

5. Identifying archaeological jet and jet-like artifacts using FTIR

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5.1 Summary

Recent work has illustrated the problems with identifying the various materials that were used to make black lithic ornaments in the past. The different materials that were worked include jet, cannel coal, oil shale, lignite and solidified bitumens - all of these originate from sedimentary deposits rich in organic material. Fourier Transform Infrared Spectroscopy (FTIR) has shown potential as a technique for identifying the materials; the analysis of geological samples from different material types produces distinctive spectra which can be related to their geological depositional environment, based on their chemical composition.

This paper will discuss the problems encountered when the method is applied to archaeological material; the artifacts are often small and intricately carved, so that they are difficult to sample. Where artifacts have been conserved, the resins used for conservation produce an infrared spectrum which can mask the spectrum from the original material. This problem is lessened when the resin is identified and spectral subtraction methods are used to remove its IR spectrum from that of the jet or jet-like material. Oxidation of jet and coals in the archaeological burial environment can also alter their characteristic infrared spectra, and affect the success of FTIR as a discriminating technique. The results of FTIR analyses from two archaeological assemblages - from York and Verulamium - will be discussed to illustrate these points.

5.2 Abstract

Recent work has illustrated the difficulty of identifying the various materials that were used to make black lithic ornaments in the past. The materials which were worked all originate from sedimentary deposits rich in organic material. Fourier transform infrared spectroscopy has shown potential as a technique for identifying the different materials. The analysis of geological samples from the various sources produces distinctive spectra, which can be related to their geological depositional environment, based on their chemical composition. The IR spectra of the various raw materials are discussed in this paper, together with the problems encountered in analysing archaeological artefacts using FTIR. The results of FTIR analyses of artefacts from Verulamium and York are used to illustrate some of these problems.

5.3 Introduction

Jet is one of a range of different black lithic materials that were used to make jewellery and ornaments in the past. Other materials that were worked include cannel coal, oil shale, lignite, and solidified bitumens - all of which originate from sedimentary deposits that are rich in organic material. When polished, the various materials are difficult to distinguish, and in recent years there has been considerable interest in finding reliable methods to identify and provenance them [1,2,3,4,5,6,7,8]. Fourier transform infrared spectroscopy (FTIR) showed promising results in a recent pilot study [6] - geological samples of the different materials produced distinctive spectra. Our investigation of FTIR as an identification method is part of broader research into reliable techniques for analysing archaeological jet and jet-like materials. The aims were to try and understand the infrared spectra produced by the different materials in terms of their geological depositional environment, and to apply the method to archaeological artefacts.

5.4 Approaches to identifying archaeological jet-like materials

Previous research into identifying these black lithic materials has approached the problem in three different ways. Much of the work has concentrated on studying the inorganic elemental characteristics of jet and the other organic-rich rocks [1,6,7]. X-ray fluorescence, often in conjunction with X-radiography, has been used to discriminate between the different materials. This approach has many advantages, in that it is an inexpensive, quick, accessible and non-destructive method for studying archaeological assemblages. Iron is the main discriminant, since jet is almost pure organic material, whereas cannel coals and oil shales contain some minerals, so tend to have much higher iron levels than jet. However, the mineral contents of the different materials can be variable, so that the technique is often limited to identifying broad categories of material, such as 'jet', 'shale' and 'intermediate' [6].

Petrological and palynological methods have also been applied, to identify the material type microscopically, and to extract the spores from the rock, and find out which geological horizon it belongs to [5,8,9]. In certain specific cases this approach has proved extremely informative, and has provided precise provenances for individual artefacts. The disadvantages with these methods are that they require access to specialist equipment and expertise which is expensive for analysing whole assemblages, as well as needing large samples which are too destructive for many of the small, delicate ornaments that were made from jet-like materials.

Organic chemical methods have been used to characterise the organic components of the different materials. The methods applied include electron spin resonance [6,10] nuclear magnetic resonance [11], pyrolysis-gas chromatography/mass spectrometry [12], and Fourier Transform infrared spectrometry [6,13]. Chemical differences between the organic matter in these materials should prove more reliable for identifying them, since the nature of the organic matter is directly related to the original precursor organisms, and their environment of deposition. Apart from electron spin resonance, which produced disappointing results [6], the initial research has proved promising. Until now, this approach has been limited to pilot studies which analysed geological samples. This is the first time FTIR has been used to identify assemblages of archaeological jet-like artefacts.

5.5 Geology and geochemistry of organic sedimentary deposits

An understanding of the geochemistry of the various sources of black lithic materials is essential for interpreting their infrared spectra. These materials were selected for creating ornaments because they are rich in organic carbon - usually in a waxy or oily form - which makes them easy to carve, and gives them a good polish. Coals and other organic-rich rocks can be studied microscopically, and the different components identified provide information on the original type of organic matter deposited, the environment of its deposition, and the maturity of the sediment. All these factors affect the chemical composition of the resultant material, so that petrological characterisation is an important technique for coal and fuel scientists.

The geological samples used in this study were selected from a reference collection of

over 100 samples, held at the University of Bradford for research on jet and similar materials. Organic petrological methods were used to study the nature of the organic material in the different geological sources, and this information was applied to the interpretation of the infrared spectra.

The microscopic constituents of coals, and other organic-rich rocks, can be distinguished by their shape and colour. The different constituents are known as coal macerals, and they are analogous to minerals in inorganic rocks. Coal macerals can be divided into three groups, each of which share some broad similarities in terms of their chemical composition [14].

