

Pseudovitrinite: Possible Implications for Gas Saturation in Coals and Surrounding Rocks

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INTRODUCTION

Coal Petrography is for many a black science using terms that are for some not only incomprehensible but also in flux. For utilization purposes, the organic component of coal is generally broken down into reactive and non-reactive macerals, often with little further differentiation of the macerals. The main incentive to differentiate further is the recognition that the degree of reactivity in vitrinite macerals and the degree of non-reactivity in inert macerals, both vary.

In terms of coalbed methane (CBM) studies it is important to consider separately gas generation, adsorption capacities and actual present gas contents (desorbed gas) in terms of the different macerals. Some CBM studies have divided coal into maceral groups that generally have different gas generation and adsorption capacities (Lamberson and Bustin, 1993). A host of environmental, as well as physical, parameters influence actual gas contents and there is no clear evidence that gas contents are strongly influenced by coal petrography (Bustin and Clarkson, 1998). Gurba *et. al.* (2001) have attempted to outline a more subtle maceral control on actual gas content in Permian Australian coals from the Gloucester basin, New South Wales. To date it appears that there is nothing in the form or texture of macerals that provides specific information about adsorption ability, actual gas contents or the environment in which coal macerals generated or retained gas.

This note proposes that a sub maceral of vitrinite called pseudovitrinite may flag changes in coal that influence its adsorptive capacity and maybe also influence its actual gas content. Pseudovitrinite is derived from grey textureless vitrinite (Photo 1) that is used for reflectance measurements and under present classification is referred to as collotelinite. It differs from collotelinite because of the presence of small elliptical slits of different sizes and different orientations (Photo 2). Pseudovitrinite is considered to be an alteration product of collotelinite derived by devolatilization, desiccation or oxidation or a combination of these processes. It is for this reason that it might at least flag changes in the adsorptive capacity of coal.

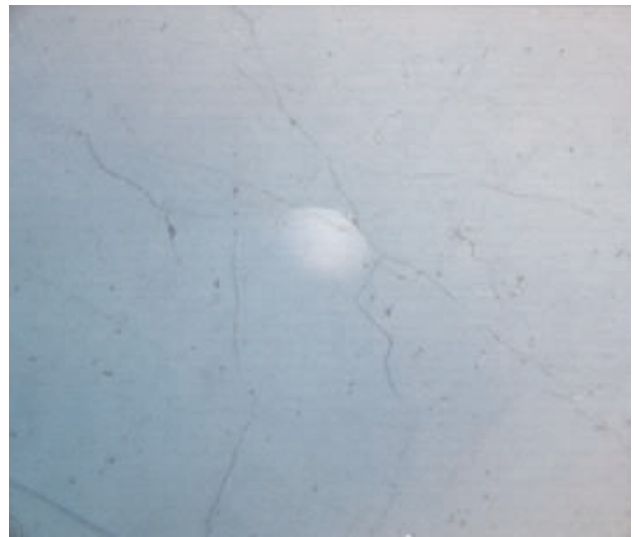


Photo 1. Collotelinite “normal vitrinite” used for rank determinations.



Photo 2. Pseudovitrinite in Comox and Gething Formation coals.

PRESENT VIEWS ON THE ORIGIN OF PSEUDOVRITRINITE

The origin of pseudovitrinite has been attributed to early oxidation, forming when vitrinite was still in a gel state (Benedict *et al.*, 1968). They considered it to act like an inert maceral during carbonization, but to be a separate sub maceral and different from oxyvitrinite, which forms during weathering of coal seams at surface. Kaegi (1985) considered pseudovitrinite to form insitu after coalification. Hagemann and Wolf (1989) considered it to form early in the maturation of coal and attributed it to gel desiccation and shrinkage. Lamberson (1993) states that none of the above explanations adequately explain the presence of pseudovitrinite in Gates Formation coals in northeastern British Columbia. Her data indicate that the ratio pseudovitrinite/vitrinite increases with the amount of total vitrinite in the samples (Figure 1). This might indicate a relationship between seam permeability and development of pseudovitrinite.

Kaegi (1985) demonstrated that pseudovitrinite formed in a sample heated in air in an oven at 50°C. Obviously this would initially dry the sample so that the production of pseudovitrinite that was seen may in part be caused by desiccation. Unfortunately Kaegi did not repeat the experiment at the same temperature in an inert atmosphere in order to distinguish between the effects of desiccation and oxidation. However his control samples, which were maintained at 23°C in an inert atmosphere, registered small increases in the amount of pseudovitrinite. It is apparent that some pseudovitrinite is formed by desiccation alone and does not require oxidation. Also it can form in vitrinite after coalification.

