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Application of Attenuated Total Reflectance Micro-Fourier Transform Infrared (ATR-FTIR) Spectroscopy to the Study of Coal Macerals: Examples from the Bowen Basin, Australia

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Abstract

Attenuated total reflectance micro-Fourier transform infrared (ATR-FTIR) spectrometry has been successfully used to characterise coal macerals, in particular telocollinite, and to investigate changes in the aromatic and aliphatic functional groups in the telocollinite, over a wide rank range (R\textsubscript{vmax} from 0.39 to 3.52\%) in coals from the Bowen Basin, Queensland, Australia. The results show that ATR-FTIR is very sensitive to the increasing aromaticity (the fraction of carbon atoms involved in aromatic units) of the telocollinites, and thus is a very useful tool to study the evolution of aromatic and aliphatic functional groups with maturation of telocollinite, and also to differentiate and characterise the various macerals in coal samples. In comparison with other micro-FTIR techniques, ATR-FTIR has many advantages: (1) no difficult and time-consuming procedures are required to obtain “pure” maceral separations, or for preparation of thin coal slices; (2) the ATR-FTIR spectra have better signal-to-noise ratio and increased sensitivity; (3) the ATR-FTIR spectra are similar to absorption spectra, but without significant spectral distortion.

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1. Introduction

Fourier transform infrared (FTIR) spectroscopy has been used for some decades to investigate a range of different materials, generally non-destructively, in chemistry, materials science and other scientific fields. This is because the infrared spectrum can provide crucial information on the molecular structure of organic compounds, in particular the functionalities such as aromatic and aliphatic groups, carbonyl, hydroxyl etc. While some early work was performed using dispersive instrumentation (Brown, 1955; Durie et al., 1966), application of infrared spectroscopy to coal characterisation did not really take off until the early 1980s with the advent of interferometric spectrometers (Brown et al., 1981; Painter et al., 1981; Fuller et al., 1982; Solomon and Carangelo, 1982; Rhoads et al., 1983). Due to the extraordinarily complex chemical structure of coal, FTIR applications in coal studies have also encountered numerous challenges, and some controversies have arisen surrounding the correct assignments of particular bands to various functional groups (Painter et al., 1985; Solomon and Carangelo, 1988; Fredericks, 1989). Nevertheless, with the recent development of accessories, such as the infrared microscope, as well as spectral handling and processing techniques, FTIR has received renewed interest for the study of functional groups in coal macerals (Sobkowiak and Painter, 1992; Mastalerz and Bustin, 1993, 1995, 1996; Ibarra et al., 1996; Mastalerz and Schimmelmann, 2002; among others)
One recent development in FTIR techniques is the incorporation of an attenuated total reflectance (ATR) crystal (~100 µm in diameter) into the objective of an FTIR microscope (Reffner et al., 1991; Lewis and Sommer, 1999). The ATR crystal is brought into contact with the sample while the FTIR spectrum is collected, allowing spectra with improved signal-to-noise ratio to be obtained, compared to other micro-FTIR techniques based on conventional transmission and specular reflection.

Transmission microspectroscopy of coal requires the preparation of thin slices (less than about 30 µm), which is difficult and time-consuming. An advantage of the ATR method applied to coal is that standard polished block samples can be used without further sample preparation (Thomasson et al., 2000).

In this respect micro-ATR is similar to conventional reflectance micro-FTIR measurement (Mastalerz and Bustin, 1995). However, the spectrum obtained by reflectance micro-FTIR from a polished block sample is a specular reflectance spectrum and is considerably distorted in comparison to an absorption spectrum. Previous workers have attempted, with varying degrees of success, to correct this distortion by applying a mathematical procedure known as the Kramers-Kröning transformation (Mastalerz and Bustin, 1996). Micro-ATR does not require this type of correction, although it should be noted that band intensities differ from those in a true absorbance spectrum because of the relationship between penetration depth and wavelength that exists in ATR spectroscopy (Harrick, 1967). This effect in ATR-FTIR spectra can be easily corrected with software based on multiplying the spectrum by a simple linear function to produce a calculated absorption spectrum, because the intensity of the bands is directly related to the wavelength of the IR light (Thomasson et al., 2000).
In a previous study of the application of micro-ATR to coal samples (Thomasson et al., 2000), it was noted that the major disadvantage of micro-ATR was the lower spatial resolution of about 100 μm, compared with about 20 μm achievable by transmission or reflectance methods. However, a recent report (Chan and Kazarian, 2003) has stated that the spatial resolution in micro-ATR is better than previously thought, because the true spatial resolution is calculated by dividing the aperture by the refractive index of the crystal material. As the refractive index of a silicon crystal is 3.4 and that of a germanium crystal is 4, the true spatial resolution (25 to 30 μm) is similar to that obtained by conventional micro-FTIR methods.

