal Grain Analysis (CGA) with collects and mosaics multiple high resolution images to provide size detail on each particle greater than 1 μm. This technique has been applied to the analysis of particles in urban dust samples. This application of CGA builds on the approach used by Diessel to provide quantitative detail of coal and non-coal particles in urban dust samples.

To undertake this work required an understanding of the types of particles that could be found in urban dust samples. CSIRO collected, prepared and imaged a suite of approximately 40 different types of particulates which could be present in urban dust samples and obtained their reflectance fingerprints. This reference library was used initially by a coal petrographer as a guide for dust characterisation and dust samples were categorised manually. A supervised learning algorithm, called Optical Dust Markers (ODM), was created to assist the coal petrographer/expert user for dust analysis. ODM uses the individual particles colour reflectance fingerprint to classify each particle and hence provide size information for each particle class. These markers can currently identify coal, combustion chars, iron fragments, quartz/dark minerals, pyrite/bright materials, soot and particulates of organic origin. Expansion of the dust reference library is vital for enhancing the capabilities of CGA and a better precision of analysis. To undertake a case study around the port of Newcastle, an additional set of 53 reference samples collected by NSW EPA (New South Wales Environmental Protection Agency) were prepared and imaged, and these reference particles were added to our reference library.

The CGA ODM module was used to evaluate the proportion of coal and other dust particulates, and their size distributions, in samples collected in an urban area near the Newcastle coal port. Particles in the analyzed samples were divided into four size classes based on their equivalent diameter, and the amount of each particle class, in each size fraction is reported. These size fractions are:

- nuisance particles >30µm;
- inhalable - particles in the range from 10µm to 30µm;
- thoracic - particles in the range from 2.5µm to 10µm and
- respirable - particles 1µm to 2.5µm.

The respirable size class has a lower limit of 1µm as this is the resolution limitation of the CGA technique. The projected area diameter of a circular particle with the same area as the 2D projection of the investigated non-circular particle was used as the equivalent diameter for each particle. The particle volume was computed from the equivalent diameter assuming the particles were spherical. This approximation is reasonable given the observed shapes of most of the particles.

Results can be viewed using web-based image viewing software, which displays the individual particles pre and post classification and gives size detail for any selected particle in the sample. The links below provide show the results for two of the samples from the Newcastle study.

https://cloudimaging.csiro.au/Sample/Viewer/bd54f52c-3949-4552-b5d8-6a69e07c5145

https://cloudimaging.csiro.au/Sample/Viewer/e2e0ee06-77f2-4e09-b201-38d181aac17c

Environmental impact of coal handling on the coastal area of Gijon (Northern Spain): A petrographic approach.

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Abstract

Organic petrography contributes to the environmental pollution studies through e.g., identification of organic particulates derived from activities related to coal such as coal mining, preparation, transport, blending, management and shipment, storage and utilization, and by making of coke, coal-tar, pitch, manufactured gas plants and coal gas, among others (Suárez-Ruiz et al., 2012). Taking into account this, the objective of the present work is to determine via petrographic analysis the environmental impact of coal handling (including loading, shipment and downloading of coal and coke from vessels at the seaport), transport, storage and utilization, on the coastal area (mainly at the San Lorenzo beach, Cimadevilla, and La Calzada, as the most polluted part of Gijon City).

Gijon City is surrounded and therefore affected by a coal power plant, a coal park, a cement industry using coal as fuel, a steel industry with coke plants, and specially by the activities developed at the El Musel commercial seaport that manages around 7 million tons of coal and coke per year (Port Authority of Gijon, 2018). Preliminary results shows that the San Lorenzo Beach, and depending on the tides and gales, periodically shows a contamination by coal, coke and char particulates in different percentages but with a predominating particle size in the range 0.5-1.0 mm, although in some areas of the beach rounded fragments of coals of higher size were also found (~2-4 cm). At the buildings from the Cimadevilla and La Calzada areas (within the Gijon City) the pollution is mainly due to airborne particles of coal, char and coke (with a size ranging from 40 to 60 microns) and other inorganic components. In all cases the coal particles are blends of coals ranging from high volatile bituminous coal to anthracite and meta-anthracite coal rank representing the activities developed by the different industries and facilities using coal.

The importance of coal particles (and other organic particulates) in the environment derives from the existing relationships between these particles and the concentration of the PCBs, PAHs, and PCDD/Fs organic pollutants due to the sorption properties of organic particulates. Moreover, it is well-known that the coal and residues of coal utilization (such as chars and coke particles) also contain hazardous trace elements (such as Hg, As, V, Se, etc.).

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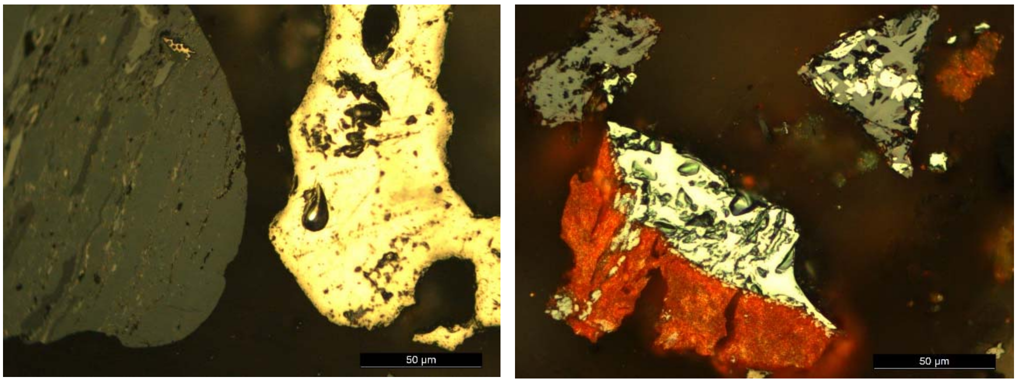

Fig.1: Optical microscopy images of some organic (coal) and inorganic particles polluting the San Lorenzo Beach (left side) and the windows of the Gijon buildings (right side). Long side of the images is about ~200 microns

Evidences of organic matter reworking across the Cenomanian-Turonian boundary of the Subalpine Basin (SE France)

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Abstract

During the Cenomanian-Turonian boundary (CTB) the Subalpine Basin developed as a western gulf in the European Alpine realm of the northern Tethys Ocean, located at a paleolatitude of 25º- 30ºN (Vocontian Trough) (Grosheny et al., 2006). This paleogeography was later deformed by the sinistral play of strike-slip faults (Nîmes and Durance faults) during the Late Cretaceous to Cenozoic closing of the basin and concomitant opening of the Bay of Biscay (Pyrenean Orogeny), and early Alpine folding phases (Grosheny et al., 2017). The CTB is characterized by the deposition of organic-rich sediments that constitute one of the best developed on-land expressions of the Oceanic Anoxic Event 2 (Jarvis et al., 2011). With these premises, the objective of this study is to describe the presence of reworked organic matter in the Cenomanian-Turonian sedimentary succession of the Vocontian Trough, Subalpine Basin.

The analysis of these samples revealed the occurrence of reworked organic matter, most significantly, Graptolite fragments (middle Cambrian-Carboniferous; Fig.1A), Chitinozoans (lowest Ordovician-lowest Carboniferous; Fig.1B) and *Navifusa* ssp. (Devonian acritarch; Fig.1C). The Graptolite debris exhibit a dark brown color under transmitted white light (TWL) and no fluorescence, being their identification possible by the display of the characteristic curving interthecal septa. Chitinozoa specimens display vesicle shape and aperture, sometimes with the presence of appendices, with dark brown to black color under TWL and no fluorescence. *Navifusa* ssp. is characterized by elongated naviform vesicles with rounded ends, no polar processes, and no surface ornamentation. These acritarchs present light brown color under TWL and yellow to brown fluorescence. Other reworked particles include sporomorphs, cuticles (Fig.1D), Prasinophytes and Salviniaceae (massulae of freshwater spore fern) that display dark brown to black color under TWL and no or brown fluorescence. The presence of round-shaped particles in plugs of kerogen isolate, under reflected white light, was also identified.

