Assessment of Organic Petrology Methodology and Characterization of Primary and Secondary Dispersed Organic Matter in Liquid-Rich Shales: Using optical microscopy methodologies*

L. D. Stasiuk¹, Amr El-Ezhary¹, Michelle Asgar-Deen², Volker Dieckmann³, Rob Robinson², and Gary Green²

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Abstract

Traditionally, organic petrology (OP) method is defined as an optical-based, reflected white and fluorescent light, microscopic characterization-classification-quantification, and measurement of % reflectance in oil (%Ro) and VIS of individual primary (kerogen) and secondary (e.g., bitumens, oils, solid hydrocarbons, pyrobitumens) organic matter, referred to as macerals. Combined, primary and secondary organic matter, make up total weight percent of organic carbon in sedimentary rocks, including liquid-rich shale (LRS). Perfecting the sample preparation of fine-grained rocks is not achieved in many labs, thus negatively impacting analytical data and interpretations. Ideally LRS samples for OP maceral analyses should be whole rocks, very gently crushed and or sized such that random, parallel, and perpendicular to bedding, and or microfractures are represented, and impregnated with new, very low viscosity, epoxy, under vacuum conditions. Use of solid, bored, acrylic rods and epoxy are an ideal combination for superior preparation of LRS samples, thus ensuring integrity and retention of all microtextural elements and organic (e.g., friable 'solid bitumens') and mineral (e.g., clays) matter that are otherwise prone to plucking during the grinding and polishing process, resulting in poor quality data, poor interpretations.

Commonly, the assessment of microscopic organic matter is inadequate because of lack of versatility in classifications and under-recognition of secondary organic matter in LRS. A new, maceral classification for primary and secondary dispersed organic matter in sedimentary rocks, adopted by the International Committee for Coal and Organic Petrology (I.C.C.P) in 2013, will improve consistency in nomenclature between commercial labs, if explicitly requested by clients. In order to adequately evaluate original kerogen type, organic facies, and paleodepositional settings of LRS, based on data collected following the I.C.C.P. (2013) classification of dispersed organic matter (DOM), higher levels of macerals classification identification (e.g., maceral type, maceral variety, maceral sub-variety (Table below) needs to be augmented with inorganic microfossil components. Examples are provided from ahe WCSB.

^{*}Adapted from oral presentation given at Geoscience Technology Workshop, Hydrocarbon Charge Considerations in Liquid-Rich Unconventional Petroleum Systems, Vancouver, BC, Canada, November 5, 2013

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Secondary organic matter likely constitutes more of the TOC in LRS than has previously been recognized by OP; that is, there are higher amounts of solid hydrocarbon and pyrobitumens derived from kerogen to bitumen to oil conversion and from oil to gas and oil to condensate cracking. This has implications for assessing original petroleum potential of an LRS, and potential, for linking OM-related nano-porosity to individual secondary maceral types using combined OP and FIBSEM types of analyses

Maceral Group	Maceral	Maceral type	Maceral Variety	Maceral Sub-variety		
Liptinite	Alginite	Telalginite	Botryococcus	Tenui-Botryococcus; Large diameter/length colonies, Usually >50 micron, cell walls are highly thickened		
				Crassi-Botryococcus; Small diameter/length colonies, Usually 5 - < 50 microns, cell walls are thins		

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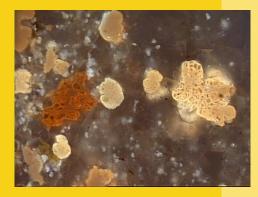
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Using optical microscopy methodologies



L.D. Stasiuk¹, Amr El-Ezhary¹, Michelle Asgar-Deen², Volker Dieckmann³, Rob Robinson², Gary Green²

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SIEP restricted October 2013

1.0 Introduction to Organic Petrology What is It?

'Organic Petrology is a branch of the Earth Science that studies fossil organic matter in sedimentary sequences including coal and the dispersed organic matter in rocks (DOM)'

'Organic petrology is usually expressed by two fundamental parameters: the nature and proportions of the organic constituents, and by the rank or maturity of these organic components. '

(Modified from Suárez-Ruiz et al., 2012)

Largely based on optical microscopic methods

1.0 INTRODUCTION TO ORGANIC PETROLOGY (OP)

Analysis of polished whole rock samples, using reflected white and fluorescent light, optical microscopy. OP can address questions such as:

- (i) What was the primary biological matter contributing to the kerogen (e.g., algae, spores, woody matter, bacteria); organic facies or paleodepositional setting, and control on original kerogen type/petroleum potential?
- (ii) What are the secondary products in the Organic Matter asemblage (oils, bitumens, solid hydrocarbons, pyrobitumens)
- (iii) What is the level of thermal maturity of the kerogen and secondary products (e.g., solid bitumens, oil inclusions), immature-, early oil-, light liquids-, condensate-, dry gas-windows

1.0 SAMPLE PREPARATION & INSTRUMENTATION

Sample preparation for microscopy:

Reflected light white and fluorescent light microscopy:

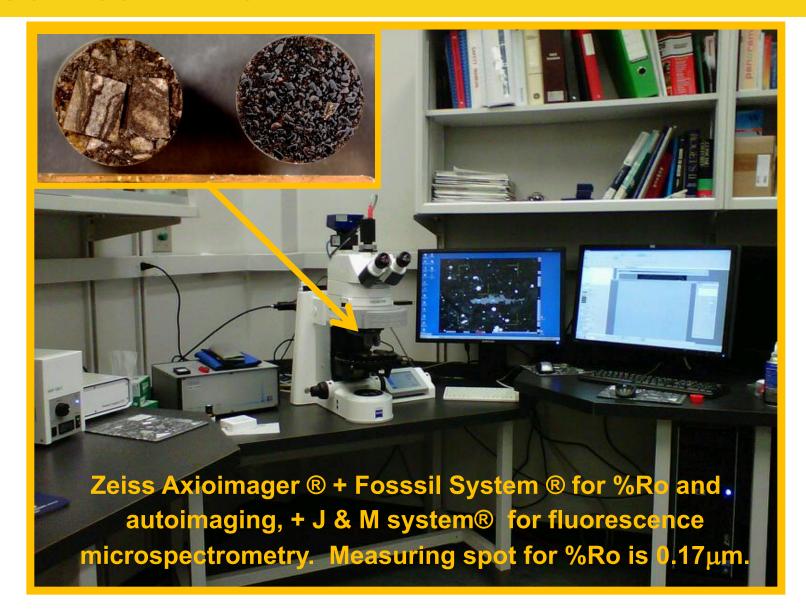




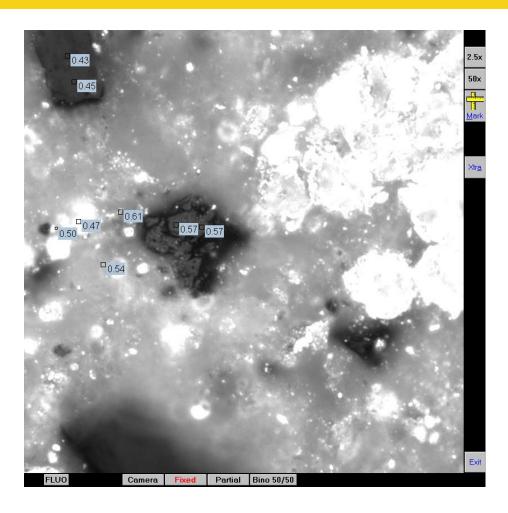


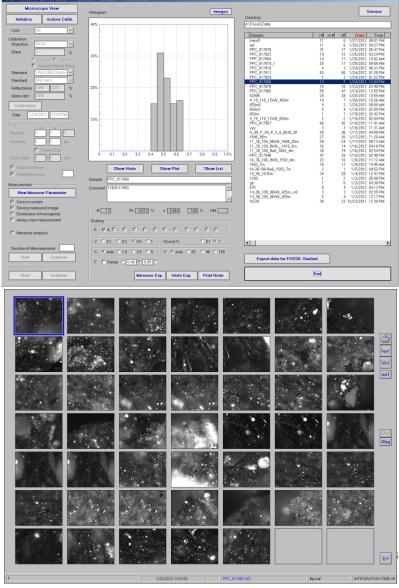
January 2012

1.0 OPTICAL MICROSCOPY: REFLECTED WHITE & FLUORESCENT LIGHT



1.0 VITRINITE %RO: ZEISS ® AND FOSSIL SYSTEM ® EXPANDED CAPABILITIES FOR %RO MEASUREMENTS FOR THERMAL MATURITY ASSESSMENT; DIAMETER 0.17UM X 0.17 UM, WITH A RECORD OF ALL MEASUREMENTS