In this paper we report the application of ATR-FTIR to the study of macerals directly in polished coal samples. The objectives of this study are: (1) to investigate the applicability of ATR-FTIR to independent study of different macerals; (2) to examine the advantages and disadvantages of ATR-FTIR over conventional micro-FTIR techniques; (3) to test the capability of using ATR-FTIR to evaluate the maceral structure, in particular the variations of both aromatic and aliphatic functional groups with rank maturation.

2. Samples and Experimental Procedures

A series of Permian coals from the Bowen Basin of Queensland, covering a rank range from sub-bituminous (Rvmax 0.39%) to anthracite (Rvmax 3.52%) were subjected to ATR-FTIR study (Table 1). A torbanite sample from the Sydney Basin, New South Wales, was also examined, but only for comparison with other macerals in the Bowen Basin suite. The locations of the Bowen Basin samples are given in Figure 1.
This work complements a parallel study of the elemental composition of the individual macerals in the same suite of Bowen Basin coals using electron microprobe techniques (Ward et al., 2004, 2005).

A Nicolet Nexus FTIR spectrometer (Fig. 2), equipped with a Nicolet Continum microscope incorporating an attenuated total reflectance (ATR) objective, was used to collect spectra for the samples examined in the study. The microscope has a fully programmable stage, which allows samples to be mapped in the X and Y directions. In this study, the FTIR spectra were obtained in ATR mode from polished sections, following similar procedures to those described by Thomasson et al. (2000). Polished sections of coal blocks or large (> 5 mm) coal fragments were mounted on the microscope stage, and set in a horizontal orientation using plasticine (modelling clay). After visually selecting the desired area for analysis, the silicon ATR crystal was moved into place and the sample stage raised so that contact was made with the polished surface of the coal sample. This was done very gently, controlled by a pressure minoring device to ensure reproducible contact pressures and minimise damage to the sample surface, which can occur if pressure from the crystal on the sample surface is too high.

The principles of spectrum collection using this instrument are described by Thomasson et al. (2000). IR light is directed into the crystal element at such an angle that it is totally reflected at each point of incidence inside the crystal. At each reflection part of the light, called the evanescent wave, passes beyond the crystal interface and interacts with the sample, placed in close contact with the crystal. The light ultimately exits the crystal and is directed to the IR detector, from which a signal
is passed to the data processing system. A liquid-nitrogen-cooled mercury-cadmium-
telluride (MCT) detector was employed, and 512 scans were co-added to give the
final spectrum, after ratioing to the background spectrum. The background spectrum
was obtained by using the ATR element under the same settings but without
contacting the sample surface. The measurement time for each spectrum collection
was around 30 s. The contact area was circular and approximately 100 μm in
diameter. However, the area of the sample actually measured is given by the contact
area divided by the refractive index of the crystal, and hence is about 30 μm in this
case; it can be further reduced to near the diffraction limit by the use of an aperture.

ATR-FTIR spectra were collected in the range of 4000 to 700 cm\(^{-1}\) at a gain of 4 and a
resolution of 8 cm\(^{-1}\), which gave good signal-to-noise ratio as well as a reasonable
compensation for the narrow water and CO\(_2\) bands of the background. Because the
ATR-FTIR spectra were collected from such a small area (~30 μm) and can be pre-
selected from mineral-free areas under a microscope, the technique not only avoids
difficult and time-consuming maceral separation procedures, but also eliminates
possible spectral interference caused by the contribution from other macerals and/or
minerals. A total of 58 ATR-FTIR spectra were collected from the various macerals
(including telocollinites, desmocollinites, sporinites, semifusinites and fusinites) in 9
samples (see Table 1), with excellent sensitivity and reproducibility.