This organic matter association suggests that the majority of the reworked material dates from the Paleozoic, possibly Silurian-Carboniferous. Therefore, this study presents the first description of the presence of reworked Paleozoic organic matter in the Cenomanian-Turonian sedimentary record of the Vocontian Trough, Subalpine Basin.

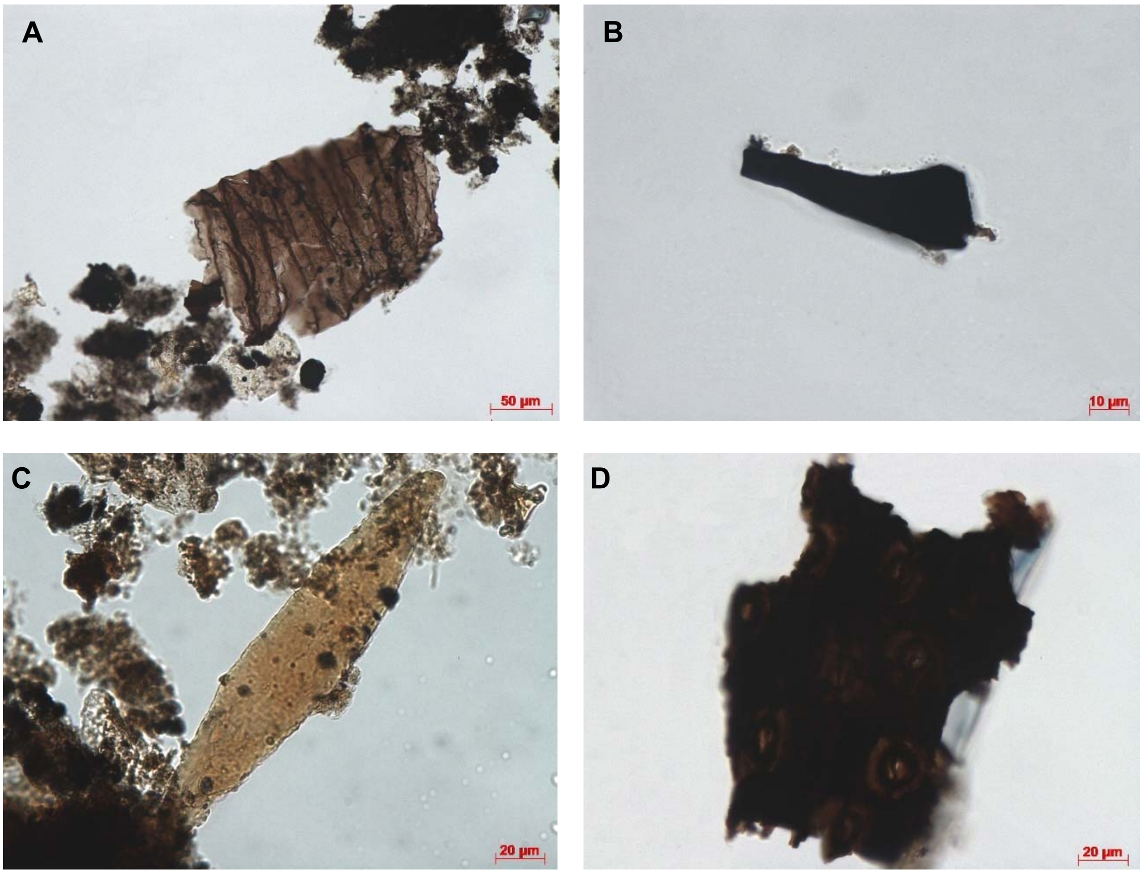

Fig. 1: Photomicrographs of reworked organic matter in the upper Cenomanian-lowest Turonian of the Pont d'Issole section. A – Graptolite fragment displaying curving interthecal septa; B – Chitinozoa; C – *Navifusa* ssp.; D – Cuticle.

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Identification of coal phyterals in the Middle Permian, Moatize Formation, Mozambique

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Abstract

The Middle Permian coals of Mozambique represent peat deposition synchronous to other Middle Permian coal deposits of high-latitude Gondwana. These coals are, as they directly overlay glacial sediments deposited at the terminus of the Late Palaeozoic Ice Age (LPIA). This period in geological history was accompanied by divergent evolution of land plants, coincident with increasing atmospheric concentrations of carbon dioxide. This study combines petrographic and palaeobotanical techniques to recognize different plant components (phyterals) in coals of the Moatize Formation, Moatize Basin, Mozambique.

Variations in the occurrence of coal phyterals are compared with organic stable carbon isotope values of the same samples, to assess how changes in atmospheric and depositional conditions may have affected plant type and preservation. Important observations made in these samples include the exceptional xylemic preservation of telovitrinite macerals, which exhibit minimal compression and degradation despite our current understanding of peat compaction and coalification.

Furthermore, the successful application of etching techniques on semifusinite, bring into question the origins of the cryptomacerals funginite and secretinite. From the development of these techniques it is possible to reconstruct peat-forming conditions and ecological changes in sub-bituminous to bituminous rank coals.