1.0 Organic Matter Classification using optical Microscopy

Maceral Group Vitrinite



Maceral examples

Precursor biological matter

telovitrinite

cell walls of wood (and other lignin etc.) Derivatives

Liptinite/Exinite



Sporinite

resinite
cutinite
Chlorophyllinite
Resinite

Alginites, Dinoflagellates, Acritarchs

fusinite semi-fusinite

terrestrial plant spores, pollen

higher and lower waxy plant leaves, cuticles,

chlorophyll plant resins

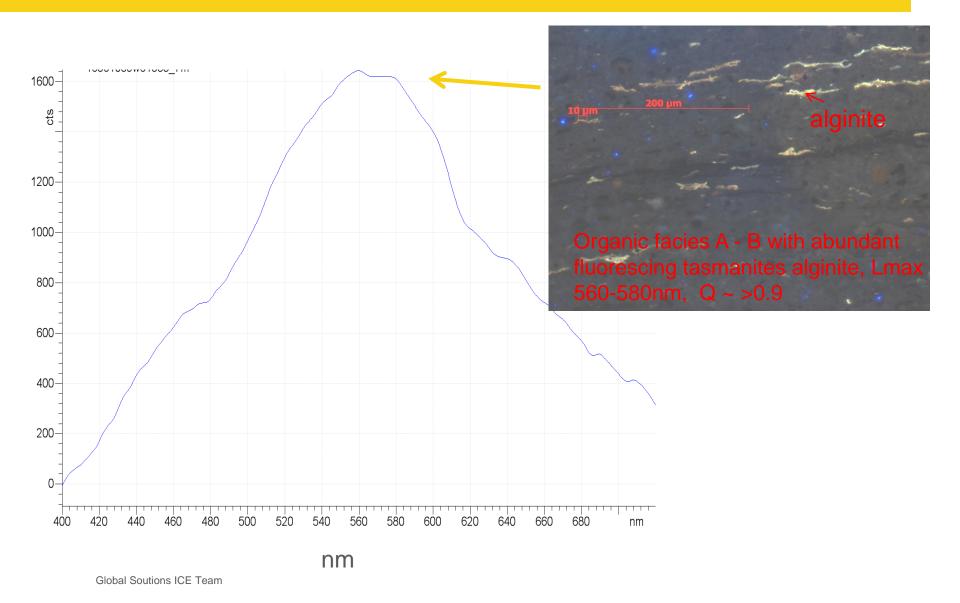
Inertinite



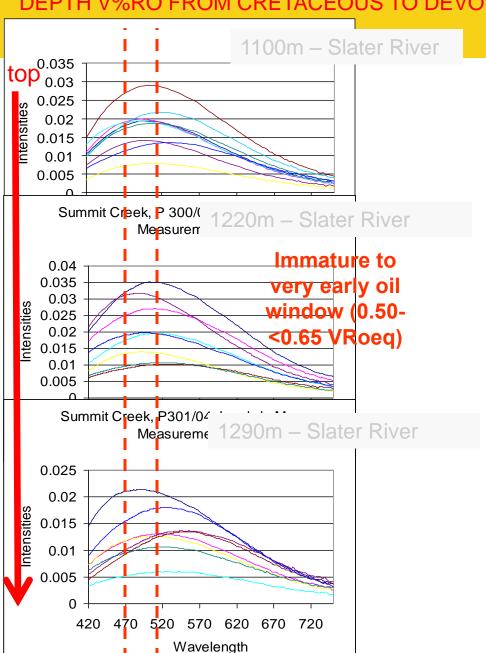
marine and freshwater algae, benthic or planktonic

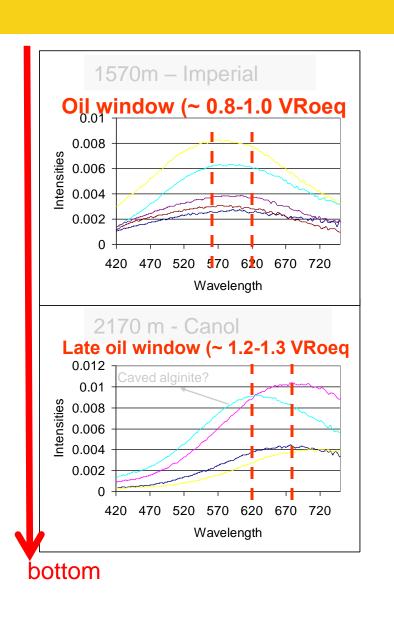
wood-derived charcoal; oxidized organic matter floral faunal-bacterial,

1.0 METHODOLOGY: J AND M VIS MICROFLUORESCENCE SPECTRA FOR PRASINOPHYTE ALGINITE IN MUSKWA FM, ~ > 0.70 - < 0.80 V%RO EQUIV.

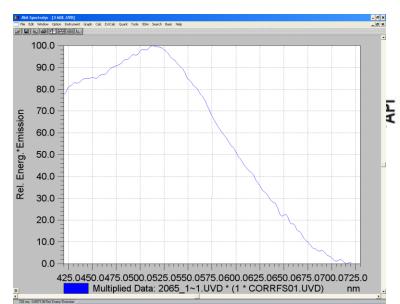


VIS FLUORESCENCE OF *TASMANITES* SHOWING RED SHIFT WITH INCREASING DEPTH V%RO FROM CRETACEOUS TO DEVONIAN, MATURATION SERIES AT B-45,





1.0 VIS flourescence microspectrometry of petroleum inclusions and API gravity.



Crude oils: Application of fluorescence microscopy and VIS fluorescence microspectrometry to the study of oil charactes (e.g., API estimate) migration using petroleum inclusions

Fluorescence colors (parameters used are: %.o Lambda maximum = Wavelength of maximum emission and Q = Int650 nm/Int 500nm)

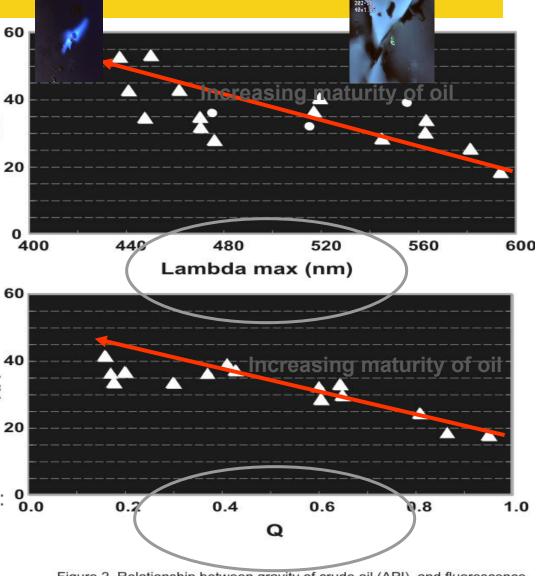
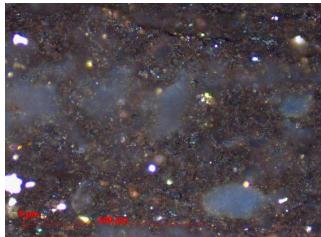
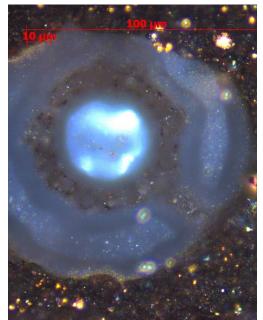
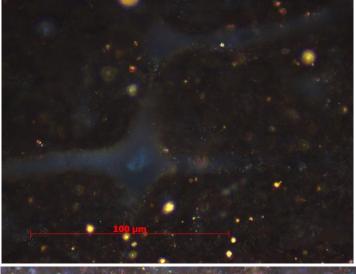


