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Shale gas characteristics of Permian black shales in South Africa: results from recent drilling in the Eccra Group (Eastern Cape)

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Abstract

Detailed lithological, sedimentological, petrographic and geochemical analyses of the lower three formations of the Eccra Group in the Greystone area, Eastern Cape, South Africa, close to the tectonic front of the Cape Fold Belt, are described with a particular focus on the black shales of the Whitehill Formation. Fresh core samples from a 300m deep borehole were analysed as follows: petrography, XRD and XRF, open system pyrolysis and thermovaporization, TOC/Rock Eval, SEM microscopy, vitrinite reflectance, stable isotopes and porosity using mercury intrusion. This ongoing investigation gives insight into the gas potential of the Karoo black shales.

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1. Shale Gas in South Africa

South Africa has recently expressed interest in natural gas resources trapped in deep underground shales of the Karoo Basin. An optimistic prospective area of 183 000 km² was originally suggested by Kuuskraa et al. 2011 [1] but a smaller area of 155,000 km² is being considered related to thinning of

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Karoo Formations to the north. The most recent resource assessment conducted by Decker and Marot, 2012 [2] predicts that the Southern Karoo has potential reserves between of 32Tcf to 485Tcf, and that the most conservative prediction is still a significant gas resource. Thermal degassing of the shale, fluid invasion or natural fracturing (gas escape) by the intrusion of the Karoo dolerite suite and/or by the thermo-tectonism related to the formation of the Cape Fold Belt (CFB) (Fig. 1), has not yet been incorporated into these assessments.

The aim of this study is to investigate the lower Ecca Group of the Karoo Supergroup some 200km northwest of Port Elizabeth (Fig. 1) using various analytical techniques to identify characteristics that would classify the shales as a potential resource for unconventional gas, where they occur close to the CFB. Samples for analysis of the Prince Albert, Whitehill and Collingham Formations were obtained from a borehole drilled to 300m below the surface, near the basin edge in the foreland of the Cape Fold Belt. Details of this drilling operation can be found on www.karooshalegas.org.

2. Regional geology of the Karoo Basin

2.1. Karoo Basin

The Karoo basin of South Africa covers around 300 000km² and represents about 100 million years of sedimentation, from ca. 280Ma to ca. 180Ma. The onset of deposition of Karoo sedimentary rocks occurred during the late Carboniferous across Gondwana (Fig. 1), and continued until the on-set of the break-up of this supercontinent during the Middle Jurassic (ca. 183 Ma). The Karoo basin has been interpreted as a foreland trough formed during the shallow-angle subduction of the paleo-Pacific plate beneath Gondwana supercontinent. This lead to the building of the wide fold thrust belt, which is referred to today as the Cape Fold Belt (CFB) [3]. This model has been contested by Tankard et al., 2012[4] who suggests that the CFB is a strike slip belt and the adjacent Karoo Basin more akin to a flexural foreland basin. By contrast Lindeque et al., 2011[5] and Pángaro and Ramos, 2012 [6] proposed that the CFB may be a [Jura-type] fold belt that formed as a consequence of arc-continent collision with subduction to the south.