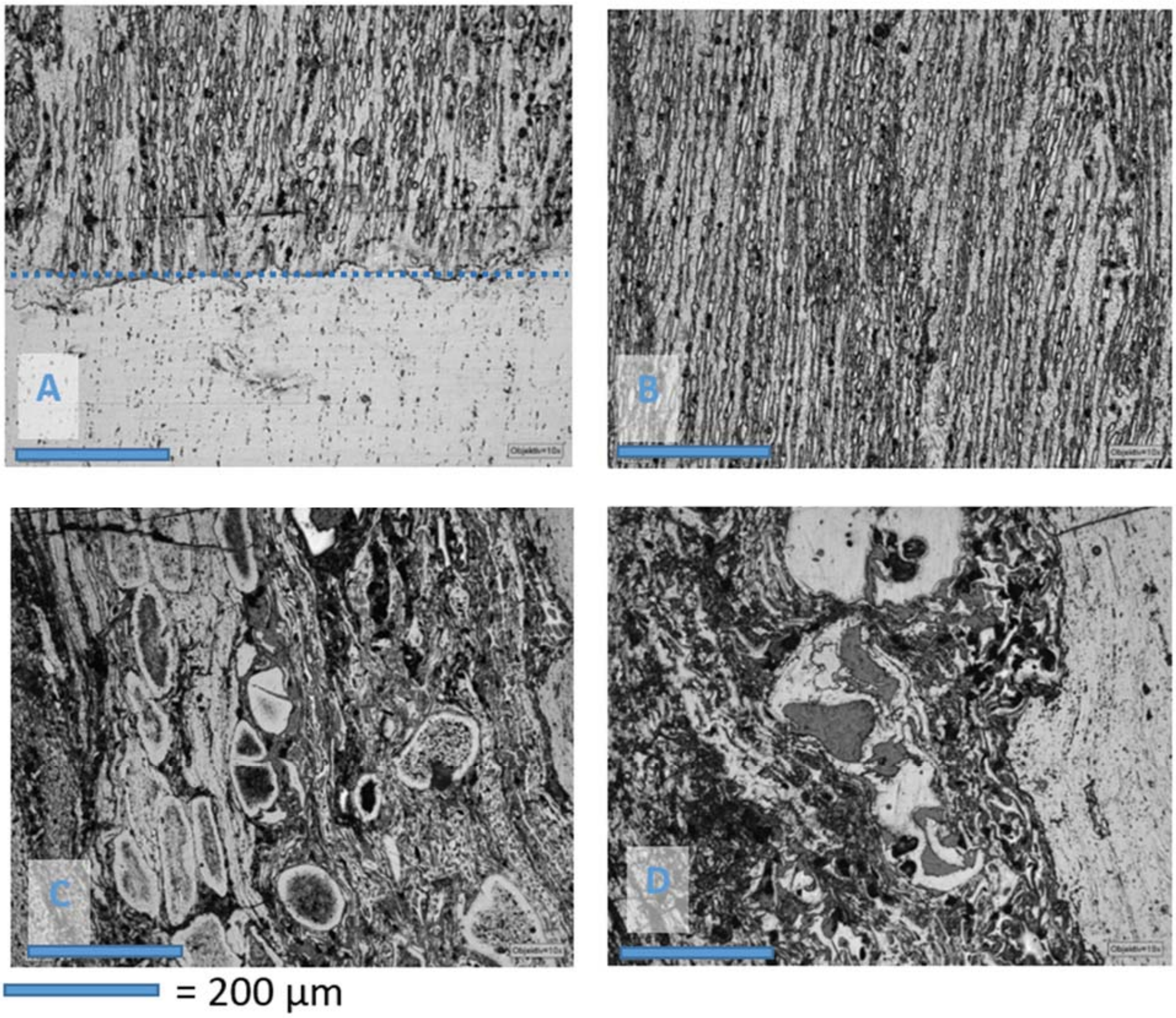

Fig. 1: Microphotographs of etched coal blocks in air in reflected light (10 x objective), bedding plane orientated perpendicular to the image long-axis: (A) blue dotted line indicates extent of etching, above the line is etched to reveal the phyteral’s cellular structure, below the line, unetched vitrinite, (B) etched telovitrinite exhibiting minimal cell-wall compression and intact xylemic structures, (C) etched semifusinite exhibiting extensive funginite, secretinite and structured fungal tissues, (D) etched semifusinite exhibiting mineral (likely clay) infilling of fungal tissues.

Magnetic susceptibility in coal: Is it a possibility?

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Abstract

Igneous dykes and sills that have intruded into coal basins can often reduce the economic value of the coal through changing the coal quality and creating dilution. When adding a heat source from intrusions, coals can undergo contact metamorphism which can lead to large amounts of naturally coked coal, which has negative economic implications on exploration and development (Chen et al. 2014). For mining companies to create reasonable assumptions and mining models, the use of standard geophysical tools such as, long and short spaced density, natural gamma, resistivity, calliper and sonic, aid the exploration process during borehole surveys (Biggs, 2005). The application of magnetic susceptibility is not used in a basic suite of wireline logs during exploration boreholes for the coal industry. Magnetic susceptibility could be used to determine if, in the heat affected coal, there is an exchange of mineralisation from the heat of the intrusion or from mineralisation caused by the geochemistry of the intrusion.

Intrusions may not always show magnetic susceptibility due to the composition of the intrusion or lack of ferromagnetic minerals from both the intrusions and the coal. Due to this variation of magnetic susceptibility in intrusions, this research aims to understand the magnetic susceptibility of: 1) the associated heat affected coal zones, 2) the fresh coal and; 3) the intrusion. This will be done through: - Petrological descriptions of intrusions, logging magnetic susceptibility on fresh and heat affected coal, geochemical analysis of coal and intrusions and analysis of wireline logs. Samples will be collected from two field sites, the Moatize Basin, Mozambique and the Bowen Basin, Queensland, Australia.

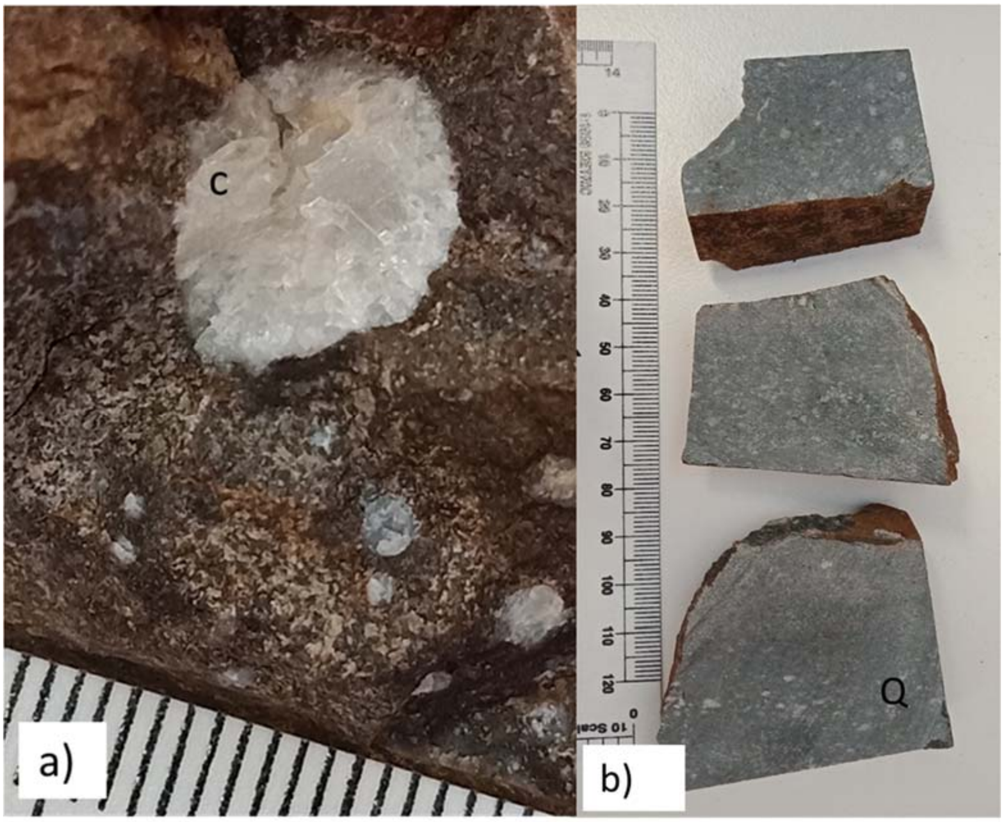

Fig. 1: Sample DO-25 from Moatize Basin. An intermediate composition dyke exhibits a) large calcite filled vesicles (C) b) white quartz grains (Q)

Acknowledgements:

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Organic composition, depositional environment, and hydrocarbon source potential of the lignite deposits from Sonari mine, Barmer Basin (Rajasthan), north-western India: petrographical, palaeopalynological and geochemical approaches

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Abstract

The lignite-bearing sequence of Sonari mine associated with Akli Formation (Eocene) of the Barmer Basin in north-western Indian state of Rajasthan has been investigated. The lignite and associated shale samples were subjected to petrographical, palynological, palynofacies and bulk geochemical studies to understand the nature of organic matter, source vegetation, and the palaeoenvironmental conditions prevailed during the sedimentation and peat accumulation. An attempt has also been made to assess the thermal maturity and hydrocarbon generation potential of these deposits. The results revealed the dominance of huminite group of macerals (av. 55 vol.%) and phytoclasts OM (av. 78%) indicating the occurrence of forested vegetation in the vicinity of deposition site. The palynological assembleges mainly represent dominance of pollen grains refererable to tropical families such as Arecaceae, Annonaceae, Liliaceae, Oleaceae and Onagraceae. The most abundant angiospermic pollen grains (*Spinizonocolpites, Kapurdipollenites, Retiverrumonosulcites, Acanthotricolpites, Clavadiporopollenites, Palmidites, Longapertites, Palmaepollenites*) are mainly referable to palms family Arecaceae, and one of the oldest monocots (*Proxapertites*) family Araceae. Also, the preliminary study of dinoflagellate cysts assemblage including age-diagnostic taxa recorded at several levels indicates early Eocene (? Ypresian) age.

A high CPI (5.03-9.44), TAR (5.09-20.01), and liptinite macerals (mainly sporinite, cutinite and resinite; av. 11 vol.%) values suggest the dominant inputs of terrestrial higher plants. Further, the dominance of detrohumintes (attrinite+densinite: av. 27 vol.%) along with a high frequency of non-biostructure (biodegraded) phytoclast OM (av. 43%) and hopanoids in the studied samples indicating a significant herbaceous plants input, and an exceeding level of bacterial biomass degradation. The terpenoid compounds (*m/z* 191) are mainly constituted by pentacyclic triterpenoids dominated by 30-norneohop-13(18)-ene, neohop-13(18)-ene, and 17β (H), 21β(H)-homohopane. The tetracyclic compound de-A-fernene has been identified which indicating the presence of pteridophytes in the peat-forming vegetation. The low to moderate amounts of inertinite macerals (av. 22 vol.%) and opaque phytoclasts OM (av. 28%) with a relative high pristane/phytane ratio (1-3, av. 2.6) in the samples are suggesting the prevalence of suboxic conditions during deposition. The substantial amount of pyrite (up to 17 vol.%) including framboidal, and a high diversity of mangrove *Nypa*-like pollen (*Spinizonocolpites*) and marine dinofalgellate cysts suggest a mrginal marine lagoonal swamp conditions.