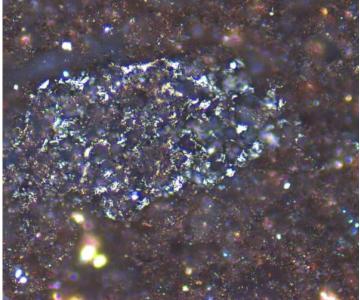
Figure 2. Relationship between gravity of crude oil (API) and fluorescence parameters, Lmax and Q (from Stasiuk and Snowdon, 1997).

1.0 METHODOLOGY: REFLECTED WHITE LIGHT MICROSCOPY OF POLISHED WHOLE ROCK PARALLEL TO BEDDING VIEW, BIOGENIC SILICA, ORGANIC FACIES D, UPPER DEVONIAN, MUSKWA, V%R 1.0



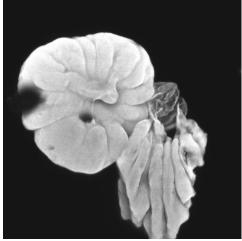


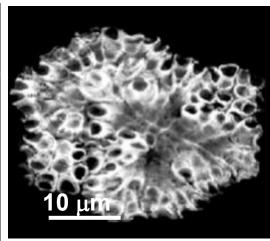


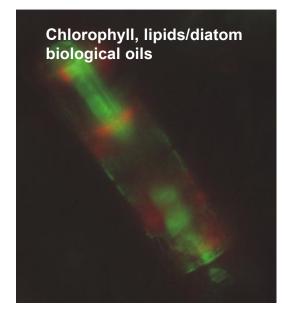


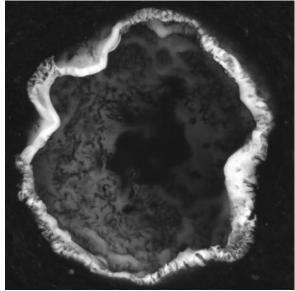
1.0 METHODOLOGY: CONFOCAL LASER SCANNING FLUORESCENCE MICROSCOPY











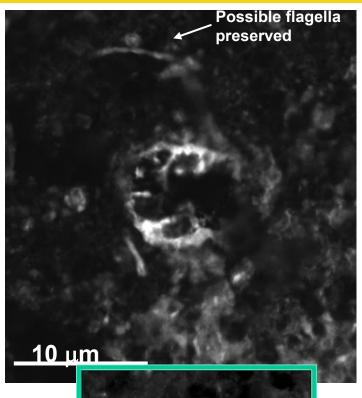


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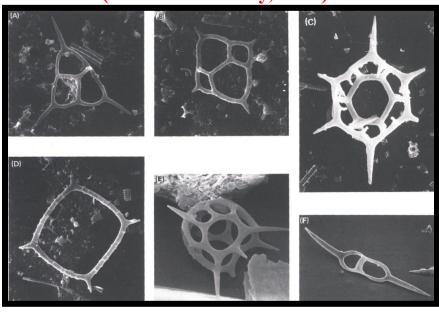
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1.0 METHODOLOGY: CONFOCAL LASER SCANNING FLUORESCENCE MICROSCOPY, PROTEROZOIC BIOGENIC SILICA PROBABLY RADIOLARIA-SILICOFLAGELLATE SKELETONS: INFRA-CAMBRIAN,~ 650 MA

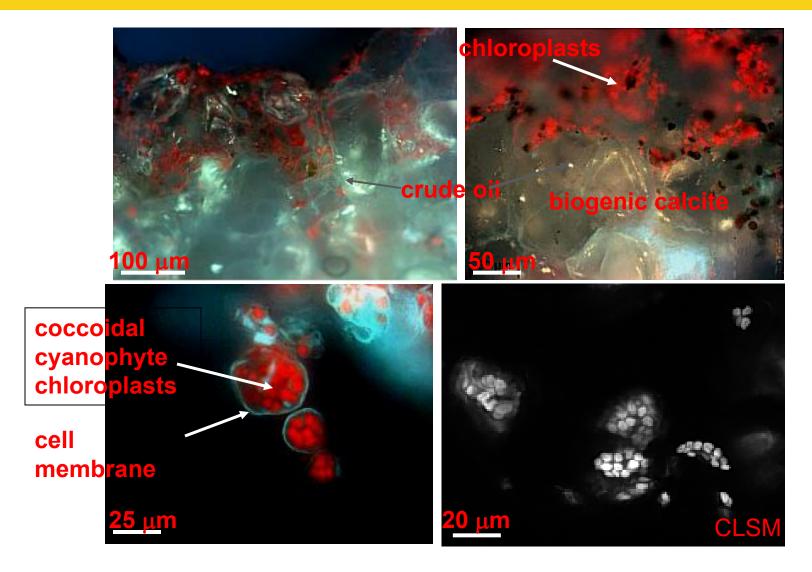


SEM images of Recent silicoflagellates (From McCartney, 1993)



➤ Age range in literature : 'first appear in Early Cretaceous'

Outcrop cyanobacterial 'mat' from desert in 4 corners area, SW USA-related to oil seep at surface (Recent and ancient organic matter)



2.0

Optical Microscopic Classification of Organic Matter

2.0 TSOP and ICCP Classification of Dispersed Organic Matter (macerals) in sedimentary rocks and isolated organic matter¹

MACERAL	MACERAL ³		CERAL	MACERAL ³
GROUP		Gi	ROUP	
Vitrinite	1.5-0.146.000-00-00-00-00-00-00-00-00-00-00-00-00	Z	ooclasts	Scolecodont
	Collotelinite			G II
·- ···	Vitrodetrinite		e III to II	Chitinozoa
(Type III	Collodetrinite	_ ke	rogen)	
kerogen)	Gelinite	C		Foram lining
- 1	Corpogelinite		ondary	(Migra)bitumen
Liptinite	Alginite	Pr	oducts	
	Bituminite			Oil
	(Amorphinite) ⁴			Pyrobitumen
(Type I to II	Liptodetrinite		_	=======================================
kerogen)	Sporinite	7	10	Rock Eval Source Hydrocarbo Kerogen Types Organisms potential
Kerogeri)	Cutinite			
	Suberinite		80	
	Resinite	7	×	Mandal Formation, Central Graben prone
	Chlorophyllinite	7	eo eo	algae of
Inertinite	Fusinite		Hydrogen Index	mixtures Waxy oil
	Semifusinite	_	출 40	Resin, and cuticle, cuticle, prone
(Type IV	Funginite	_	l	Land plants:
	Secretinite	-	20	TVDE III tissue,
kerogen)	Macrinite	\dashv		Altered 'Dead (oxidised) carbon'
	-	\dashv	0	0 50 100 150
	Micrinite	-	-	Changes with Oxygen Index maturation
	Inertodetrinite			