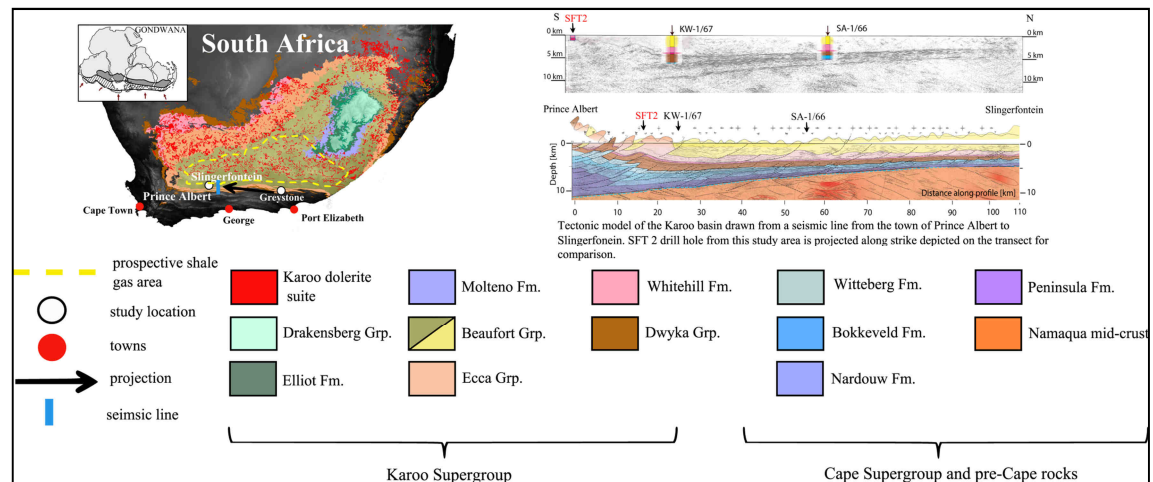


Fig. 1. Seismic profiles from the town of Prince Albert to Slingerfontein (upper right inserts from a study conducted by Lindeque, 2011 [5]) provides the basis for a new tectonic model for the Karoo Basin, its stratigraphy and structure of the upper crust. Our drill hole (SFT2) has been projected to the west (arrow in left top insert) from where the study was conducted (Greystone area), into the seismic section.

2.2. Deposition of Karoo Sedimentary Rocks

Glaciation of southern Africa occurred when Gondwana drifted over the South Pole and a large ice-sheet covered the south of the continent. At the end of this glaciation period an extensive shallow lake formed, which was fed by meltwater resulting in the deposition of the Dwyka diamictites [7]. Dropstones are found at the bottom of the overlying Ecca Group (Prince Albert Formation) due to the gradational contact with the Dwyka Group. The Prince Albert Formation is composed of mudstone with shales and some small sandstone units. The overlying Whitehill Formation comprises fine-grained, finely laminated black organic rich shale which weathers white due to subaerial pyrite oxidation to gypsum. The sediments are considered to be deposited in an anoxic environment [8]. Besides pyrite, the shales contain dolomite lenses near the base (Fig. 2). The rocks are highly folded and faulted, and thus, interpreted as 'decollement' [9], [5]. The Collingham Formation comprises dark grey mudstones, intercalated with thin yellow clay-like layers of ashfall tuff (K-bentonite), with zircons that have been dated at ca. 274-270 Ma (eg. [10]) (Fig. 2). The mudstone beds are generally parallel laminated, but often exhibit flaser and wavy bedding at the contact of the mudstone and the tuff. The mudstone contains trace fossils such as Planolites and other epichnial grooves. At the top of the formation the mudstone grades into sandstone which contains plant impressions (sp. *Glossopteris*) [11]. Pyrite is common in the lower half of this formation.

The early Ecca deposition took place during the early stages of the Cape Orogeny (~250Ma), when mountain ranges developed in the far south. Subsequently, material from these mountain ranges, as well as from the highland areas in the north east and the west, drained into the basin to form the upper Ecca turbidites and prograding deltas [7]. Many authors have inferred that the Dwyka diamictites and lower Karoo were deposited in an open marine setting (eg.[7], [8]), while others have proposed a marine-lacustrine setting [12]. Alternatively only a short marine ingressions related to eustatic sea level rise occurred as a response to global deglaciation [13]. After the later progressive basin filling and the deposition of delta-slope sediment, the lacustrine period changed to a fluvial environment during deposition of the Beaufort Group. The northerly progression of the Cape Fold Belt resulted in the mountain range encroaching inland and forcing the fluvial deposition for most of the overlying Stormberg Group farther northwards. During that time the climate changed to semi-arid [14]. The increase of more clastic sediments resulted in coarser-grained sandstones that comprise the upper sequences such as the Katberg Member and the Molteno Formation. In the final stages of deposition there were wadii and playa lake type environments forming the Elliot Formation, and sand dunes that dominated the Clarens Formation. The volcanic activity of the Drakensberg Group brought the Karoo depositional sequence to an end at ca 183 Ma [15].