Pseudovitrinite is different from surface weathering of vitrinite, which produces low reflecting halos and fracturing. Kaegi (1985) suggests that pseudovitrinite becomes oxyvitrinite with progressive oxidation. It is certainly possible for vitrinite to become oxidized without going through the stage of first becoming pseudovitrinite, be-

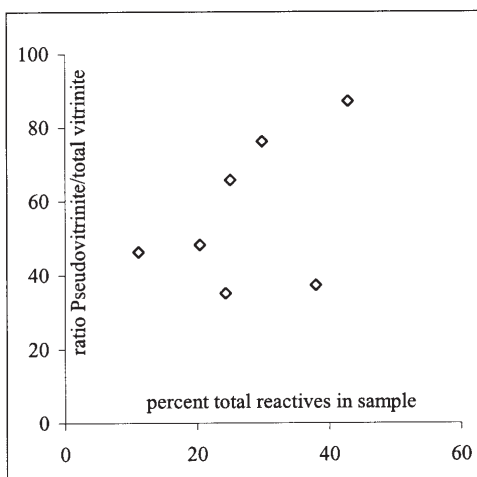


Figure 1. Relationship between amount of pseudovitrinite and total vitrinite in Gates coals from northeastern British Columbia data from Lamberson (1993).

cause there are lots of examples of weathered coals from the Mist Mountain Formation in southeastern British Columbia that do not contain pseudovitrinite.

Taylor, *et al.* (1998) conclude that pseudovitrinite is formed by mild long-term oxidation that can occur at depth in mines especially where coal is in contact with permeable sandstone roofs. They state that pyrite is not present in pseudovitrinite grains because it has been removed by oxidation. This is not always the case for samples from drill holes on Vancouver Island (Photo 3).

Diessel and Gammidge (1998) consider pseudovitrinite to form by drying under relatively shallow overburden. They suggest that coal once dried cannot regain its original moisture on re wetting possibly because of dewatered and collapsed pores. They also noted that in their study of Gates Formation coals and coals collected from the Bowen and Sydney coal basins, pseudovitrinite was only seen in Gates coals.

The reflectance of pseudovitrinite is typically 0.025% higher than that of vitrinite (Stach, *et al.*, 1982). Small increases in reflectance are also characteristic of low rank coals that have been dried (DeVanne, 2001). High temperature oxidation of coal increases reflectance where as low temperature weathering lowers reflectance. Surface oxidation is usually associated with a decrease in reflectance and some times a swelling of vitrinite grains producing micro fracturing.

DESCRIPTION AND OCCURRENCE OF PSEUDOVRITRINITE

Most authors consider pseudovitrinite to be a form of collotelinite characterized by elliptical slits. Other criteria mentioned by Kaegi (1985) include higher reflectance, stepped fracture patterns, higher relief and absence of pyrite inclusions. By far the most obvious characteristic is the presence of elliptical slits and for the purpose of this paper, they are the only criteria used to distinguished pseudovitrinite from collotelinite. In practical terms, when viewing a grain of collotelinite using a reflecting microscope, if a number of slits are present within a small viewing area of about 0.1 mm square, then the grain-count is



Photo 3. Pyrite in pseudovitrinite grains.

considered to be pseudovitrinite. Slits can be of any size ranging in length up to 2×10^{-5} metres long and up to 1×10^{-6} metres wide. They are often weakly aligned along one or 2 directions in the grains (Photo 4) and are therefore not always parallel to remnant layering in macerals. The angle between the two orientations appears to be variable. Generally slits do not show evidence of deformation such as s or z shapes. The slits only form in homogeneous macerals and ones that have higher volatile and moisture contents. They are therefore seen mainly in collotelinite and sometimes in collodetrinite but never in the inert macerals such as macrinite.

Samples from Vancouver Island, Gething and Gates formations in northeastern British Columbia and the Tulameen deposit in south central British Columbia were studied using an optical microscope and a Scanning Electron Microscope (SEM) at the University of British Columbia. Under reflected light the slits appear black and it is very difficult to determine if they are mineral filled. However using an SEM and back scattered electron images it is obvious which slits are mineral filled and which are not. In fact in many respects SEM images are the negative of reflecting microscope images. In the former mineral grains are bright and coal dark and in the latter the reverse is true.

Preliminary SEM work indicates that the majority of the slits are filled with minerals (Photo 5). EDS scans allow semi quantitative identification of some elements present in minerals. Scans indicated that most of the slits are filled with kaolinite (Figure 2) and some of the larger slits filled with calcite or dolomite (Figure 2). Slits in the high-volatile A coals from the Comox Formation often form 2 directions one parallel compositional layering and one across it. The slits parallel layering are filled with kaolinite and those across layering with carbonate, indicating the possibility of 2 generations of slits. In the low-volatile bituminous Gething Formation coals, slits are generally smaller and are filled mainly with kaolinite. It was difficult to identify any unfilled slits and those that were tentatively identified could be original cell structure and not slits, consequently it appears that of the Gething and Comox samples examined nearly all the slits are mineral filled. Any slit-like structure



Photo 4. Pseudovitrinite with multiple orientations of slits.