Presenter's notes: Footnotes—1. Outcrop, core, side-wall core, well cuttings samples at moderate thermal maturity (within the oil window 0.5 to 1.3% Ro); 2. Sample processed with HCl and HF acids. 3. Using transmitted light and kerogen concentrates, it may not be possible to subdivide the vitrinite group into macerals; therefore vitrinite must be used. This may also apply to macerals within the inertinite group. 4. Bituminite is a defined ICCP maceral term, variety amorphinite is more commonly applied to this type of DOM and and can be further expanded upon using the following recommendations: (i) fluorescing (fluoramorphinite) and non-fluorescing (hebamorphinite) amorphinite (Sentfle et al., 1987) or (ii) Types A, B, C, D (Thompson and Dembicki, 1986). May 22/02

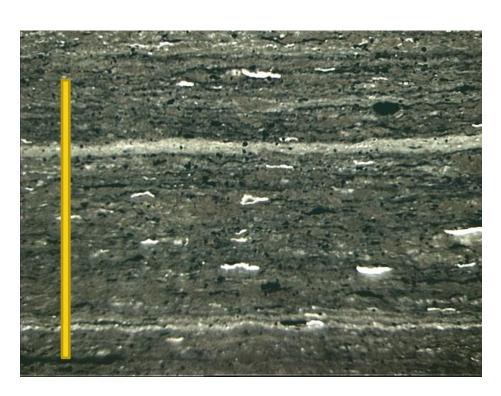
2.0 CLASSIFICATION EXPANSION OF ALGINITE MACERAL FOR ORGANIC FACIES

Maceral		Maceral		
Group	Maceral	type	Maceral Variety	Maceral Sub-variety
Liptinite	Alginite	Telalginite	Botryococcus	Tenui-Botryococcus; Large diameter/length colonies, Usually >50 micron, cell walls are highly thickened
p	7	retuige	Jour you out the	Crassi-Botryococcus; Small diameter/length colonies, Usually 5 - < 50 microns, cell walls are thins,
			Tasmanites	Large diameter/length, usually > 75 microns, thick walled, cyst - like
				Small diameter/length thin usually 5 microns - < 50 microns
			Leiosphaeridia	Large diameter/length, usually > 75 microns, thick walled, cyst - like
				Small diameter/length thin usually 5 microns - < 50 microns
			Pila	
			Reinschia	
			Gloeocapsa	Large diameter/length colonies, Usually >50 micron, cell walls are highly thickened; forms stromatolites/ algal mats
				Small diameter/length colonies, Usually 5 - < 50 microns, cell walls are thins,
			Pediastrum	
			Acritarchs	Acanthomorphic-like
				Veryachium -like
				Michysridium - like
			Dinoflagellates	theca; non resting cells
				chorate cysts Restricted October 2013 1177
		Lamalginite	Filamentous	

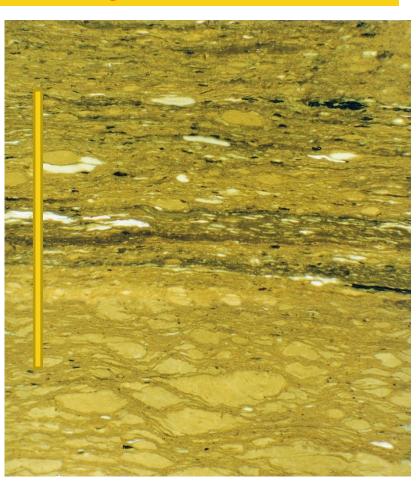
2.0 CLASSIFICATION EXPANSION OF MICROFOSSIL AND ZOOCLAST 'MACERAL GROUP' FOR ORGANIC FACIES INTERPRETATION

Maceral Group	Maceral	Maceral type		
	biogenic siliceous			
Non-organic Microfossils	microfossils	radiolaria		
		Diatoms, silicoflagellates;		
		chrysophytes		
		tentaculites		
	Pyritized microfossils	algal filaments and cells		
		forams		
		wood		
	phosphatic microfossils	conodonts		
		pellets		
		bone		
	calcareous micro- and	Coccoliths, calcareous		
	nanoplankton fossils	nanoplankton,		
		microforams		
		cricoconarids		
Zooclasts	Graptolite	telagraptolite		
		collograptolite		
	Chitinozoa			
	Scolecodont			

2.0 THE DISTRIBUTION OF ORGANIC MATTER TYPE, QUALITY, QUANTITY, AND INORGANIC MICROFOSSILS (E.G., BIOGENIC SILICA) CAN VARY SIGNIFICANTLY VERTICALLY AND LATERALLY IN LRS



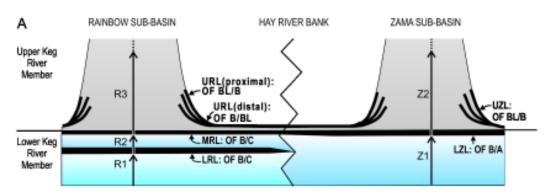
UV excitation, polished whole rock, perpendicular to bedding



Blue light excitation, polished whole rock, perpendicular to bedding

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2.0 THE DISTRIBUTION OF ORGANIC MATTER TYPE, QUALITY, QUANTITY, AND INORGANIC MICROFOSSILS CAN VARY SIGNIFICANTLY VERTICALLY AND LATERALLY IN LRS



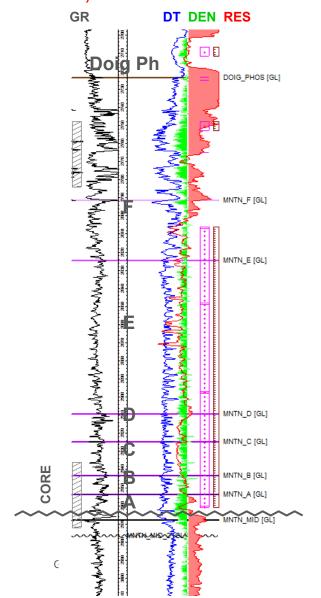
В		Rainbow	Zama Sub-basin			
Controls	Lower Laminite (LRL)	Middle Laminite (MRL)	Upper Lam (Distal Foreslope)	(Proximal	Lower Laminite (LZL)	Upper Laminite (UZL)
Preservation (anoxia)	Primary	Primary	Primary	Secondary	Primary	Secondary
Productivity			Secondary	Primary		Primary
Sedimentation rate	Minor	Minor	Minor	Clastic dilution	Minor	Clastic dilution
Supply of terrestrial organic matter		-	-		_	

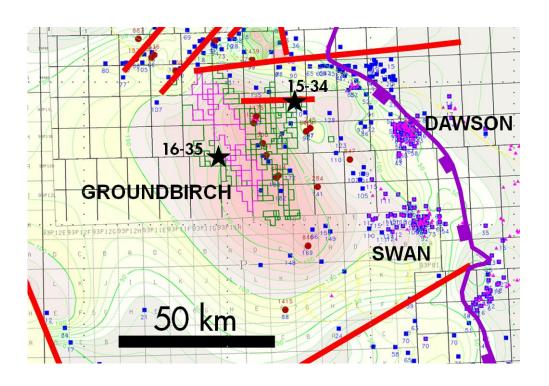
Figure 11. A)S chematic diagram illustrating the proposed correlation between the Rainbow and Zama sub-basins. Decametre-scale cycles are labeled R1, R2, and R3 in the Rainbow Sub-basin, and Z1, and Z2 in the Zama Sub-basin. LRL = lower Rainbow laminite, MRL = middle Rainbow laminite, URL = upper Rainbow laminite, LRL = lower Zama laminite, URL = upper Zama laminite OF = organic facies. B) Summary of interpreted controls on organic matter accumulation and preservation in the Rainbow and Zama sub-basins. Refer to text for details.