The petrographical indices (GI-TPI, GWI-VI) indicate that the precursors of peat were deposited in a limno-telmatic condition, under back-barrier regime with rheotrophic hydrological conditions, mostly with high tissue-destruction. Similarly, the extrapolation of organic matter data on Tyson's APP diagram shows that the peat was accumulated in

marginal dysoxic-anoxic basin (Field-II) conditions. Consequently, the moderate content of amorphous OM (av. 18%) and high amount of non-biostructure phytoclast OM along with the constant occurrence of foraminiferal linings and dinoflagellate cysts in the samples reflect a regular marine incursions and the inter-mixing of terrestrial influx in the proximal settings.

The deposits have relatively higher abundance of C_{27} and C_{29} *n*-alkane hydrocarbons. The gross calorific values (av. 4601 k cal./g) and the T_{max} (av. 412 °C) showing that the lignites are of immature nature. These lignites are characterized by low to moderate moisture contents (av.13 wt.%) and ash yields (av.15^{db} wt.%). While, the volatile matter yields (av.51^{daf} wt.%) and the fixed carbon contents (av. 49^{daf} wt.%) are moderatly high. Fuel ratio is between 0.77 to 1.32. The TOC contents range from 1.17 to 54.84 wt.% with an average value of 24.78 wt.%, and HI values vary between 32 and 361 mg HC/g rock; indicating the excellent potential of the samples to generate hydrocarbons. The HI *vs.* OI, HI *vs.* T_{max} and S_2 *vs.* TOC cross plots suggest that the studied lignite-bearing sequence contains mostly Type III kerogen/OM, and has the potential to generate mainly gaseous hydrocarbons.

Acknowledgements:

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Organic matter petrography from well cuttings of the Cuyo Group (Jurassic, Neuquen basin, Argentina)

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Abstract

The Neuquén Basin lies on the eastern side of the evolving Andean mountain chain and contains a Late Triassic–Early Cenozoic succession, being one of the main gas- and oil-producing basins in Argentina. The first major marine depositional episode after basin formation comprises the Cuyo Group, a more than 2,500 m thick succession involving a Hettangian–Pliensbachian transgressive interval followed by regressive deposits reaching middle Callovian (Zavala et al., 2011). The Lower–Middle Jurassic petroleum system comprises source rocks (Los Molles Fm.), and reservoir rocks and seals (Lajas Fm. and the overlying Callovian–Oxfordian Lotena Gp), which were deposited in a scenario strongly influenced by the topography inherited from the underlying synrift stage (Howell et al., 2005).

In this study, the petrography of the organic matter from well cuttings of the source rock (prodeltaic marine shales) and, secondarily, of the reservoir rocks (delta front, delta plain and alluvial sandstones) from the Cuyo Group has been investigated (García-Sánchez, 2018). Samples were selected from two YPF-owned wells located 76 km apart on the Huincul High. These two wells, fictitiously named A and C, were selected as representative of the deposits lying on a paleohigh and a relative depocenter, respectively. The study was aimed at detecting differences in the type of organic matter and the degree of maturation between the two wells. Selected samples, 24 (well A) and 15 (well C), contained a high proportion of dark mudstones and siltstones supposed to be organic-rich in thin section. The work was carried through a combination of transmitted- and reflected-light optical microscopy, and scanning electron microscopy using Energy Dispersive X-ray spectroscopy for the identification of elements (SEM-EDX).

Two main organic-matter assemblages were detected that could be associated to the two wells. The organic matter of the well A, located on a paleohigh, is dominated by humic components (inertinite>>>vitrinite). Common accumulations of small, well-sorted particles, were detected, mainly to the top, whereas less and larger particles were found towards the bottom, differences that could not be detected in the study of the thin sections. These components are associated to gas-prone type III and to type IV kerogen. The second assemblage of organic matter, typical of the well C, corresponds to the deepest (=more distal) samples, and is dominated by diffuse kerogen found dispersed in the clay matrix, and less commonly in inter-crystalline spaces of pore-filling cements or, occasionally, in interparticle spaces of sandstones. Humic components were also common with inertinite dominating over vitrinite. The samples did not exhibit fluorescence due to their high maturity and therefore a tentative precursor for the bitumen, likely autochthonous organic matter, could not be identified. These results are consistent with the palynofacies studies of Martinez et al. (2008) who found organic matter in locations close to the studied wells dominated by phytoclasts and terrestrially derived palynomorphs. Fig. 1 shows a scheme explaining the interpreted relationships between type of organic matter and paleohigh vs depocenter distribution.

The identification of the indigenous vitrinite population was extremely difficult in these samples, not because of scarcity but of the coexistence of: i) a population of suppressed vitrinite, frequently associated with abundant framboidal pyrite, but without other distinguishing features, ii) a population of bitumen with lower, but close reflectance to the vitrinite, and iii) the abundance of inertinite, quite often of very small size, with a significant variability of optical properties. In addition, the presence of vitrinite from cavings from upper levels was not uncommon. A second bitumen population of higher reflectance than the vitrinite was also identified in well C. Despite these difficulties, an indigenous vitrinite population could be identified, whose reflectance increases with depth at a rate of 0.08-0.16% per 100 m, depending on the mathematical adjust used. The degree of thermal maturity reached corresponds to catagenesis and metagenesis. Linear correlations have been established between vitrinite and bitumen reflectance, which will allow for maturation estimation when direct vitrinite-reflectance measurements are not available.

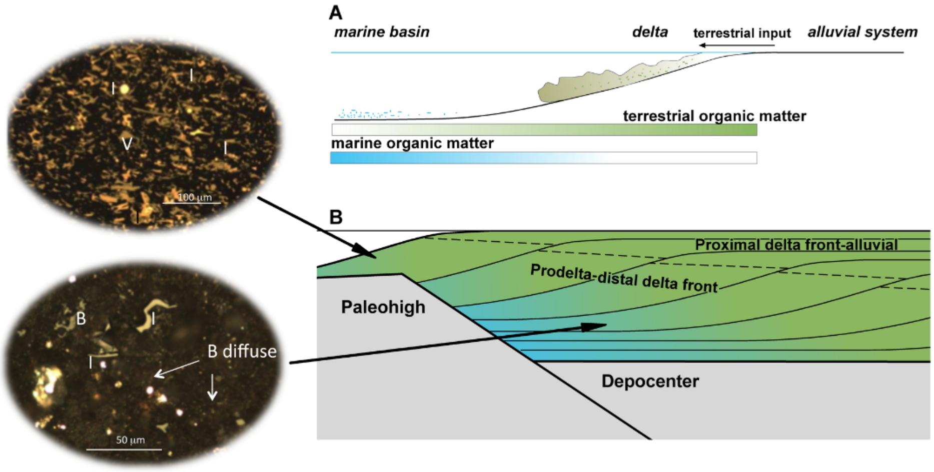

Fig. 1: Idealized sketch showing a tentative explanation for the variations in the organic assemblages found in the wells A and C. Micrographs taken with incident light oil immersion: I=inertinite; B=Bitumen; V=vitrinite.