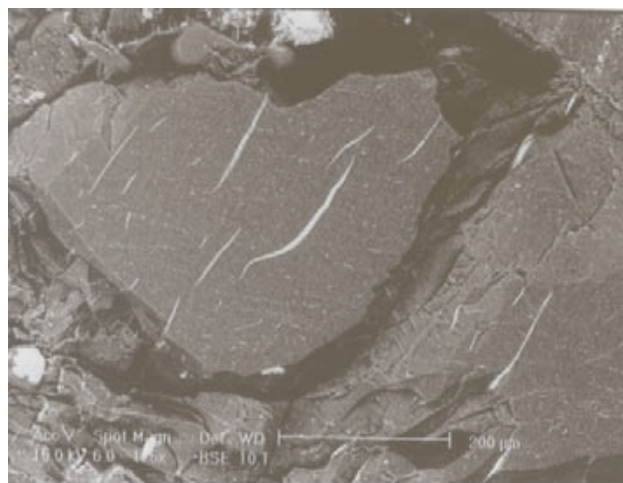


Photo 5. Mineralized slits back scattered electron images scanning electron microscope.

in collotelinite not parallel to remnant layering almost certainly indicates pseudovitrinite.

COMMENTS ON THE APPARENT ORIGIN OF PSEUDOVITRINITE

The occurrence of pseudovitrinite in “fresh coal” from mines and open pits in the Gates and Gething formations coals from northeast British Columbia has been documented by many authors (Lamberson, 1993 and Diessel and Gammidge, 1998). Most of the samples in which pseudovitrinite was identified came from near surface exposures either test pits or surface mines. However samples from the Comox Formation discussed in this paper came from drill holes intersecting coal at depths up to 500 metres and though the amount of pseudovitrinite decreases with depth it is still present at a depth of 486 metres (Figure 3). A

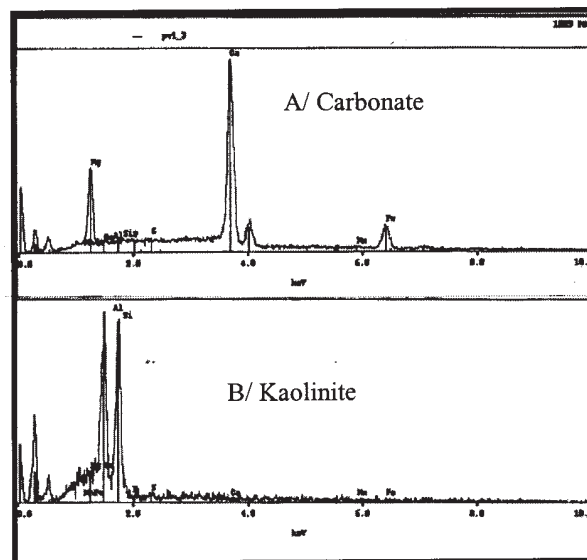


Figure 2. EDS scans of minerals in slits; A/carbonate and B/kaolinite.

similar pattern of decreasing pseudovitrinite with depth is seen in samples of Gething coal collected from a shallow drill hole in northeastern British Columbia (Figure 3). The amount of pseudovitrinite correlates in part with the amount of vitrinite in the sample, which is also seen in the Gates samples from Lamberson (1993). The Comox samples are close to 100% vitrinite macerals on a mineral matter free basis so there is no relationship between the amounts of pseudovitrinite and vitrinite in the samples.

Samples of Gates coals from a number of deep holes were provided to the author by industry. Initial petrography on these samples measured the percentage of pseudovitrinite compared to the total of collotelinite plus pseudovitrinite. Pseudovitrinite is present in samples covering a depth range of 600 to 2000 metres with little relationship to depth. The percent of collotelinite classified as pseudovitrinite varies from 39% to 14% in the samples (Figure 4).

The presence of slits in collotelinite collected from a range of depths indicates that they are not necessarily formed close to surface. Also the presence of mineral filling in slits indicates that they did not form recently and have not formed as a result of bringing the drill core to surface and suddenly relaxing the insitu stress. Spears and Caswell (1986) studied mineralization on cleats. They state that kaolinite forms at temperatures in the range of 55° to 100°C and calcite at around 100°C or at higher temperatures. This would suggest that kaolite is deposited in slits as the coal traverses the ranks sub bituminous to high volatile bituminous and calcite and other carbonates at ranks of high volatile bituminous or higher. Dating the filling of slits does not necessarily date the time of formation of slits with reference to coalification, but it is unlikely that slits would remain open unless mineral filled soon after formation.