Wiebe et al., 2013

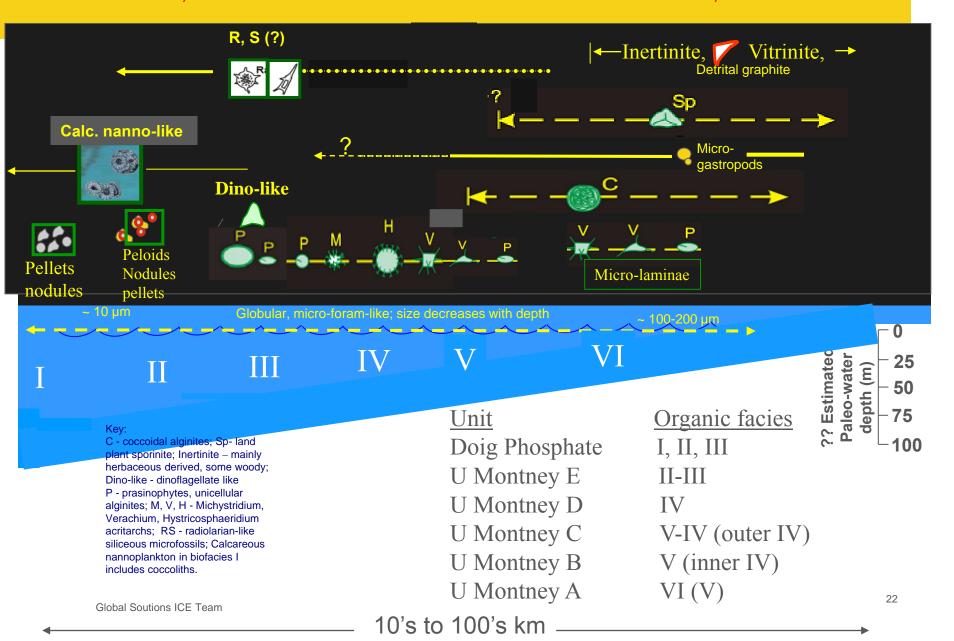
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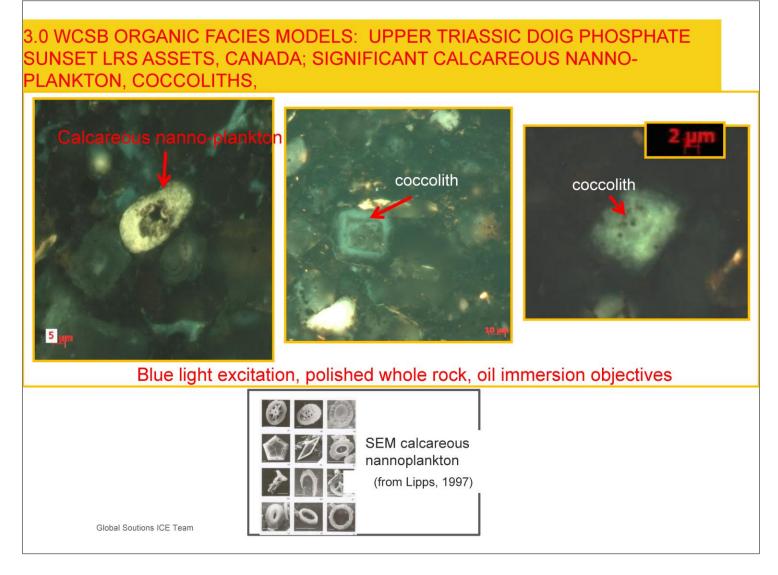
3.0 WCSB ORGANIC FACIES MODELS: UPPER TRIASSIC, DOIG & UPPER MONTNEY FMS; GROUNDBIRCH & SUNSET SHALE GAS ASSETS, CANADA





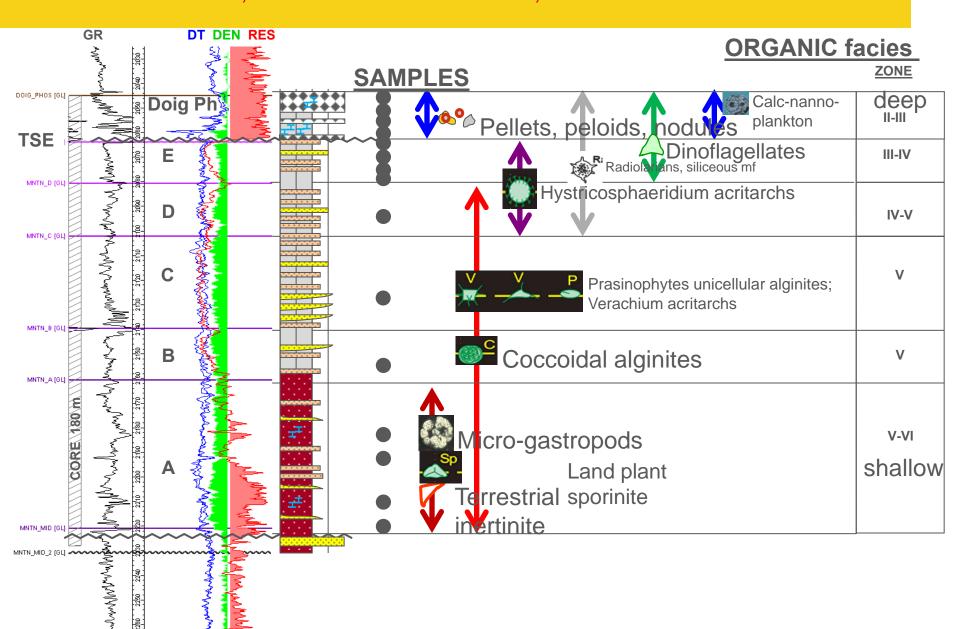
3.0 WCSB ORGANIC FACIES MODELS: UPPER TRIASSIC, DOIG & UPPER MONTNEY FMS; GROUNDBIRCH GAS & SUNSET LRS ASSETS, CANADA



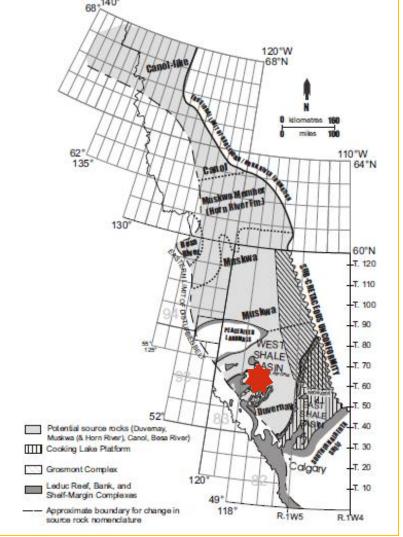


Presenter's notes: What are calcareous nannoplankton? The calcareous nannoplankton are unicellular photosynthetic protists, members of the group Haptophyta. They may take on many different shapes, but all have shells composed of tiny, calcium carbonate plates called "coccoliths." These tiny plankton are found mostly in tropical marine environments and are important primary producers. After the organisms die, their coccoliths accumulate on the bottom of the ocean, forming calcareous oozes that can harden into chalks and other fine-grained limestones. Fossils of calcareous nannoplankton have been found in limestones and chalks since the Jurassic.