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Organic petrology and geochemistry of coal from Santa Susana Basin (Portugal)

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Abstract

Carboniferous sedimentary rocks are present in all tectonostratigraphic zones of the Iberian Variscides. In the Central Iberian and Ossa Morena zones, Pennsylvanian rocks occur in continental intramontane basins restricted to narrow outcrop stretches located along major Variscan shear zones, such as Douro, Buçaco and Santa Susana Basins. The Santa Susana Basin (SSB) is a Pennsylvanian continental basin that currently crops out ca. 10 km NE of Alcácer do Sal (SW of Portugal). It extends over 15 km in length along a NNW-SSE trend and is up to 1 km wide. The basin is located along the Santa Susana Shear Zone (Machado et al., 2012) that separates the South Portuguese and the Ossa-Morena Zones. The SSB rests over two older (Carboniferous) Ossa-Morena Zone units: the Toca da Moura Volcano-Sedimentary Complex and the Cuba Group magmatic rocks. The basal unit of the basin is essentially composed by coarse-grained basal conglomerate with felsic magmatic boulders overlain by coarse sandstones and polymictic conglomerates. The upper unit includes quartz/quartzite-rich gravel conglomerates, sandstones, shales and coal seams. Despite the fact that coal from the SSB was exploited in the last century, studies on the petrography and geochemistry of the coal are scarce (Fernandes et al., 2016; Sousa et al., 2010). The main goal of this study is the petrographic and geochemical characterization of coals from this intramontane basin.

In the SSB there are three main outliers (from N to S): Jongeis, Remeiras and Vale de Figueira. Sampling included the collection of two coal seams and coaly silt-claystones in Vale de Figueira and coal samples from a seam and from mining waste deposits in Jongeis. Optical microscopy was used for maceral analysis and for the determination of vitrinite reflectance, which give information about the coal rank and the basin thermal maturity. The petrographic observations and measurements were carried out using a Leica 4000 M microscope equipped with a Discus-Fossil system under standard conditions, in polished whole rock blocks prepared according to standard procedures. The petrographic characterization of the organic matter followed the International Committee for Coal and Organic Petrology (ICCP) 1994 System nomenclature. The methodologies used for the geochemical characterization included: proximate analysis and inductively coupled plasma mass spectrometry for the determination of the inorganic composition of samples in major, minor and trace elements.

The microscopic observations reveal that the organic matter of coal from SSB is mainly composed of vitrinite followed by small amounts of inertinite and liptinite that is scarce. The vitrinite particles include collotelinite and macerals of detrovitrinite sub-group. The inertinite includes, mainly, fusinite, semifusinite and inertodetrinite and, rarely, macrinite and funginite. Sporinite was observed within vitrinite particles only in samples from Vale de Figueira, which can be attributed to depositional environmental conditions. The organic particles occur isolated or interbedded with mineral matter. The mineral matter includes detrital minerals (mainly quartz and clays), iron oxides and oxidized and non-oxidized framboidal pyrite. The iron oxides occur as groundmasses and, occasionally, filling some fractures. Epigenetic carbonates were observed only in samples from Jongeis.

The vitrinite random reflectance of the studied samples ranges between 0.90% and 1.25%, indicating a bituminous coal rank. Samples from Jongeis present vitrinite reflectance ranging between 0.95% - 1.25% whereas samples from Vale de Figueira have reflectance varying between 0.90% - 0.98%. The slightly higher vitrinite reflectance and the occurrence of epigenetic carbonates in samples from Jongeis can be attributed to differentiated burial history of the basin and post-depositional processes.

The results of proximate analysis reveal a medium to high ash yield. Considering the major elements, the geochemical composition of samples is generally similar, being Al, Ca, Fe, and K the most abundant elements (>1%), followed by lower concentrations of Mg, Na, P, S, and Ti (<1%). Exceptionally Ca and Mg exhibit significantly higher content in the majority of samples from Jongeis, which is related with the presence of epigenetic carbonates that were identified during petrographic observations. The geochemical composition in minor and trace elements is also generally similar between the samples from each area; the Mn and Y have significantly higher concentration in samples from Jongeis, while the other elements have generally higher concentration in samples from Vale de Figueira. The Pearson's correlation coefficients ($p<0.05$) indicate that the majority of minor and trace elements are positively correlated with ash yield, indicating their association with mineral matter. On the contrary, carbon is positively correlated with S, Cd, Mo, Pb and Se indicating the association of these elements with organic matter; the negative correlation of these elements with the ash yield corroborates their organic affinity. In the literature it is reported the organic affinity of these elements (Finkelman et al., 2018).The Ca has strong positive correlation with Mg, Mn, and Te, which indicates the affinity of these elements with the carbonates.

Acknowledgements

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Petrographic characterization of DOM in Proterozoic source rocks, McArthur Basin, Australia

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Abstract

The Palaeo- to Mesoproterozoic intracratonic McArthur Basin comprises unmetamorphosed sediments from fluvial, lacustrine to shallow marine environments. This basin is one of the oldest petroleum systems in the world and has been explored intensively in the past few years for oil and gas. This abstract describes the organic matter found in the two main source rocks: the Wollogorang Formation (ca. 1730 Ma) of the Tawallah Group and Barney Creek Formation (ca. 1640 Ma) of the McArthur Group. The Proterozoic organic matter was petrographically characterised using existing nomenclature (ICCP) and based on the observations of Crick (1989) and Glikson et al. (1992) for samples from the McArthur Basin.

Given the age of the sediments, the precursor of hydrocarbons was petrographically recognised as **lamalginite** (Figure 1), which is defined as finely banded alginite parallel to bedding (Pickel et al., 2016) interbedded with mineral matter (**Error! Reference source not found.**1). Lamalginite can appear as isolated films or as aggregations of films forming thick layers. Depending on its maturation stage, it may present strong fluorescence in yellow and orange, tending to brown colours as its reflectance or maturation increases. In this study (as in Crick (1989)) **non-fluorescent lamalginite** was used to classify the organic material that appears as individual lamellae but without fluorescence. Both fluorescent and non-fluorescent lamalginite were observed in the two formations.

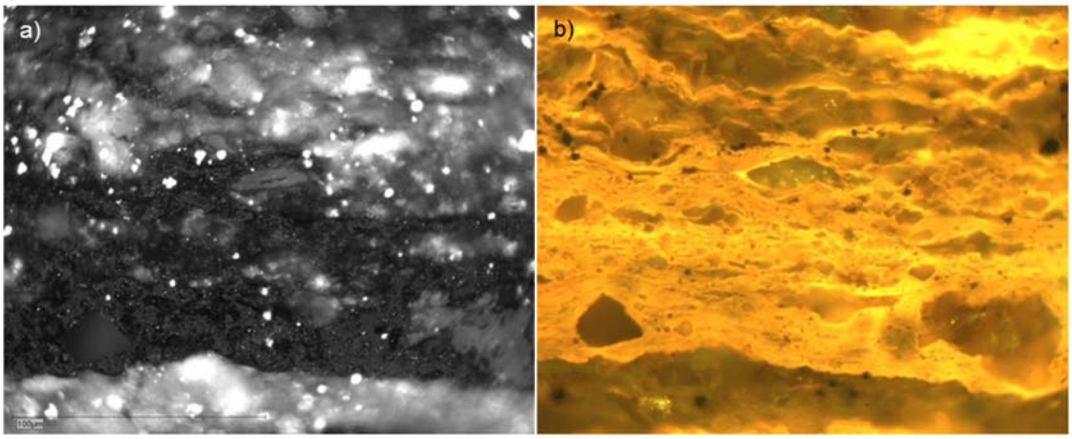

Fig. 1: Thick layers of lamalginite in the Barney Creek Formation. Optical photomicrographs taken in reflected white light (a) and the corresponding image in fluorescent light (b).