Slits do not reveal evidence of excessive deformation, which seems to dispel the idea of early formation. They may form as a result of oxidation or desiccation of vitrinite (Kaegi, 1985) or by devolatilization. In that all coals lose volatile matter as rank increases and not all medium-volatile bituminous coals contain pseudovitrinite it appears that

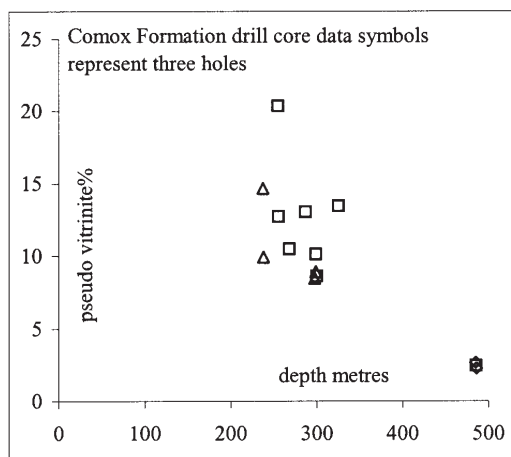


Figure 3. Depth versus percent pseudovitrinite for Comox coals and Willow Creek coals.

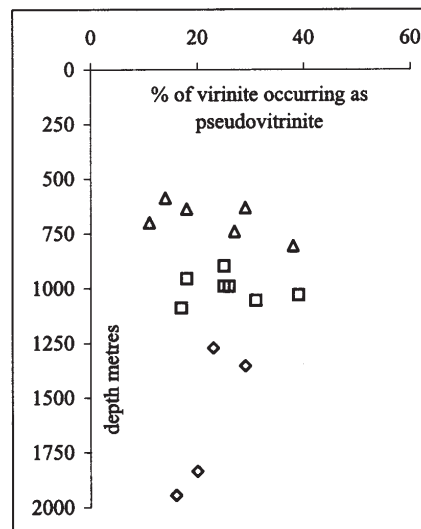


Figure 4. Depth versus percent pseudovitrinite in collotelinite Gates Formation coals.

if slits form by devolatilization it is not a normal process. The process could be unusually rapid coalification or rapid degassing of adsorbed gas. The latter possibility is unlikely because unmineralized slits are not ubiquitous in degassed core samples. The former possibility cannot be proved or disproved based on the data at hand.

Data indicates or at least leaves open the possibility that slits form at depth after the coal has reached ranks at which it is generating thermogenic methane. It is possible that either or both oxidation and desiccation are operating at depths in excess of 1000 metres in some but not all formations, because pseudovitrinite is much less common in coals from the Mist Mountain Formation in the southeast British Columbia than in coals in the Gates and Gething formations.

Coal is sometimes washed using froth floatation, which uses the difference in hydrophobicity of rock and coal to float the coal. Rock and oxidized coal sink (have high wettability low hydrophobicity) and an oxidized coal floats. In fact vitrinite macerals float better than the inert macerals and studies have shown (Arnold and Aplan, 1988) that pseudovitrinite floats better than vitrinite. This indicates that pseudovitrinite is not an oxidized variety of vitrinite.

Desiccation of coals at depth can only occur if the seam is gas saturated. The ability of gases to retain water varies with the conditions and the gas composition. The solubility of water in CH₄ is generally low and increases as the temperature increases or as the pressure falls. In order to estimate the solubility of water in CH₄ at depth it is necessary to superimpose a geothermal gradient on a solubility plot (McKetta and Wehe, 1958), in this case re plotted using linear scales (Figure 5). It appears that at about 3000 metres one cubic metre of CH₄ can hold about 3 grams of water in solution based on a geothermal gradient of 25°C/Km. The effect of this on coal depends on the volume of gas adjacent to the seam. Figure 6 illustrates the amount of water in solu-

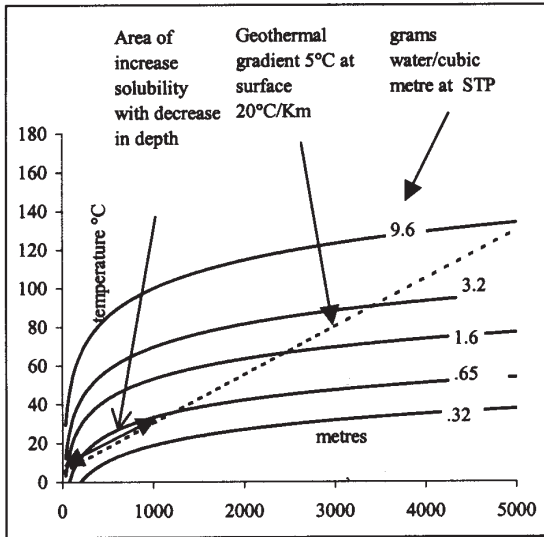


Figure 5. Solubility of water in CH₄ adapted from McKetta and Wehe (1958).

tion in gas pressured into a rock with 5% porosity as hydrostatic pressure increases. The volume of rock was calculated to be the same as the volume of 1 tonne of coal. It therefore appears that at 3000 metres the gas in a porous rock adjacent to the tonne of coal can hold 25 grams of water. In order to have an appreciable drying effect there would have to be at least 10 cubic metres of gas filled porous rock for every cubic metre of coal.