3.0 WCSB ORGANIC FACIES MODELS : UPPER TRIASSIC DOIG AND UPPER MONTNEY, SUNSET LRS ASSETS, CORE 180 M



3.0 WCSB ORGANIC FACIES MODELS: UPPER DEVONIAN DUVERNAY FORMATION – LRS PLAY IN WCSB



SCAN
Kaybob well,
Fox Creek
Play area

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3.0 WCSB ORGANIC FACIES MODELS: UPPER DEVONIAN DUVERNAY FORMATION SHALES

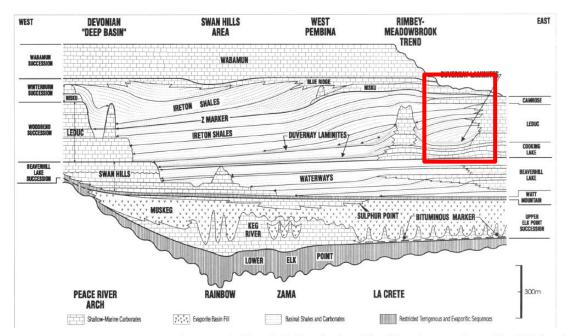
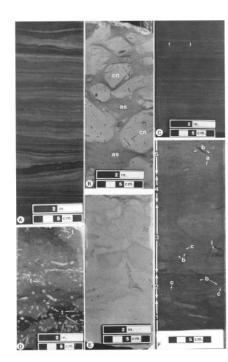


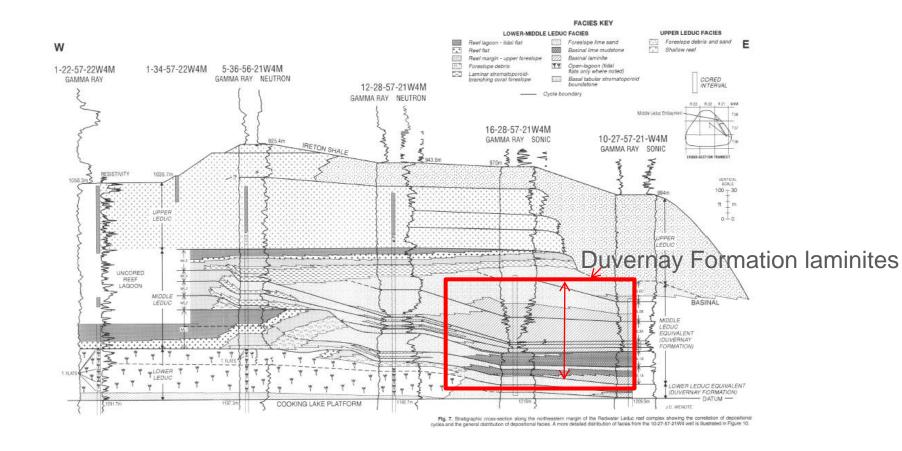
Fig. 2. Composite schematic cross-section across the Alberta Basin illustrating the cyclicity of Devonian successions and the distribution of major facies. The portion of the cross-section above the base of the Watt Mountain Formation is from central Alberta and corresponds to the geographic locations listed above the section. The lower portion of the cross-section, below the Watt Mountain Formation, is from northern Alberta and corresponds to the geographic locations listed below the sections. The bituminous marker is part of the Upper Elk Point succession and the Duvernay laminites are part of the Woodbend succession.



Sediment laminous consist of personal services of the control of t

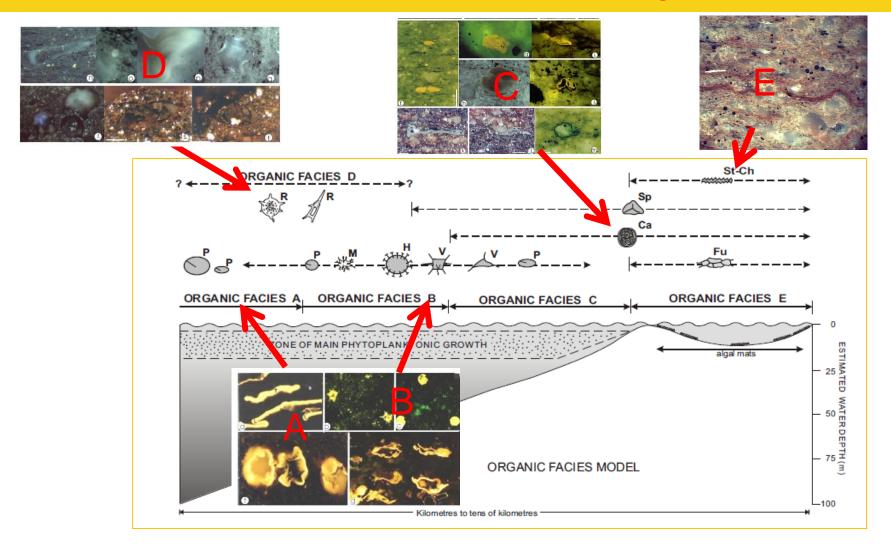
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3.0 WCSB ORGANIC FACIES MODELS: UPPER DEVONIAN DUVERNAY FORMATION AT RIMBEY LEDUC REEFS

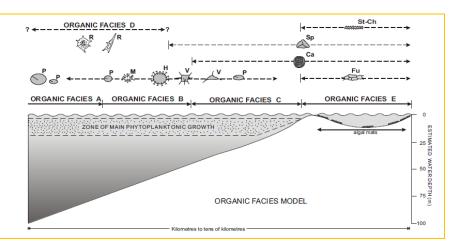


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3.0 WCSB Organic Facies Model for Devonian shales, based on macerals and siliceous microfossil assemblage



3.0 WCSB ORGANIC FACIES: UPPER DEVONIAN DUVERNAY FORMATION – LRS; VERTICAL VARIATION IN ORGANIC FACIES AT RIMBEY LEDUC REEFS



OF and TOC's are related to fore-stepping and back-stepping of reef, water depth

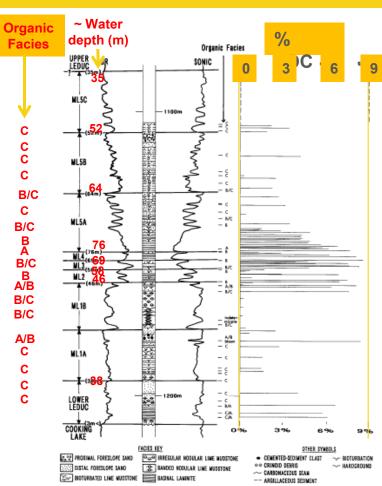
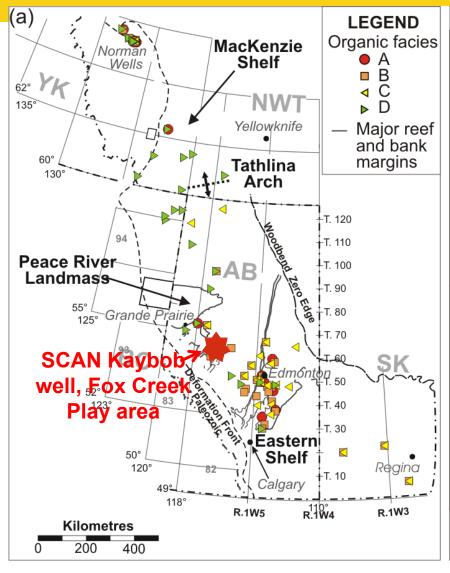
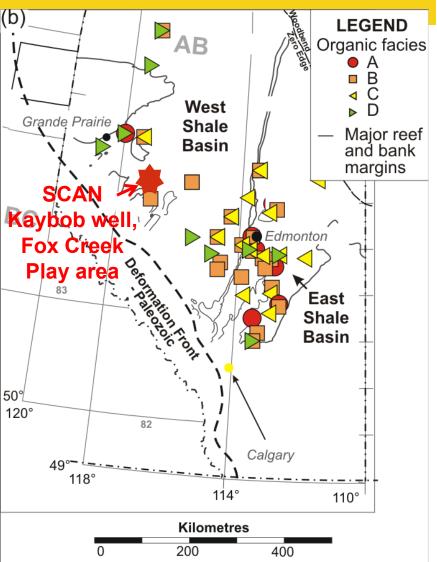


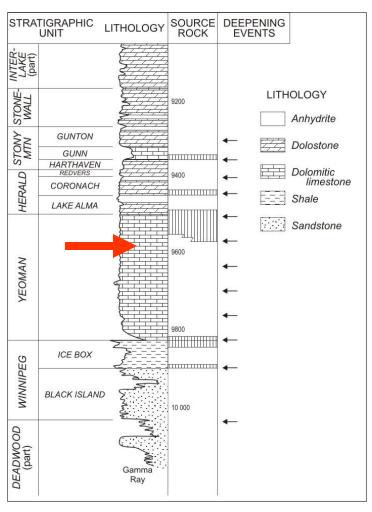
Fig. 10. Lithologio-wireline log section of Duvernay strats in the 10-27-57-21W4 well showing the depositional cycles and facies, the gammaray and sortiolog signatures, organic facies and TOC. Proximal forestope sands are paloidal lime grainstones, whereas distal forestope sands are paloidal and skeletal lime packatones. The numbers in parentheses refer to the estimated water depths at the top of the major depositional cycles.