The other major organic components in the samples are **solid bitumens**, which are mainly secondary organic materials with shapes adapted to the form of the cavities they occupy (Jacob, 1989). They are solid phase accumulations of predominantly hydrocarbons produced by petroleum generation in a source rock (Landis and Castaño, 1995); that is, they indicate that hydrocarbons were formed and migrated. They can occur in the same sample as lamalginite indicating an *in situ* source, especially when their reflectance values are close to those of lamalginite (Glikson et al., 1992). Following the same classification scheme as Crick

(1989) but slightly simplified, in this work, solid bitumens were differentiated based on their mode of occurrence and shapes. They can occur in the rock **matrix (Figure 2a)** or in **fractures (Figure 2b)**. Solid bitumens in the matrix can occur as filling between crystal grains or in voids with no particular shape, or as discrete bodies, the latter, divided into **rounded bitumens** generally of low reflectance, **patchy bitumens**, with very granular texture and low reflectance, and **thucholite**, which has a mineral nucleus (usually a radioactive mineral) surrounded by organic matter. Solid bitumens in fractures can occur as a wavy coating along the border of a fracture, often in association with pyrite, or as an individual filling between the crystals inside the fractures. All solid bitumen forms occured in both formations.

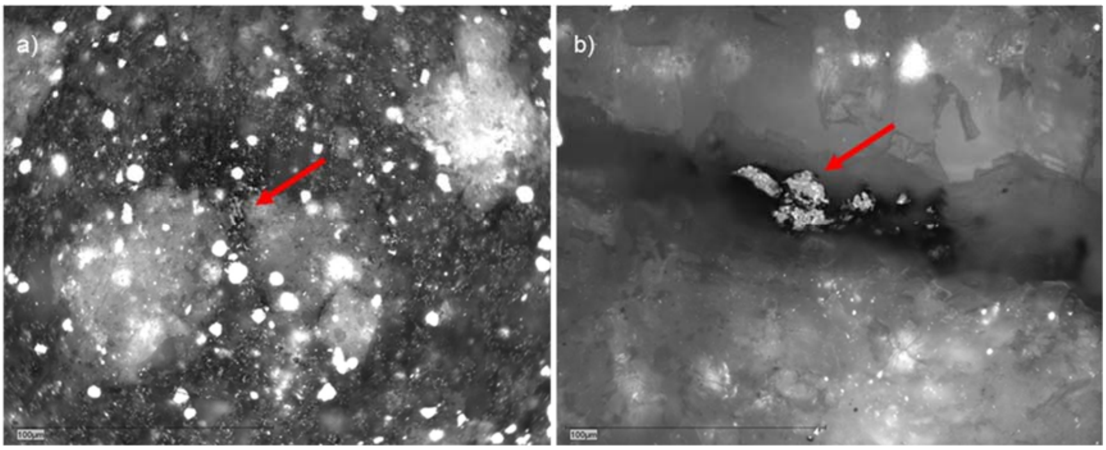

Fig. 2: Solid bitumen (red arrows) in the matrix (a) and in the fractures (b). Optical photomicrographs taken in reflected white light.

A Precambrian hydrocarbon source rock differs from most conventional Phanerozoic source rocks in that the organic matter is exclusively of bacterial or algal origin. Previously, only solid bitumen and thucholites had been identified in Wollogorang Formation. New petrographic analysis has confirmed the presence of solid bitumen as the main organic component, but has also identified primary kerogen (lamalginite), supporting the idea that some hydrocarbons in the McArthur Group may have been sourced from the underlying Tawallah Group.

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Phenanthrene sorption behavior in coals and cokes: the effect of rank and inertinite content

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Abstract

The study of PAHs sorption capacities of coals and carbonaceous rocks is a major subject in the fields of environmental research (e.g. Ligouis et al., 2005), since the fate of certain pollutants can be predicted in the natural environment. Previous studies showed that the sorption of phenanthrene in low rank coals is controlled, among others, through the inertinite content (e.g. Kalaitzidis et al., 2006).

In the current study, the sorption behavior of phenanthrene in a series of various rank coals, as well as cokes, is examined. The aim is to evaluate the impact of coal rank upon sorption attributes, but also to test the hypothesis that sorption will increase proportionally to inertinite content in higher rank coals, as it was observed for low rank.

The samples include lignite, bituminous coals, semi-anthracite and anthracite, naturally coked coals and industrial cokes; the rank-series represents a wide range of coal basins and coal-forming periods, ranging from Gondwana coals from Australia and South-Africa, Laurasian coals from China and Poland, and Neogene lignite from Czech Republic, thus demonstrating variable physical-chemical characteristics and most importantly, maceral contents.

The results showed that anthracite exhibited the highest sorption capacity (Kd) from all samples but also its behavior was totally different from this of the other samples. In fact, anthracite had a nonlinear Freundlich exponent (N) higher than 1, which suggested that the more the sorbed phenanthrene the higher the affinity of the sorbent for phenanthrene. Bituminous coal sorption behavior seemed to be affected by the inertinite content in terms of their nonlinearity (N<1); nonlinearity increases (N decreases) with increasing inertinite amount. If the organic carbon content was taken into account, lignite followed the same pattern that was observed in a previous study (e.g. Kalaitzidis et al., 2006). The sorption behavior of the two coke samples seem being inaffected by the inertinite content; nevertheless, these two samples demonstrated the highest sorption affinity (Koc; which is the sorption capacity normalized for the fraction of organic carbon content) for phenanthrene at low concentrations.

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Reassessment of organic matter present in the Mississippian Deposits in the southeastern Moravia

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Abstract

The Mississippian sediments occur on the territory of the Czech Republic in the Upper Silesian Coal Basin (USCB) that is considered as one of the most significant coal basins of European Variscides. The southernmost documented occurrences of Mississippian coal bearing units can be found in "Němčičky Basin", that is considered by several authors to be the southern continuation of USCB. The south-eastern Moravia also belongs to the most perspective area for hydrocarbons and the Mississippian deposits are considered to be one of the potential source rock. Thus, the systematic evaluation of the Carboniferous units in Czech Republic with regards to the organic matter kerogen type and thermal maturity was performed. Part of the study also involved the assessment of the regional and sedimentary sequence distribution.

Samples of coal and siltstones from 8 boreholes were investigated. The geological setting and regional extent of the Mississippian in Němčičky Basin were interpreted using 3D seismic. Polished surfaces were prepared for the wells where the core was available and the vitrinite reflectance was measured. The polished surfaces were prepared from rock chips and the reflectance measurement was carried out in oil in non-polarized light (R_r) using a microscope Carl Zeiss Axio Imager M2m. The maceral composition was determined using the same equipment at the same conditions according to ICCP (1998 and 2001). The liptinite composition was determined using the same microscope with a fluorescence mode. Maceral identification followed the recommendation described by Taylor et al. (1998) and Pickel et al. (2017). The RockEval archival geochemical data from the Dambořice 1, Němčičky 1, 2 and 3 boreholes were evaluated to establish the prevailing kerogen type.

The samples uniformly represent the lithology of the silt to the coal. The total organic carbon from the Carboniferous strata varies between 0.6-75.1 weight %. Hydrogen index (HI) ranges from 0 to 384 $mg.g^{-1}$TOC. The increased values of HI evidenced the fact that the hydrocarbon potential has not been exhausted. Thermal maturity expressed by the T_{max} parameter ranges from 430-491 °C. Based on the relationship between HI and T_{max}, it was found that the Mississippian rock contains kerogen type II-III. The vitrinite reflectance (R_r) increases regularly with depth from 0.54 to 1.15%R_r in the Šitbořice 1, Borkovany 101 and Dambořice 1 boreholes. The random vitrinite reflectance in other deep-seated samples vary from 0.6 – 1.20 % and creates a relatively steep gradient with depth. The measured values of vitrinite mean reflectance R_r = 0.54 - 1.20 % correspond to the medium rank coal so called high to medium volatile bituminous coal. The prevailing macerals within the liptinite group are alginite and sporinite (Fig. 1) accompanied by liptodetrinite. Vitrinite reflectance in the Mississippian sediments changes with the depth in the three regionally distant regions and demonstrates the existence of a comparable geothermal gradient. The position of the samples corresponds to their present depth, which is given after Paleozoic development.