3. New borehole core from the Greystone Area

The study site is located in the Eastern Cape Province, south of Jansenville in the Greystone area (Fig.3). This area was selected for mapping (at scale 1:50000), structural and sedimentary evaluation, core drilling and geochemical and petrological analysis of core samples. Two boreholes were drilled, the first (SFT1) to a depth of 100m and the second (SFT2) to a depth of 300m of which the first 100m was percussion drilling.

The drill location was selected in the region where the Prince Albert, Whitehill and Collingham Formations were predicted to occur close to the surface. The first borehole (SFT1) was highly weathered in the first 40m and core was only sampled, therefore, from the second borehole (SFT2) below 100m from the surface. Simplified logs of SFT2 are shown on Figure 2.

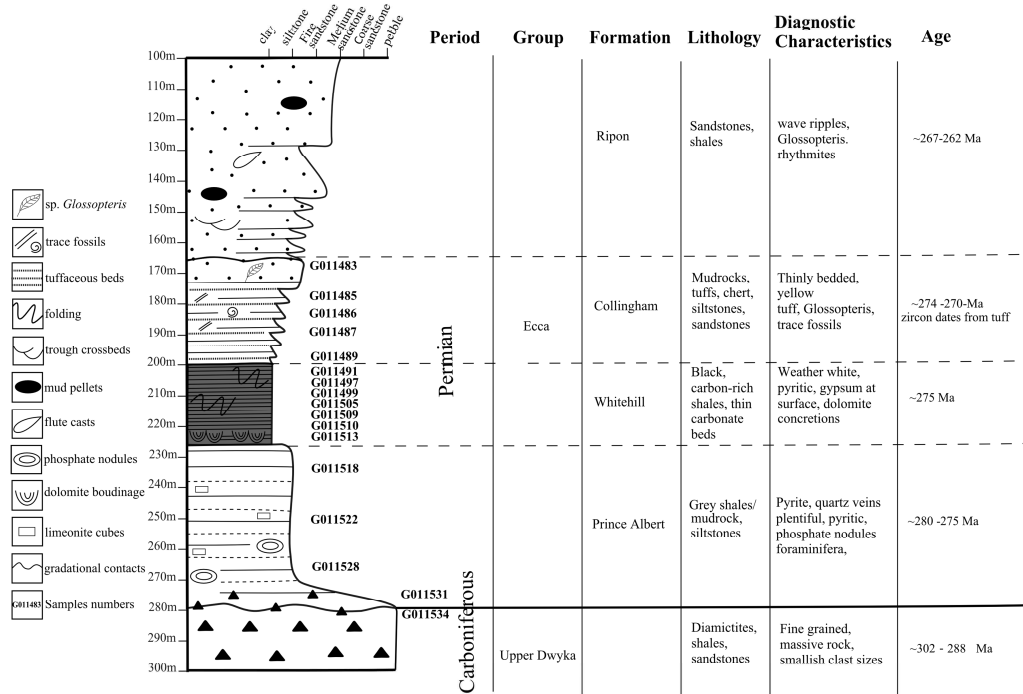


Fig. 2. Lithological log compiled from core and field data, showing the location of the core samples eg. G011483. Age dates have been acquired from Fildani, 2009 [10].