There is also a rule-of-thumb used in the industry that assumes that up to 22 barrels of water can be dissolved in one million cubic feet of gas. This is equivalent to 124 grams per cubic metre, which is more than is indicated by the graphs of McKetta and Wehe (1958). Using this estimate the gas in the 5% porous rock could hold 4.76 kilograms of water at a depth of 3000 metres. It would not take many cubic metres of gas-saturated rock adjacent to a coal seam to hold much of the free water in the seam. The solu-

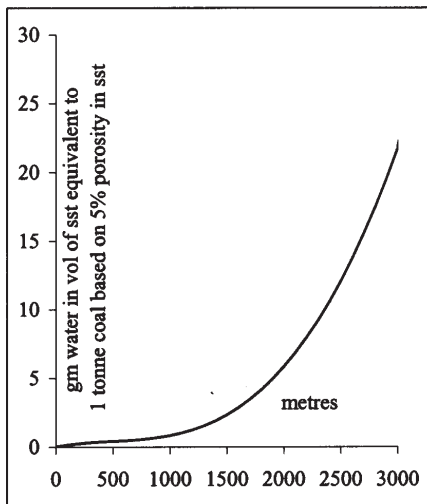


Figure 6. Water in solution in 1 cubic metre of sandstone with 5% porosity.

bility of water in CO₂ is much higher so that if the coal is in contact with a mixture of gases it would be much easier to remove water from the coal.

The ability of gas to hold water in solution does not mean that it has the ability to extract water from the coal because the gas may already be saturated. However when a burial uplift tract constructed assuming a normal geothermal gradient is superimposed on the diagram of McKetta and Wehe (1958), it forms a curve (Figure 7). Traversing along the curve provides values of water solubility in gas during progressive burial or uplift. It is apparent that above a depth of about 1000 metres the ability of gas to hold water starts to increase and below this depth the ability of gas to hold water decreases. The equilibrium moisture content of coal decreases with increasing temperature and therefore depth (Bustin and Clarkson, 1998)(Figure 8) so that at depths of less than 1000 metres, as coal is uplifted, equilibrium moisture value is increasing but the ability of gas to hold water is also increasing. The coal is attempting to regain moisture to reach equilibrium moisture content at the

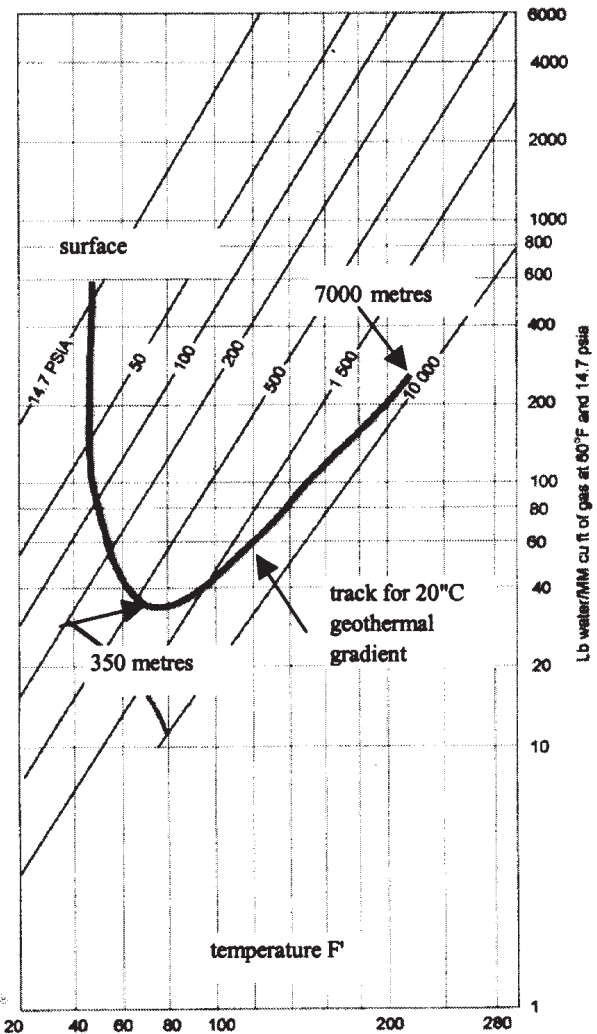


Figure 7. Burial tract superimposed on the solubility curves of McKetta and Wehe (1958)

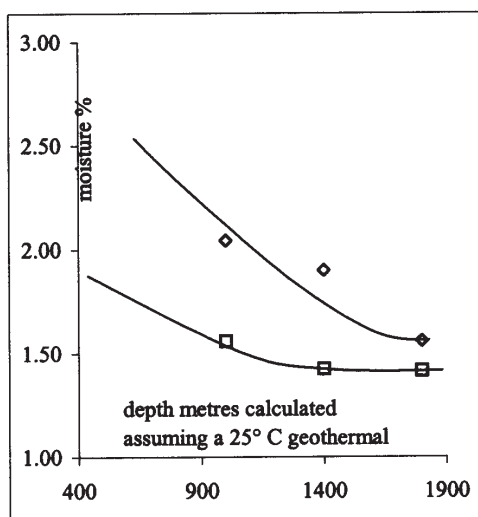


Figure 8. Variation of Equilibrium moisture with depth. Data from Bustin and Clarkson (1998):

same time that the gas has the ability to hold more water in solution. This environment may produce mild desiccation of the coal. Conditions for desiccation are improved if the geothermal gradient is low.