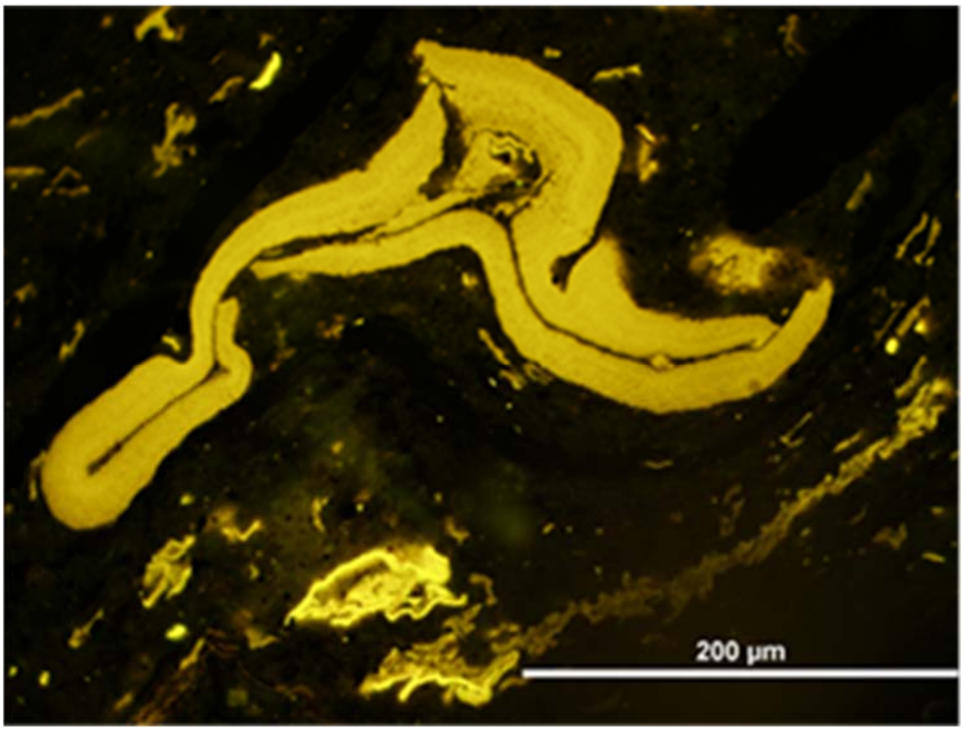

Fig. 1: Borkovany: Yellow sporinite (megaspore), fragment of alginite and liptodetrinite in clayey matter (fluorescence mode).

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Sources of coaly organic matter in the fluvial sediments of the Jiu River (Romania)

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Abstract

Despite the well-known environmental challenges of coal mining and coal processing activities, the hydrological consequences have received little consideration to date and the processes by which the smallest coal particles reach the river system have been often overlooked. Following the downfall of the coal mining industry over the last three decades, several Romanian watersheds draining coal mining/waste source areas face a new issue in terms of hydro-sedimentary dynamics with the presence of coaly matter in the fine sediments posing a threat to river quality and water management. The investigation of coal particles in sediments may be useful for determining the origin(s) of the suspended sediment load.
The aim of our study is to explore the relationship between sediment source areas and the coal matter in the fine sediments. The study area is the Jiu River Basin (10,080 km^2), a coal-rich watershed situated in south-western Romania, where coal mining and coal processing activities dominate both its upper sector (Petroşani coal basin) and middle sector (Motru-Rovinari coal basin). It is thus generally considered that the coal extraction- and processing activities play a significant role in the formation of fine sediments of mixed natural- and anthropogenic components.

The proposed methodology makes use of various coal petrographic properties. Due to the lack of hydro-sedimentary data-series at small time scales, the coaly matter in the sediments has been adopted here as a proxy for analysing fine sediment transport instead of the conventional methodologies used in hydrology. A field- and laboratory study was made on environmental- and bulk-coal samples. To assess the fine sediment deposits, the sampling started from a point bar in the Jiu River's reach downstream from almost all potential sedimentary sources. In total, 12 fine-sediment samples were collected from the alluvial layers of the riverbank deposit built by the largest floods of the last few decades. To allow for a comparison between the coal content of the samples collected downstream and the possible anthropogenic sources of sediment, additional environmental samples from the Jiu river and its tributaries, and coal samples from coal seams and sterile dumps were subjected to vitrinite reflectance measurements and petrographic analysis to determine the content of lignite, hard coal, chars and minerals. To answer a broader research question, the research was also aimed at eventually helping to reconstruct the magnitude of floods and sediment sources within the sub-watersheds of the Jiu river basin.

In the environmental samples, coaly matter was found to comprise <25% from the total organic- and inorganic mass. In terms of its rank, the presence of both hard coal and lignite particles was confirmed, particularly in the downstream alluvial samples, which demonstrates the contribution of different upstream source areas to the sediment delivery. Generally, it was assumed that the greater the amount of coal, the stronger the flood event that had transported and deposited it. Narrow reflectograms for bulk coal-samples represent a homogeneous particle pattern, and a dominant species of coal. Typically, a random reflectance (R_r) of ~0.6%

is specific for the hard coal samples from Western Jiu branch of Petroşani coal basin, while a reflectance of 0.35-0.5% characterizes the coal from the Eastern Jiu source. The same range being also characterises the lignite samples from the middle sector of the Jiu River basin. Chars increase in quantity in the alluvial deposits from the upstream sources to the downstream areas due to contributions from anthropogenic coal sources. An important difference between the bulk coal samples representative of a major part of the source areas and the coaly matter in the environmental samples is the higher mean value of the hard-coal reflectance of the latter. This is probably due to the presence of variable numbers of coal particles with a reflectance greater than the reflectance in bulk coal samples from the source areas. These values exceed the maximum measured on the source coal samples, thus opening two avenues for in-depth investigations of this problem, supported by microscopic analyses of the coal particles. Firstly, there may be other hard-coal seams in the Petrosani basin with much higher vitrinite reflectance than those already sampled. Secondly, apart from the unaltered coal particles with reflectance between 0.75-0.85%, the thermally altered samples (industrial combustion followed, in some cases, by alteration under riverine conditions) with reflectance between 0.7-1%, could not have been captured by analysis of samples from the coal source areas. Finally, the correlative analysis between the reflectograms of the samples allowed for a good estimate of the lignite- and hard-coal content in the river sediments, their proportions being comparable with the results from the petrographic analysis. However, we cannot reliably identify the sub-bituminous coal content from the Eastern Jiu river basin and from the lignite coal extraction areas, given their similar reflectance values.

The first results of our study encourage further analysis of the coaly matter in the fluvial sediments, as coal has proven its suitability as a hydro-sedimentary tracer of anthropogenic origin that we can identify in the fine sediments of the Jiu River. Establishing the origin of the organic component of the Jiu River's fine sediments would contribute to providing management solutions for the stabilization of waste dumps and the enforcement of legal actions for controlling post-mining erosion processes to minimize downstream impacts.

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The combination of SEM-EDS and RLOM in the investigation of coal-waste organic- and mineral matter. A novel proposal.

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Abstract

Coal waste is typically composed of mudstones, sandstones, and clays forming layers of varying thicknesses. Dispersed organic matter occurs in all in varying amounts, usually a few %. Commonly, coal waste is altered by weathering, and by self-heating leading to self-combustion. The processes that change both organic- and mineral matter in coal waste occur over a wide temperature range from 20-1300°C, and are influenced by moisture- and oxygen contents, and the rate and duration of heating. The these processes include organic-matter oxidation, plasticizing of organic- matter, devolatilization, formation of strongly-altered organic particles, mineral phase oxidation (e.g., of pyrite and marcasite to Fe_2O_3), mineral dehydration (the release of H_2O or OH groups from clay minerals and micas), mineral-structure rebuilding/recrystallization (enantiotropic transformations of e.g., SiO_2), metastable-phase formation, high-temperature mineral growth (e.g., mullite), mineral crystallization from melt (e.g., indialite), and weathering after dump quenching (Ciesielczuk et al., 2014).

These processes result in a host of new mineral phases and organic forms. For possible usage of coal waste as a by-product, and to investigate environment implications, the co-occurrence of organic- and mineral matter in waste dumps is being researched by the authors. The task is difficult due to the typically small size of particles being investigated. Differing approaches and a variety of methods are required.