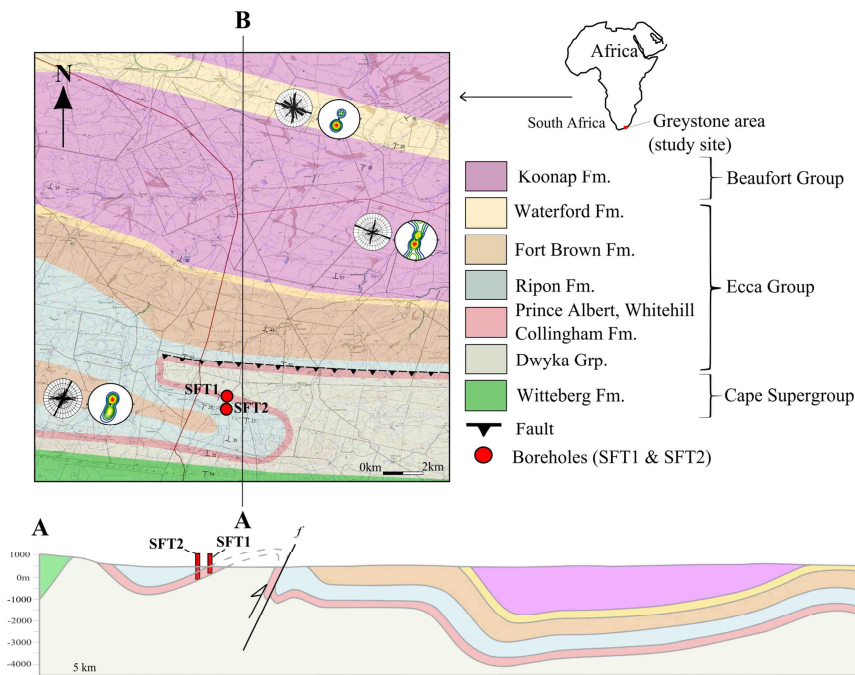


Fig. 3. Geological map of the study area, created with QGIS, accompanied with a cross section running through the transect A-B, showing the location of two boreholes drilled during this study (SFT1 and SFT2). Stereoplots (poles to bedding) shows the orientation of fold axes and rose diagrams depict overall jointing.

4. Results

4.1. Lithology and Petrology

The Prince Albert Formation conformably overlies the Dwyka Group and is mostly an olive-grey mudrock (Figures 2 and 4c). A variety of foraminifera were observed under SEM (Figure 4g) and phosphate and carbonate lenses were identified in the field) Oxidized iron rich bands are found between mudstone layers which is representative of weathered pyrite. The Whitehill Formation is mostly a white weathering black shale with dolomite concretions at the base of the formation. The shales comprise mostly clays (eg. illite), which is seen to be tightly packed in SEM micrographs. SEM micrographs of the dolomite show a great degree of porosity. The Collingham Formation, a grey mudstone, is distinctly jointed, well layered and intercalated with tuff in the field. Under light microscopy, the boundaries between clay minerals and the tuffs are distinct, while under SEM framboidal pyrite is detected close to the contact with the Whitehill Formation.

4.2. TOC/Rock Eval

TOC average values are 4.5% in the Whitehill Formation. The results are represented in Figure 5 in depth profiles. Rock Eval data consists of Tmax, OI and HI, S1 and S2 values. Tmax is the analytical temperature at which hydrocarbons are released from kerogen and reveal a proxy of thermal maturity. Tmax values lower than 435°C indicate immature organic material, between 435 °C and 455°C considered mature organic matter and over 470°C represents the wet gas zone or over mature organic matter. The lower Ecca Group has high Tmax values indicating over-maturity (average 406 °C-572°C).