There are provisional arguments that indicate the possibility of drying coal below equilibrium moisture at depths of 1000 metres. Slits were seen in Gates coals at depth greater than this depth so it is certainly not proved that desiccation at depth is the only process forming pseudovitrinite. However there are enough data to indicate the possibility.

RELATIONSHIP OF PSEUDOVITRINITE TO ADSORPTION CAPACITY AND GAS CONTENT

Before discussing the implications of pseudovitrinite on adsorption it is interesting to note that all isotherm anal-

yses are performed on coals that have adsorbed oxygen on exposure to the atmosphere and they may therefore not be the same as isotherms that would be obtained on coal at depth in an oxygen deficient environment.

The presence of pseudovitrinite may indicate desiccation that precedes oxidation and or early stages of oxidation. There are a number of papers that document the effects of oxidation and desiccation on the adsorption characteristics of coal. Ettinger *et al.* (1967) discusses the effects of mild oxidation on the adsorption and desorption of carbon dioxide and methane. Initial oxidation produces a surface effect that makes it harder for CH_4 to desorb and easier for coal to adsorb CO_2 . This might explain the initial release of CO_2 from desorption canisters. The effect may not influence the amount of CH_4 previously adsorbed on the coal but could slow down desorption making it the rate controlling process for production. Clarkson (1992) found that laboratory oxidation slightly decreased adsorption capacity. Vessay (1999) quoting a number of authors states that surface oxidation causes a slight decrease in adsorptive capacity but that laboratory oxidation resulted in an increase in adsorptive capacity. It is possible that high temperature oxidation that occurs in the absence of water is not the same as low temperature oxidation occurring at surface under moist conditions (Huggins *et al.*, 1983)

The effect of moisture on adsorption was investigated by Joubert *et al.* (1973) who measured CH_4 adsorption isotherms on a number of coals of different rank and at different moistures ranging from 0% to equilibrium moisture. They considered equilibrium moisture to be independent of pressure though they did not consider the effects of temperature. Bustin and Clarkson (1998) indicate that equilibrium moisture of Permian Australian coals decreases as temperature increases (Figure 8). The best estimate of the effect of natural desiccation of coal is probably obtained by calculating the difference between the equilibrium moisture isotherm and one measured at or about air-dried moisture. This is done for three coals studied by Joubert *et al.* (1973) (Figure 9, 10). At low to intermediate rank a small amount of

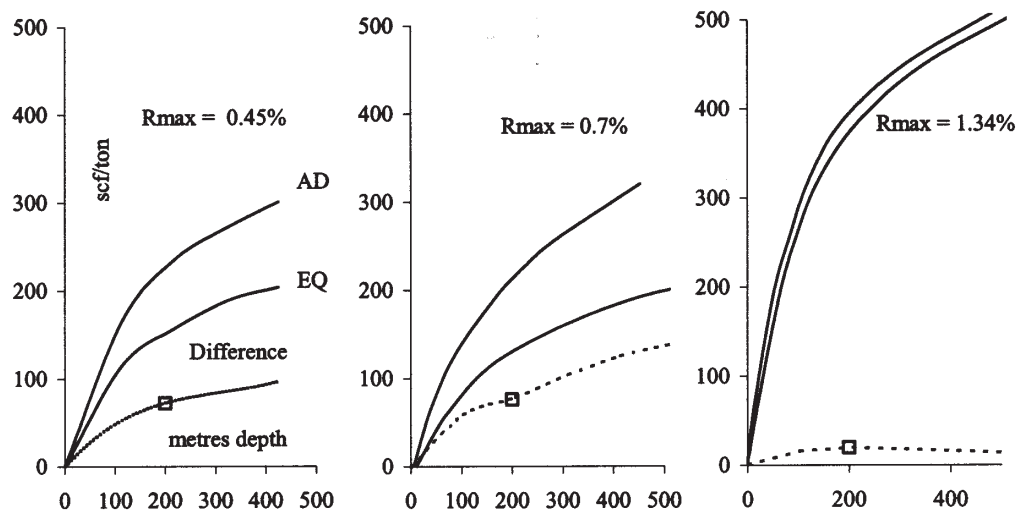


Figure 9. Effect of desiccation on adsorption ability of coals of different rank data from Joubert *et al.* (1973).