Similar problems were recently addressed by e.g., Hackley et al. (2017) and Liu et al. (2017). However, their approach to the investigation of relationships between organic- and mineral matter differed regarding the preparation of samples and methods used. The method proposed by us is not expensive. It uses the same samples and equipment. No additional apparatus is required. The order of the analyses is changed.

The aim of this project was to elaborate a procedure that would combine various methods of both organic- and mineral-matter examination using as an example coal waste of different degree of thermal alteration. The methods used were Scanning Electron Microscopy combined with EDS (to give rough chemical compositions and preliminary identifications of mineral phases), X-Ray diffraction (to identify mineral phases) and Reflected Light Optical Microscopy (for identification of organic matter and reflectance measurement). Detection limits and minimum particle sizes differ for the various methods. SEM-EDS and RLOM can investigate objects of sizes ranging from ca 1-10 µm whereas the X-ray micro-diffraction technique allows identification of objects of ca 100 µm in minimum diameter. As XRD is a destructive method, it should be the final one used. RLOM and SEM samples can be prepared in the same way. Polished blocks or thin sections are best. However, for reflected-light optical microscopy, they must be covered with immersion oil - a procedure incompatible with SEM. Furthermore, the same objects observed with SEM and RLOM look so different that, without some indication, they can be difficult to find. A combination of SEM and RLOM allows not only

different forms of co-occurrence of organic- and mineral matter in coal waste to be established but also the mineral composition to be determined which, because of alteration during self-heating, is very difficult in reflected light.

To overcome the difficulties in combining these methods, we propose the following procedure for the investigation of mineral- and organic matter. (1) Attach a piece(s) of aluminium tape of known shape(s) to the polished section. (2) Draw or photograph the sample. (3) Examine the sample using SEM in known/marked places. (4) Find objects using RLOM in marked places. (5) Use a binocular microscope to find objects already investigated by SEM-EDS and RLOM and, where >100 µm, powder for X-ray micro-diffraction.

The proposed method is time consuming. However, it allows mineral composition in a given place to be determined. The investigated area within a sample is chosen “by chance”, without knowledge of the location of other interesting objects within the sample. Nevertheless, the method allows determination of the form of co-occurrence of organic- and mineral matter, establishing types of organic- and mineral matter as well as mineral composition. The method can be applied to any other rock or material, e.g., fly ash, where organic- and mineral matter occur together.

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Occurrence of Mercury and enrichment source in coals from the Douro Carboniferous Basin, São Pedro da Cova area (NW Portugal)

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Abstract

The Douro Carboniferous Basin (DCB) is located in the northwestern Portugal and is the major Carboniferous Basin present in the Douro-Beira Carboniferous Trough (Sulco Carbonífero Dúrico-Beirão) occurring in the Central Iberian Zone. This basin, with general orientation NW-SE, consists in a narrow strip extending for 53 km with a width rarely greater than 500 m (Pinto de Jesus, 2003). The stratigraphy and the sedimentary facies of the DCB were described considering several tectono-sedimentary units (TSU) (Pinto de Jesus, 2003). The coal of this basin is classified as high rank A coal (anthracite A) according to the ISO 11760 (2005), and it was exploited for many years (1795-1994) in two main mining areas, S. Pedro da Cova and Pejão, mainly as fuel for power generation.

In order to quantify the mercury concentration, which is an element of environmental concern and also to identify its mode of occurrence and source of enrichment, a total of five anthracite samples (codes: 15, 274, 108/116, 721, and 77) were selected its study. The concentration of Hg in the coal samples was determined by using an ICP-MS. Aiming the determination of the Hg mode of occurrence, samples 15, 274 and 721 were analyzed using a high resolution environmental scanning electron microscope (ESEM).

Results regarding the Hg concentration revealed high and variable concentrations, ranging from 0.10 to 44.02 ppm. Thereby all the coal samples, with the exception of sample 77 (0.10 ppm), are considered enriched in Hg when compared to the world hard coals (Ketris and Yudovich, 2009). The concentration coefficients (CC) of Hg for the studied samples were also calculated and classified considering the categories established by Dai et al. (2016). Sample 15 exhibits an unusual enrichment of Hg and a CC higher than 100.

Mercury geochemical affinities were obtained through Pearson correlation coefficients of Hg with the ash yield (ash) and total sulfur (St) content. Mercury displays an intermediate affinity with ash yield ($r_{Ash\text{-}Hg}$ = -0.18) and a sulfur affinity ($r_{St\text{-}Hg}$ = 0.67). The intermediate affinity may indicate both an organic and inorganic association of Hg in the studied coals. However, the positive affinity with St point out to an association with sulfide minerals.

SEM-EDS analysis revealed the occurrence of cinnabar (HgS) in two samples (samples 15 and 721). This Hg sulfide occurs, frequently, in particles of about 50 µm of size. Pyrite, which is the main carrier of Hg in coals (Yudovich and Ketris, 2005), was observed both in framboidal form and filling cleats, but the occurrence of Hg was not identified neither in pyrite nor in organic matter. The cinnabar mineralization is clearly of epigenetic origin and sometimes cinnabar appears filling devolatilization vacuoles in coal (Fig. 1). These devolatilization vacuoles are the result of significant thermal effects and reflect the high degree of pressure and temperature reached during the coalification process. This mineralization is thus clearly subsequent to the coal formation and its thermal alteration.

The primary factors of Hg enrichment in coal deposits are intrusion of low-temperature hydrothermal fluids and magmatic-hydrothermal fluids (Dai et al., 2015; Yudovich and Ketris 2005). A porphyry intrusion was observed inside of the São Pedro da Cova mine (Teixeira and Fonseca, 1945), immediately at the top of TSU B1, thereby since coal sample 15 is the one with the highest Hg content and belongs to TSU B1 this may be associated with Hg unusual enrichment observed in the coal sample from this area. Furthermore, Pereira (1945) described the magmatic rock as a leucocratic and a granodiorite, both enriched in Na minerals, this may explain the Hg-Na ($r_{Hg\text{-}Na}$ = 0.82) high correlation coefficient, pointing out the magmatic intrusion as the origin for the Hg enrichment.

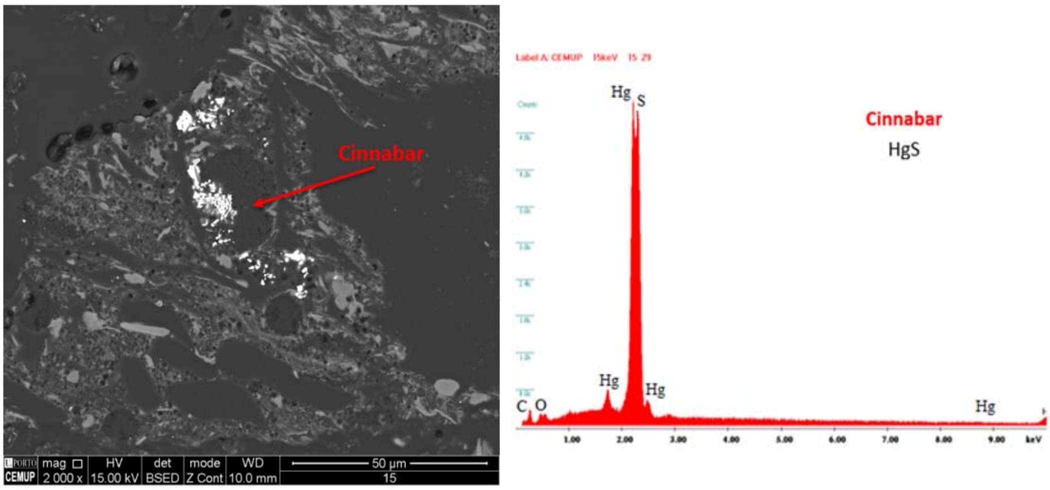

Fig. 1: SEM (BSE image) of cinnabar filling devolatilization vacuoles in the coal sample 15 from the DCB and EDS spectrum of its mineral phase (EDS spectrum).

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Organisation

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Dr Joan Esterle
Mr Peter Handley
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