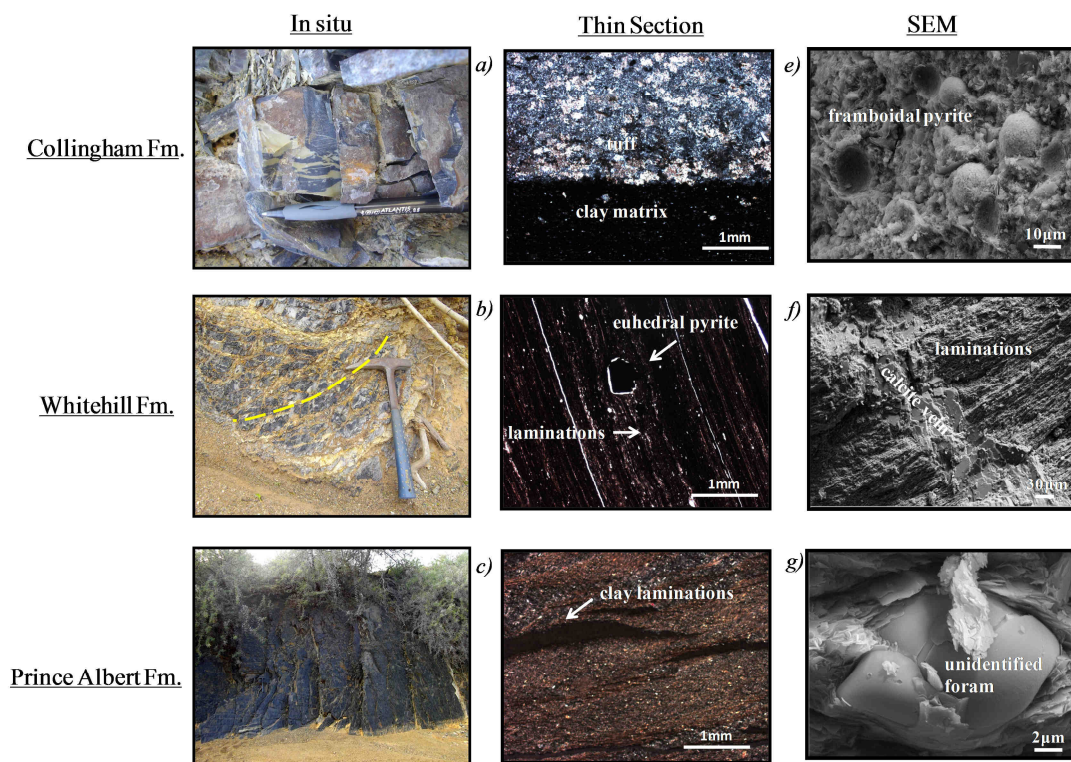
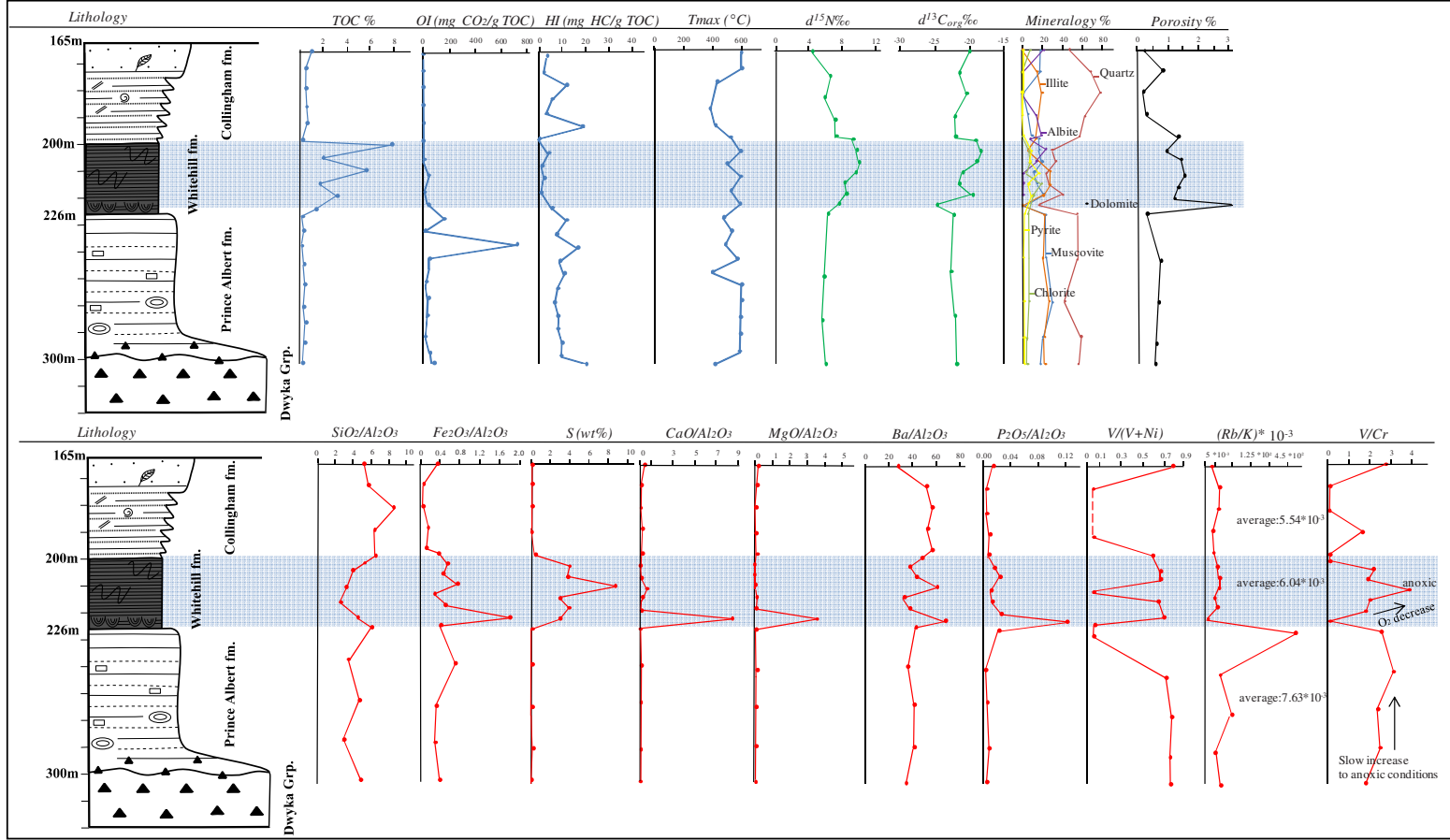