3.0 WCSB ORGANIC FACIES; DUVERNAY FORMATION REGIONAL VARIATION IN ORGANIC FACIES





3.0 ORGANIC FACIES IN MARINE PETROLEUM SOURCE ROCKS, UPPER ORDOVICIAN, WILLISTON BASIN: SUBTIDAL LIME MUDSTONES, VERY HIGH TOC'S, TYPE I AND II KEROGEN



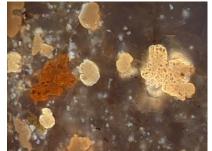




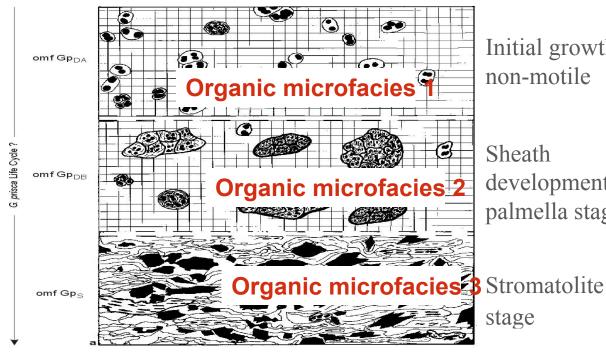


3.0 Paleodepositional Model for *G. prisca* alginite in Upper Ordovician lime mudstones, Type I and II kerogen





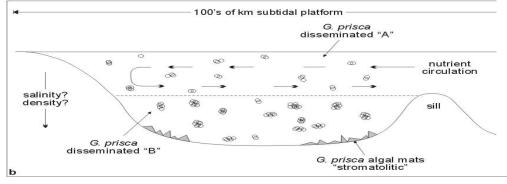




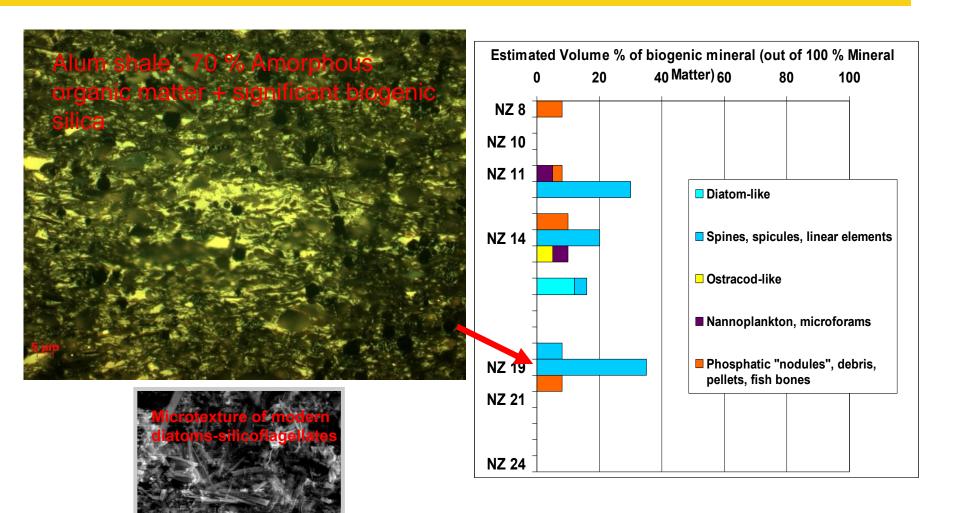
Initial growth – non-motile

Sheath development – palmella stage

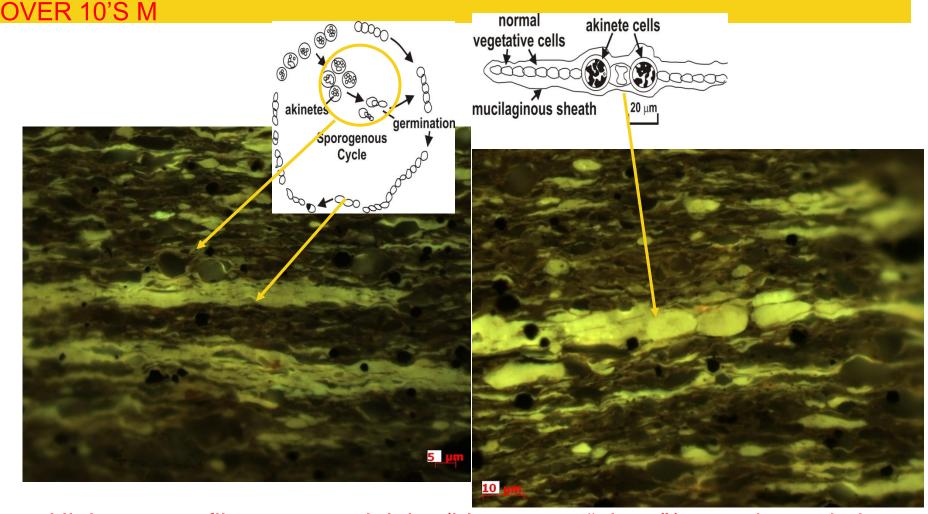
stage



3.0 VERTICAL VARIATIONS IN INORGANIC MICROFOSSIL, BIOGENIC SILICA, IN CAMBRIAN ALUM SHALE, SWEDEN, OVER 10'S M

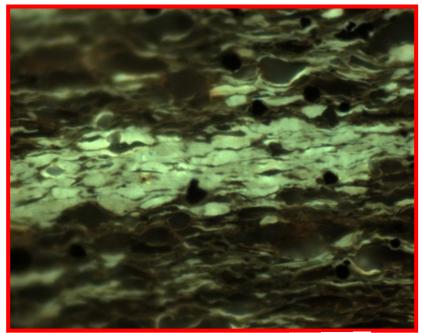


3.0 VERTICAL VARIATIONS IN HC POTENTIAL AND TYPE CONTROLLED BY DOM CONTENT – ALGINITE TYPE, IN CAMBRIAN ALUM SHALE, SWEDEN,



High amount filamentous alginite (blue-green "algae"/ cyanobacteria in vegetative state with akinete cell-heterocysts, ~ tannins) with small prasinophytes + amorphous organic matter

3.0 VERTICAL VARIATIONS IN HC POTENTIAL AND TYPE CONTROLLED BY DOM CONTENT – ALGINITE TYPE, IN CAMBRIAN ALUM SHALE, SWEDEN, OVER 10'S M, CAMBRIAN ALUM SHALE SWEDEN



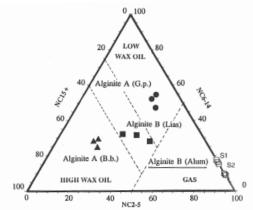
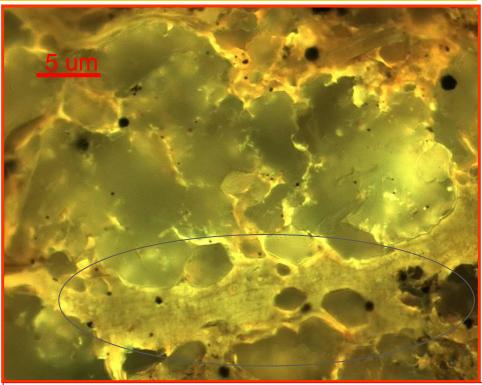


Figure 9. Distribution of normal paraffins in the products released and residues of artificially matured Alum Shale kerogen, as compared to other alginites.