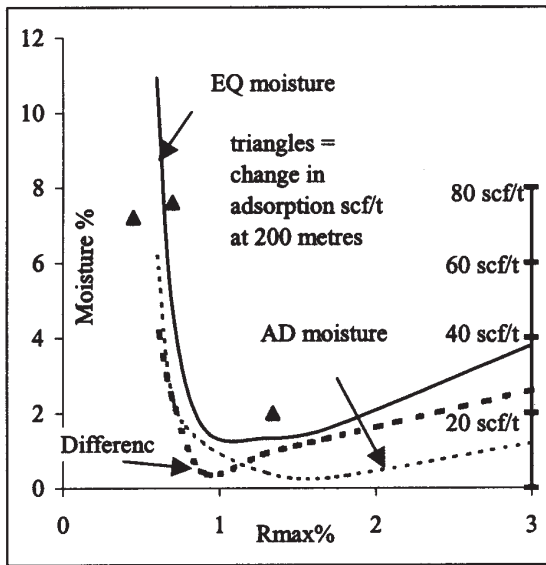


Figure 10. Relationship between the difference equilibrium moisture and air-dried moisture *versus* rank and for the difference in adsorption ability for equilibrium moisture and air dried samples *versus* rank.

desiccation can increase adsorption capacity by up to 100 scf/t at depths less than 500 metres. At medium-volatile ranks the increase is much less. However the difference between equilibrium and air-dried moisture increases again at higher ranks above 1.5 Rmax, which would impact some of the Gething coals in northeast British Columbia.

At constant rank, equilibrium moisture is strongly dependent on vitrinite content. There is insufficient data to attempt to unravel the relationship between vitrinite and pseudovitrinite contents on an ash-free basis and equilibrium moisture available for this study but this is an obvious direction to pursue in the future.

No data have been located that address directly the effect of pseudovitrinite on adsorption. Re interpreting existing data indicates that the presence of pseudovitrinite may improve adsorption of coals, based on using Gething Formation adsorption data (Ryan and Lane, 2002) and re doing the petrography of the samples to estimate the amount of pseudovitrinite present. A tertiary plot of vitrinite pseudovitrinite and inertinite on a mineral matter free basis (Figure 11) indicates a possible increase in Langmuir volumes with increasing pseudovitrinite content. The rank of these coals is high (1.6% Rmax) so a difference in adsorption capacity is possible based on the results from Figures 9 and 10. Obviously these samples were not collected from a gas saturated environment, but when coal is dried below equilibrium moisture the micro porous structure is irreversibly altered and rewetting the coal will not undo the damage and presumably the increase in adsorptive capacity remains.

Unfortunately the paired adsorption isotherms from Comox and Tulameen coals were obtained from coal samples with very similar contents of pseudovitrinite.

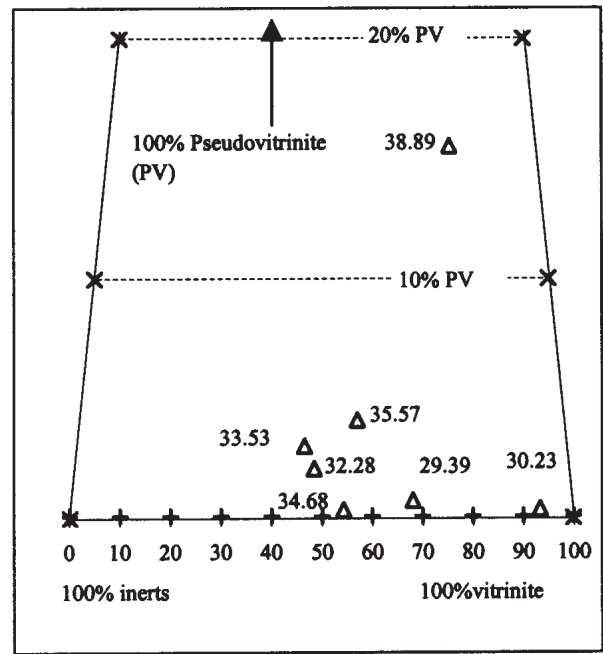


Figure 11. Tertiary plot of vitrinite, pseudovitrinite and inert macerals, indicating adsorption capacity on a dry-ash-free basis.

Desorption data for Comox coals reported previously (Ryan, 2002) exists for samples with varying amounts of pseudovitrinite. It is possible to plot gas contents on a daF basis *versus* percent pseudovitrinite for samples collected from adjacent depths (Figure 12). The samples all have high vitrinite contents on a mineral-matter-free basis, so it