Fig. 4. Photographs showing typical outcrops of the Prince Albert, Whitehill and Collingham Formations and representation of their features seen in petrographic section and SEM micrographs of fresh core.

Fig. 5. Depth profiles and chemostratigraphy across 135m of core from SFT2.



HI ((S2/TOC)·100) versus OI ((S3/TOC·100) provides an indication of Kerogen type. Aquatic organic matter has a high HI content whereas terrestrially derived organic matter a low HI, but high OI. The variations of HI values can represent the changing setting of depositional environments. Results are plotted on a modified van Krevelen Diagram (Fig. 6).

4.3. XRD and XRF

Quartz/mica ratios (expressed as SiO₂/Al₂O₃) are the highest in the Prince Albert and the lowest in the Whitehill Formation (Figure 5). Dolomite occurs at the base of the Whitehill Formation (peaks CaO and MgO).

Sulfur wt% and Fe₂O₃/Al₂O₃ ratios peak in the Whitehill Formation. On an iron-sulfur-TOC ternary diagram (Fig. 7) data from the Whitehill Formation plot in the anoxic 'zone'. Points along the stoichiometric pyrite line (S= 1.15 Fe) suggest that all of the iron has been fixed as pyrite [16]. Samples below the line suggest that sulfur occurs in oxidized forms eg. gypsum, barite, organic-S. XRD data confirms higher concentrations of pyrite in the Whitehill Formation.

Phosphorus, calcium and magnesium ratios all peak in the dolomite at the base of the Whitehill Formation. Rb/K ratios are on average for the Prince Albert Formation = 7.63x10⁻³, Whitehill Formation = 6.04 x10⁻³ and the Collingham Formation = 5.54 x10⁻³.

4.4. Stable isotope analysis ($\delta^{15}\text{N}$, $\delta^{13}\text{C}$)

The average $\delta^{13}\text{C}$ value for the Prince Albert Formations is -23‰ (-22.6‰ to -23.8‰), the Whitehill Formations is -20‰ (-18.9‰ to -24.7‰) and the Collingham Formation is -21.4‰ (-20.1‰ to -22.4). The $\delta^{15}\text{N}$ values are highest in the Whitehill Formation ranging from 7.7-10.1‰.