From Baharti and Larter, 1995



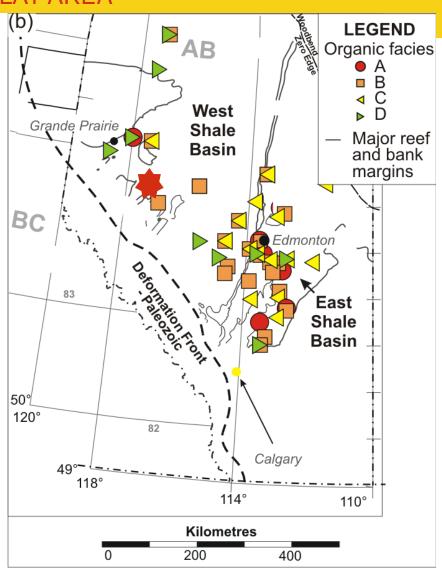
Alum shale OF with filamentous alginite (blue-green "algae"/ cyanobacteria in vegetative state with akinete cellheterocysts, ~ tannins), with small prasinophytes + amorphous organic matter

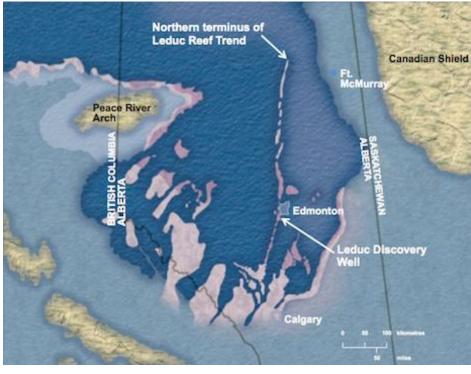


Appendix: Additional Slides

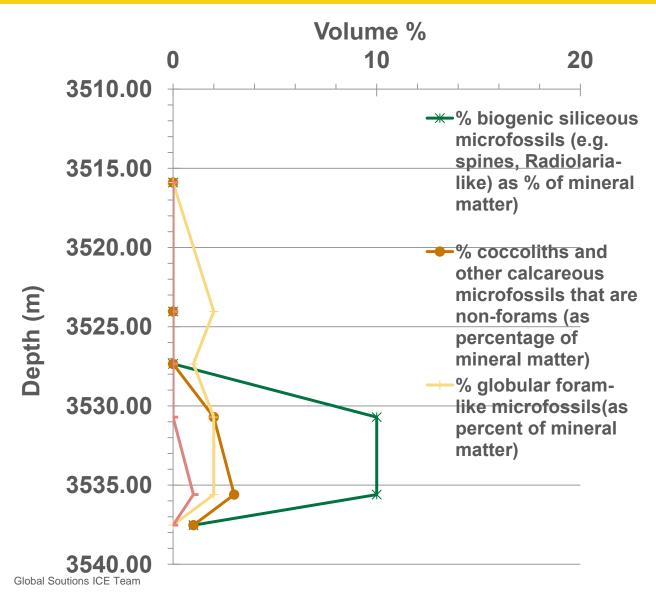
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ORGANIC FACIES IN DUVERNAY SCAN KAYBOB WELL, FOX CREEK PLAY AREA

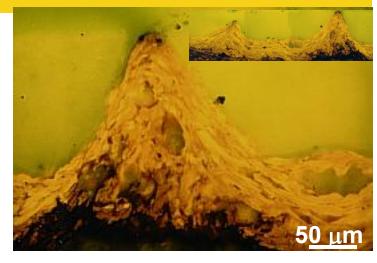


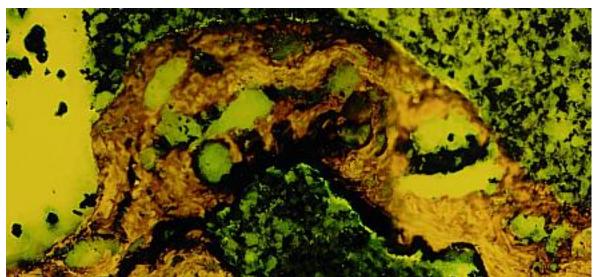


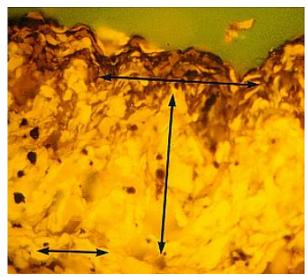
E.G., VERTICAL CHANGES IN SILICEOUS AND CALCAREOUS MICROFOSSILS – IN LRS USED IN PART TO DEFINE ORGANIC FACIES, PALEOZOIC.



4.0 Stromatolite micro-textures, *G. prisca* alginite in Upper Ordovician lime mudstones

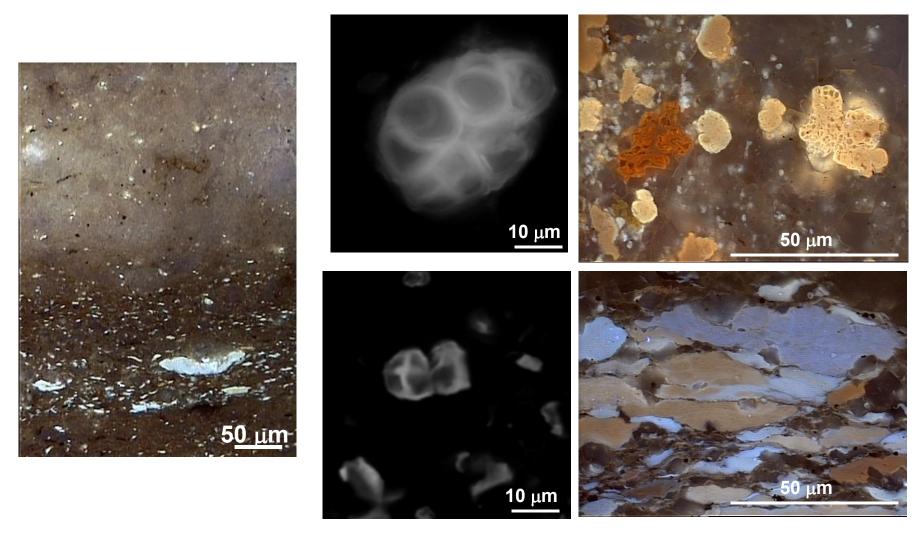






(Stasiuk et al , 1993, Org. Geochem; 1999, Geoscience Canada)

Vertical variations in organic microfacies & HC potential in Upper Ordovician, marine petroleum source rocks, Williston basin, Saskachewan. Subtidal, lime mudstones with average to very high TOC's, Type I & II kerogen, comprising microlaminated, disseminated coccoidal alginite w/ spiny acritarchs, and layered coccoidal alginite,



(Stasiuk et al , 1993, Org. Geochem; 1999, Geoscience Canada)

3.0 WCSB ORGANIC FACIES MODELS UPPER TRIASSIC UPPER MONTNEY TO DOIG ORGANIC FACIES: LRS - SHALE GAS ASSETS