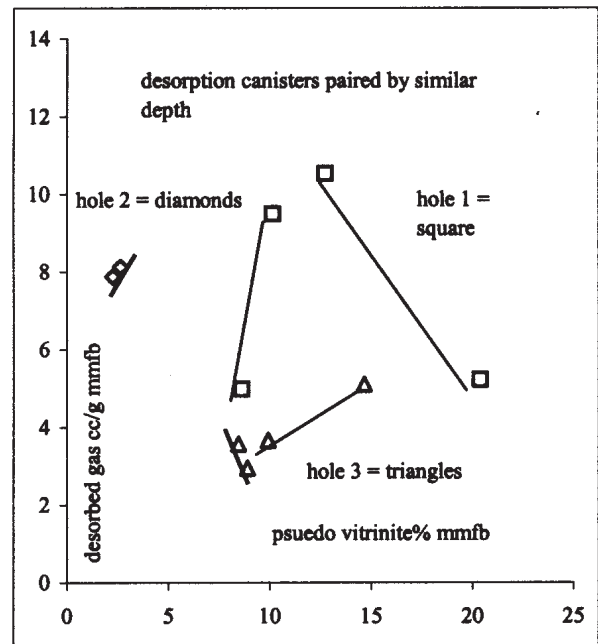


Figure 12. Plot of desorbed gas contents *versus* percent pseudovitrinite content for samples collected from similar depths, Comox Formation.

is not necessary to consider other petrographic variables. Five pairs of data exist, based on grouping data by depth, of these pairs two have very little change in gas contents and three have significant changes in gas content and pseudovitrinite content. Of these, two show a marked increase in desorbed gas content with increase in pseudovitrinite content and one a decrease. The data is obviously inconclusive but the process demonstrates a way that could prove or disprove a connection between desorbed gas content and amount of pseudovitrinite.

DISCUSSION

In terms of the importance of pseudovitrinite to CBM exploration there are a number of considerations.

Firstly, does pseudovitrinite occur at a depth where one would hope to extract CBM? If it develops during uplift at shallower depths, then its effect on gas adsorption is irrelevant in terms of CBM production. However if it affects adsorption it will cause samples to produce adsorption curves that are not characteristic of their adsorption behaviour at depth. Pseudovitrinite occurs at depths of over 200 meters in Comox Formation coals. In northeast British Columbia pseudovitrinite occurs in the Gething and Gates formations. In the Gething Formation it is documented in a shallow drill hole and in a test pit; in Gates coals it is found in the active mines, some distance below the original topography (Diessel, 1998) and in drill holes to depths of about 2000 metres. Pseudovitrinite is definitely not restricted to shallow coals.

Secondly if pseudovitrinite occurs at depth and affects adsorption then the amount of pseudovitrinite should be considered when interpreting isotherm data. No data are presented that proves a connection between the amount of pseudovitrinite in coal and its adsorption ability or desorbed gas content. However the Gething coal data leave open the possibility. Certainly desiccation increases adsorption ability though the effect is probably a minimum for medium rank coals. There is no direct evidence that pseudovitrinite is in part caused by desiccation but there is circumstantial evidence. Pseudovitrinite does not form exclusively as coal is uplifted close to the present surface because it is present at depth. It must form at depth in which case oxidation seems unlikely.

Thirdly does the environment in which pseudovitrinite forms have implications for the gas potential of the stratigraphic package in which the coal occurs? If it is accepted that pseudovitrinite forms at depth by partial desiccation of coal, then this obviously implies that at some time the coal was in a gas-saturated environment. Coal cannot be under saturated in a gas-saturated environment based on existing pressure and temperature. Also within the volume that is gas saturated there is effectively no pressure gradient and the only thing varying the adsorption capacity is temperature. The presence of pseudovitrinite can be detected from a small sample of coal chips so information about its presence is easy to obtain from drill hole chips. Compiling this type of information may be useful in locating natural gas fields.

If the adsorptive capacity of coal increases with pseudovitrinite content, then the potential desorbed gas content and micro permeability of the coal may also improve. However the coal must be able to scavenge gas. This is obviously possible if pseudovitrinite forms in a gas-saturated environment. The degree of saturation of coal may depend on the depth that the pseudovitrinite formed and the amount of subsequent uplift. If it formed at depths much greater than 2000 metres and the coal was subsequently uplifted, then adsorptive ability may increase markedly leaving the coal under saturated. If on the other hand the pseudovitrinite forms at a depth close to the present depth and in a depth production window for CBM, then the coal should be close to saturated with excellent adsorptive capacity. The coal section may be gas saturated at depth and experience partial desiccation. Later the stratigraphy may become water saturated but the coal will not be able to regain its original equilibrium moisture level nor will the pseudovitrinite revert to normal vitrinite and the coal will retain an improved adsorption capacity.

If development of pseudovitrinite in coals is accompanied by an improvement in adsorption capacity then this has interesting implications in a dynamic situation of a producing well. In the situation where desiccation or oxidation has not occurred prior to production, but commences during production from a well, any increase in adsorption capacity will hinder production. This could occur because, over a period of months to years, pumping water out of the coal seam may introduce oxygen or cause some drying of the coal seams.

The limited amount of data discussed in this paper does not prove, but does leave open the possibility that the presence of pseudovitrinite may be a useful indicator to consider when analyzing adsorption and desorption data. It may also be of interest to those studying natural gas reservoirs because of the assumed association with gas-saturated stratigraphy.

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