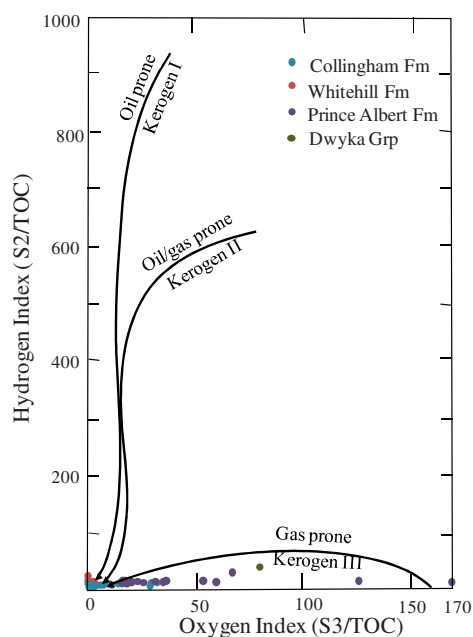


Fig. 6. Plot of HI vs OI index in a modified Van Krevelen Diagram (van Krevelen, 1950 [17])

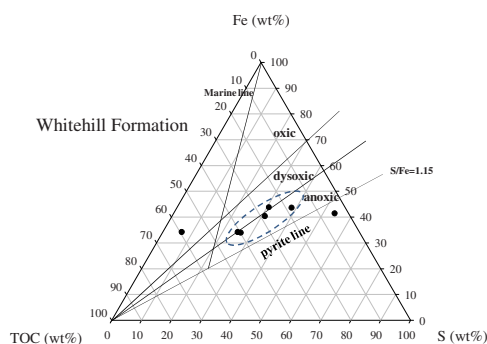


Fig. 7. Ternary diagram interpreting Fe-TOC-S (based on the original by Arthur and Dean, 1989 [16]).

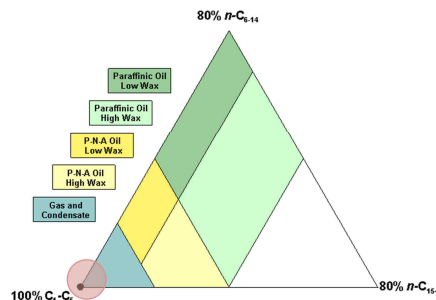


Fig. 8. Ternary diagram based on the Horsfield diagram plotting all the pyrolysis data in the C1-C5 range.

4.5. Open pyrolysis and thermovaporization

Due to the high maturity of the samples many of the chromatograms displayed peaks are too small to be measured with precision. It was decided instead to measure three major areas within the chromatograms for pyrolysis: C1-C5, C6-C14 and C15+. Pyrolysis data are represented in the Horsfield Ternary Diagram (Fig. 8). Thermovaporization data were recorded by measuring the combined C1-C5 peaks and any following individual peaks: C6, Benzene and Toluene.

4.6. Hg-intrusion porosimetry

The porosity (Fig. 5) is highest in the Whitehill Formation (1.57%), and in particular in the dolomite units (2.9 %). SEM images show finely crystalline dolomite with intracrystalline porosity. The Prince Albert Formation has an average of 0.5% porosity and the Collingham Formation has an average of 0.4% porosity.

4.7. Vitrinite reflectance

No macerals were detected under fluorescing light, likely due to the over maturity of the rock samples. Reflected light microscopy, on the other hand, revealed that solid bitumen occurred in abundance and their grey values were measured with a Zeiss Axioplan microscope and attached digital camera. There is a strong linear relationship between vitrinite reflectance (VRo) and bitumen reflectance (BRo) which allows a conversion from BRo to VRo provided that the bitumen reflectance is of high maturity [18], [19]. The reflectance measurements from the bitumen are on average ~ 4% Ro which classifies them as highly mature samples.