3. Results and Discussion

Among those 58 spectra collected on the different macerals, at least 4 spectra were
collected from telocollinite and a few spectra from other macerals (sporinite,
semifusinite and fusinite) in each sample. Whenever possible, the spectra were only
collected from areas consisting of “pure” macerals, such as telocollinite, desmocollinite, alginite, semifusinite and fusinite, without intimate admixture of any other macerals or minerals in the area under investigation. After careful examination of all 58 of the ATR-FTIR spectra of various macerals collected from the sample suite (Table 1), some distinctive characteristics in the FTIR spectra of the individual macerals in the different samples were noted.

3.1. Comparison between the ATR-FTIR spectra of telocollinites and fusinite

As expected, marked differences were noted in terms of the shape and intensity of the absorbance peaks from the different macerals (telocollinite, fusinite and alginite) in the coal samples. Figures 3 and 4 give examples of typical ATR-FTIR spectra of the different macerals observed in this study. Mastalerz and Bustin (1993) reported a similar observation, but the ATR-FTIR spectra show better sensitivity and signal-to-noise ratio and have no significant spectral distortion. For example, Figure 3 shows a comparison between the spectra of telocollinite and the fusinite in a medium-volatile bituminous coal sample (Rv_max = 1.31%). Both spectra are broadly similar in terms of band position, but the telocollinite has much stronger absorbance at 3000 and 2700 cm⁻¹ region (representing aliphatic CH, CH₂, CH₃ stretching modes) and 1442 cm⁻¹ (representing CH₂ and CH₃ bending modes) than the fusinite of the same coal (Figure 3).

In comparison with the spectrum of telocollinite, the absorbance of fusinite for the same coal has the following characteristics: (1) much stronger absorbance of aromatic CH stretching at 3046 cm⁻¹ relative to the aliphatic CH stretching at the region between 3000 to 2700 cm⁻¹, and (2) much stronger absorbance at both 1020 cm⁻¹ and
870 cm\(^{-1}\) band. The stronger absorbance of the fusinite compared with the telocollinite of the same coal at 878 cm\(^{-1}\) could be attributed to the increasing isolated aromatic H in the fusinite. The other interesting observation is that the shape of absorbance bands in the region 1000 to 1600 cm\(^{-1}\) for the medium volatile bituminous coal \(R_{v_{\text{max}}} = 1.31\%\) is more similar to those of telocollinites with \(R_{v_{\text{max}}}\) of greater than 2.14\% (see Figures 3 and 5) rather than those of telocollinites with \(R_{v_{\text{max}}}\) of lower than 1.31\%. All these observations suggest that the fusinite has a greater proportion of aromatic functionalities than the telocollinite of the same coal.

3.2. ATR-FTIR spectra of alginite
The alginite spectrum (Fig. 4) shows the strongest aliphatic CH stretching bands at 2920 cm\(^{-1}\), but the least (almost no visible) aromatic CH stretching absorption band at 3030 cm\(^{-1}\). This is mainly because: (1) alginite originated from algal bodies, which are distinguished by relatively abundant fatty and proteinaceous substances (such as fatty acids \(n\)-C16 and \(n\)-C18, and alkanes \(n\)-C15 and \(n\)-C17) and thus are particularly resistant and oil-rich (Taylor et al., 1998); (2) the aliphatic groups in alginite are mainly saturated alkyl chains (Kister et al., 1990), with the longest chains but fewest branches among the maceral groups. This is supported by the fact that the two bands at 2920 and 2850 cm\(^{-1}\) in the alginite spectrum (Fig. 4), assigned to antisymmetric and symmetric methylene CH stretching respectively, are very intense, and bands due to methyl CH stretching (expected around 2960 and 2860 cm\(^{-1}\)) are very weak, indicating few branches. Guo and Bustin (1998) also report that micro-FTIR spectra indicate that alginite has the strongest aliphatic and least aromatic absorptions, followed by bituminite, resinite, cutinite and sporinite. Micro-FTIR spectrometry therefore confirms that liptinite macerals are characterised by having greater number
of long chain aliphatic groups, fewer aromatic groups and a broader range of oxygen-containing groups than other coal macerals (Guo and Bustin, 1998).