5. Discussion and conclusion

Geochemical and petrographic studies of the lower Eccra Group rocks in an area some 200km northwest of Port Elizabeth suggest that these rocks were deposited in stratified water bodies with oxygen-deficient conditions to times with an anoxic bottom water. Deposition of the Prince Albert Formation probably occurred under conditions of a short marine incursion in response to deglaciation.

The Whitehill Formation with a TOC content averaging 4.5%, fulfils basic prerequisites of successful gas-bearing shales [20], making it the prime focus for potential shale gas prospects in the Southern Karoo. The lower Eccra Group has high Tmax values indicating over-maturity. Assuming aquatic organic material as precursors of the today's organic material in the Collingham and Whitehill Formations both plot below the Kerogen II curve, a response due to thermal degradation. The Prince Albert Formation, on the other hand, falls within the Kerogen III boundary. Kerogen II is derived from phytoplanktonic organisms and Kerogen III mostly from terrestrial organic matter.

Barium and phosphorus peaks are proxies for palaeo-productivity. Phosphorous is thought to peak during periods of nutrient regeneration [21]. Phosphate burial is directly related to calcium content, in turn related to Ca-apatite, a diagenetic product of early diagenesis and indicator of intense organic matter mineralization. The XRF ratio peaks of calcium and phosphorous in the dolomite at the base of the Whitehill Formation suggest that the high porosity of the dolomite is due to coccolith trace fossils [22], and intense phosphogenesis. V/(V+Ni) and V/Cr are attributed to different redox conditions. V/(V+Ni) ratios within the Whitehill Formation are above 0.5, which indicates that it accumulated under anoxic conditions [23]; the values for the Prince Albert Formation suggest they accumulated mostly under

euxinic conditions. V/Cr ratios suggest a period of anoxia by peaking in the Whitehill Formation. Rb/K ratios indicate variations in palaeosalinity ($> 6 \times 10^{-3}$ is indicative of marine settings and $< 4 \times 10^{-3}$ freshwater to brackish conditions [24]. Rb/K ratios for the Prince Albert and the Whitehill Formation indicate marine salinity.

Terrestrial organic material ranges between -20‰ to -30‰ $\delta^{13}\text{C}$; and in the Karoo plants that range from -16‰ to -26‰ have been recorded [14]. Marine organic matter can fall in the range of -25‰ to -5‰ $\delta^{13}\text{C}$ [25]. The measured ranges in the core samples are between -20‰ to -23‰ are therefore not diagnostic of either marine or terrestrial organic matter. $\delta^{15}\text{N}$ values for terrestrial organic matter fall in the range of 0‰ to 5.5‰ , whilst $\delta^{15}\text{N}$ of marine organic matter in the range of 4‰ to 9‰ [25]. The $\delta^{13}\text{C}$ isotope values peak slightly in the Whitehill Formation. Enrichment of $\delta^{15}\text{N}$ values in the Whitehill Formation confirms an ancient anoxic setting. $\delta^{15}\text{N}$ values in the Whitehill Formation (7‰ – 10‰) also indicate the preservation of marine organic matter, but the number of analyses is small and needs further work.

Open pyrolysis values plot in the C1-C5 region of the Horsfield ternary diagram indicating emittance of short chained hydrocarbons. Thermovaporization analyses of the Whitehill Formation display chromatograms with peaks of C1-C5, C6, benzene and toluene hydrocarbons. It is possible that some of the C1-C5 hydrocarbons may have desorbed from the samples during grinding and powdering process.

Results from Rock Eval pyrolysis, vitrinite reflectance measurements, open pyrolysis and thermovaporization analyses carried out on core samples drilled through the three lowermost formations show that organic matter has reached an advanced stage of kerogen development. These rocks can therefore be classified as over-mature, likely because of the thermotectonic processes related to the Cape Orogeny overprint on lower Karoo rocks in the study area. It is possible that the maturity of the shales decreases farther north in the Karoo basin.

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