3.3. Semi-quantitative evaluation of aromatic and aliphatic hydrogen over increasing coal rank advance

A progressive change in the aromatic and aliphatic groups of vitrinite is evident in the Bowen Basin coals with rank advance (Figure 5). Although the spectra obtained from vitrinites with different reflectance values ($R_{v,max}$ from 0.39 to 3.52%) are broadly similar in shape (positions of absorption peaks), the relative intensities of the absorbance peaks vary quite significantly (Fig. 5). For example, all of the coals have CH stretching bands at 3100 to 2700 cm$^{-1}$. Generally speaking, aromatic groups have stretching bands in the region 3100 to 3000 cm$^{-1}$, whilst the aliphatic stretching bands fall between 3000 and 2700 cm$^{-1}$.

The FTIR spectra of the vitrinite samples examined in this study are given in Figure 5. Figure 6 provides an expanded plot of the region 3400 to 2600 cm$^{-1}$ of the spectra presented in Fig. 5. Figure 5 and, in particular, Figure 6 clearly demonstrate that the absorbance of the aromatic stretching band at $\sim$3030 cm$^{-1}$ increases continuously from sub-bituminous coal to anthracite, expressed by increases in vitrinite reflectance ($R_{v,max}$) from 0.39 to 3.52%, at the expense of the absorbance of the aliphatic stretching bands near 2920 cm$^{-1}$. The absorbance of aliphatic bands at 2800-3000 cm$^{-1}$ is very low in the telocollinite of sub-bituminous coal, becomes strongest in bituminous coal at round $R_{v,max} = 0.9-1.31\%$, and is then slightly weakened from low-volatile bituminous coal to anthracite.
These observations are broadly similar to those of Mastalerz and Bustin (1993), who reported very weak to no aromatic absorbance in the 3030 cm\(^{-1}\) region, and nonexistent aliphatic absorbance in the 2800-3000 cm\(^{-1}\) region, for Carboniferous anthracite from Pennsylvania. The slightly different observations in the present study are probably a result of the increased sensitivity and higher signal-to-noise ratio of ATR-FTIR over the conventional reflectance micro-FTIR technique.

Semi-quantitative studies from the ATR-FTIR spectra show that the ratio of the aromatic CH peak intensities to the peak intensities representing the aliphatic CH functional groups (Figures 7 and 8) is clearly a rank-dependant parameter. Based on the spectra of all the telocollinites over the rank range studied, the aromatic CH group stretching bands occur at between 3100 and 3000 cm\(^{-1}\), whereas the aliphatic CH groups have stretching bands between 3000 and 2700 cm\(^{-1}\). In addition, aromatic rings have stretching bands near 1600 cm\(^{-1}\), and aromatic CH out-of-plane bends absorb at between 900 and 700 cm\(^{-1}\). However, the 1600 cm\(^{-1}\) region is not suitable for the quantitative study of aromatic groups, because (1) the highly conjugated hydrogen-bonded C=O functional groups also absorb near 1600 cm\(^{-1}\) (Painter et al., 1983; 1985), and (2) low rank coals usually contain significant amounts of COO\(^{-}\) groups, which also absorb near 1580 cm\(^{-1}\) and hence contribute to the intensity of the 1600 cm\(^{-1}\) band.

The aromatic C-H out-of-plane region (900 to 700 cm\(^{-1}\)) is not suitable for quantitative study of the aromatic functional groups, due to the fact that both aliphatics and minerals usually also show strong absorbance in this region and may produce undesirable interference. Only two regions, 3100 to 3000 cm\(^{-1}\) and 3000 to
2800 cm\(^{-1}\), are therefore representative of the “pure” aromatic and aliphatic functional groups respectively, without coupling to and mixing with other functional group vibrations. Consequently, these two regions (3100 to 3000 cm\(^{-1}\) and 3000 to 2800 cm\(^{-1}\)) were chosen to evaluate the aromatic and aliphatic functional groups in the present study.

The integrated areas in these two regions have been used to calculate the peak intensity ratio (\(H_{ar}/H_{alli}\)) of the aromatic to aliphatic functional groups for the individual vitrinites in the coal samples, following a similar procedure to that used by Fujii et al. (1970). Figure 7 clearly shows a pronounced and approximately linear relationship between the ratio of aromatic to aliphatic functional groups and their respective vitrinite reflectance (\(R_{v,max}\%\)) values for the Bowen Basin coals studied. The ratio of aromatic to aliphatic groups increases progressively with increasing rank, as well as the increasing aromaticity of the vitrinite. Figure 8 clearly demonstrates that the ratio of aromatic to aliphatic groups also increases with vitrinite carbon content, but in a different way to the increase in vitrinite reflectance (Figure 7). The ratio shows only a slight increase with rank from sub-bituminous (vitrinite with around 66% carbon) to bituminous coal, until the vitrinite in the coal reaches about 90% carbon (\(R_{v,max} \sim 2.0\%\)) (Figures 7 and 8). After this point, the ratio of aromatic to aliphatic C-H groups increases dramatically.

In a study based on a set of Japanese coals, Fujii et al. (1970) reported a similar variation, with the ratio of aromatic to aliphatic groups increasing slightly with carbon content until the carbon content reached approximately 88%, after which the ratio of aromatic to aliphatic groups increased dramatically with carbon content. This finding
is also consistent with the results of other studies (Painter et al., 1985; Pradier et al., 1992; Mastalerz and Bustin, 1993, 1996; Thomasson et al., 2000; among others). Such an observation clearly demonstrates that, with increasing rank in the Bowen Basin, from subbituminous coal to anthracite, the coalification processes involve not only increases in the absolute proportion of carbon in the vitrinite (cf. Ward et al., 2005), but also increasing aromaticity (indicated here by the $H_{ar}/H_{al}$ ratio) in the chemical structure of those vitrinite macerals.

Although both absolute carbon content and aromaticity increase with rank advance, the approximately linear relationship between the ratio of aromatic to aliphatic functionalities and vitrinite reflectance indicates that it is the aromaticity, rather than absolute carbon content in the vitrinite, that has the main impact on the vitrinite reflectance. The continuous and progressive increase in the ratio of aromatic to aliphatic functional groups from sub-bituminous coal to anthracite also confirms that the Bowen Basin coals were indeed formed mainly through normal coalification processes largely influenced by burial depth. If the coals studied had other major influences (such as igneous intrusion or marine influence), these would have been reflected in either the aromaticity or the vitrinite reflectance of the coal sequence concerned (Federicks et al., 1985).

4. Summary

Based on the results of this study, the ATR-FTIR technique has been proven to be a simple, fast and effective method to study the FTIR spectra of coal macerals in situ and independently.
Micro-FTIR has the advantage that it avoids the difficult and time-consuming procedures for “pure” maceral separation. In comparison with other micro-FTIR techniques, ATR-FTIR has many advantages: (1) unlike the transmission micro-FTIR technique, ATR-FTIR does not require the preparation of thin sections (<30 µm); (2) ATR-FTIR collects spectra from polished block samples similar to conventional specular reflectance micro-FTIR spectroscopy, but with better signal-to-noise ratio and sensitivity; (3) there is no significant spectral distortion from ATR-FTIR spectroscopy, and thus there is no need to apply complex mathematical procedures such as the Kramers-Kröning transformation, which is required by conventional reflectance micro-FTIR spectroscopy; (4) The spatial resolution of ATR-FTIR achieved in this study (25 to 30 µm), which is related to the wavelength of the IR light, the instrument apertures and the reflectance index of the ATR crystal, is very similar to that of standard micro-FTIR (~20 µm). The major disadvantage of ATR-FTIR is that the objective must contact the sample and there is the possibility of damage to the sample’s surface. This problem can be overcome by the use of a sensitive load cell to measure the contact pressure.

ATR-FTIR is also proved to be a sensitive tool to evaluate the variations of aromatic and aliphatic functional groups in telocollinites associated with coal maturation processes, indicating the sensitivity of the aromaticity of vitrinite macerals to rank advance. The absorbance intensity of the aromatic C-H groups shows a clearly increasing trend with coal rank, apparently at the expense of the aliphatic functional groups. The absorbance ratios of aromatic to aliphatic C-H groups increase systematically with rank advance, and hence provide a useful rank-indicating parameter.
Acknowledgements

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\[ y = 0.527x - 0.3179 \]

\[ R^2 = 0.86 \]
Table 1

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