Vitrinite originates from plant cell walls, and woody tissues. It tends to have a high oxygen content, and is composed of humins with aromatic nuclei surrounded by aliphatic functional groups.

Inertinite often originates from similar plant material to vitrinite, but has been oxidised at an early stage - either by increased oxidative degradation, or possibly sometimes by fire. It is rich in carbon, in the form of concentrated aromatic structures.

Liptinite which encompasses all the waxy and fatty components of plants - spores, leaf cuticles, resins and algal material. Liptinites have a high hydrogen content and are rich in aliphatic carbon. The rocks selected for carbon often contain concentrations of liptinites, since their waxy nature results in a good polish. Liptinites usually fluoresce when illuminated with ultra violet or blue light, so that fluorescence microscopy has become a standard technique for examining coals and oil shales [15].

The infrared spectra produced by the different jet-like materials should be a function of their component macerals. Coals and oil-shales also contain variable amounts of mineral matter, and some of these minerals also produce characteristic absorption peaks in the infrared spectra. By comparing the results of the petrological work on geological samples, to the infrared spectra from the same samples, we should obtain some information on how to interpret the infrared spectra produced by archaeological artefacts to identify the material from which they are made.

5.6 Experimental

The geological samples were ground in a ball mill to a particle size of 75 μm or less. 0.4 mg of the pulverised sample was mixed with 15 mg of KBr, and compressed to form a disc. The KBr discs were analysed on a Perkin Elmer 1720-X Infrared Fourier Transform Spectrometer, using Perkin Elmer IRDM software. Samples were scanned 20 times over wavenumbers 4000 cm^{-1} to 400 cm^{-1} , at a resolution of 4 cm^{-1} .

The constraints on sampling the archaeological artefacts meant that the samples taken were too small to pulverise in a ball mill, before mixing with the KBr. This is discussed below, section 5.8.1

5.7 Characterisation of geological samples

Infrared spectroscopy has a long history in the study of the chemical composition of coals and other organic-rich rocks [16,17]. FTIR spectroscopy has been widely applied to study the chemical changes that occur with increasing rank, and the variations in aromatic:aliphatic ratios and the functional groups present between different coal macerals [18,19,20,21,22,23]. The assignment of absorption bands to particular structures given here are based on this work, and are summarised in table 1.

5.7.1 Whitby jet

Jet is formed from discrete pieces of wood which have been washed into the sea, and have settled in the sea bed. As the sediment accumulated, marine planktonic material deteriorated to produce fluid hydrocarbons, which impregnated the degraded wood structure. Whitby, in North Yorkshire, is the most well-known, and certainly the most abundant source of British jet. Under the microscope, the woody structure of this jet is still visible, in a highly compressed form, so the jet appears to be almost pure vitrinite. However, under blue light excitation, the whole jet structure fluoresces, showing the presence of this oily material.

The infrared spectrum of Whitby jet shows a combination of aliphatic and aromatic structures (figure 1). The aromatic groups are indicated by the aromatic C-H stretch at 3030 cm^{-1} , a shoulder at 1500 cm^{-1} which is the aromatic C=C stretch, and the C-H deformations between 900 and 700 cm^{-1} , which are due to the different substitutions on the aromatic ring. The band at 1600 cm^{-1} is very characteristic of coals and is usually thought to an aromatic ring stretch, possibly enhanced by the presence of phenolic groups [23].

Aliphatic C-H stretches occur as strong bands just below 3000 cm^{-1} with the aliphatic C-H deformation at about 1430 cm^{-1} . Other absorptions that can be seen are the O-H stretch between 3700 and 3100 cm^{-1} , and the complex bands arising from the C-O stretch between about 1300 and 1000 cm^{-1} .

5.7.2 Lignite

Lignite is an immature coal which has passed through the peat stage in the coal series, but has not yet developed into bituminous coal. It has a high water and volatile content, so that the lower rank lignites tend to dry out and crack when exposed to the atmosphere. It is the black lignites, which are closer to the sub-bituminous coals, that are thought to have been worked in the past. Its microscopic structure is similar to jet, although the wood tissues appear less compressed.

The infrared spectrum of lignite is similar to the Whitby jet spectrum, but lignite shows a broader and more pronounced O-H stretching band between 3700 and 3100 cm^{-1} , as well as a band at 1700 cm^{-1} from C=O bonds in carbonyl and carboxylic groups (figure 2). These bands are evidence of the lower rank of the lignite; as coals mature, the O-H and C=O groups are eliminated [24].

These results were initially encouraging; it is difficult to distinguish between jet and

lignite based on their elemental characteristics [7], so if the 1700 cm^{-1} absorption in the FTIR spectra of lignites could be used differentiate them from jet, then the technique would be an improvement on identification methods based on inorganic elemental analysis.

5.7.3 Cannel coal

Cannel coals have a very different micro-structure to jet and lignite. They are formed from finely comminuted organic debris, which settles in stagnant pools on the edge of coal swamps [25]. In these conditions the liptinites are preserved, as they are more resistant to decay. The remains of spores, leaf cuticles and algal bodies can be clearly observed with the petrological microscope, and they are usually embedded in a groundmass of micrinite - a granular inertinite maceral which is described as polymerised resin [8].

The FTIR spectrum from cannel coal (figure 3) shows a less aromatic character than the jet - presumably because it lacks the concentrated wood-derived structures that are found in jet and lignite. The 1500 cm^{-1} band is absent, the aromatic C-H deformations between 700 and 900 cm^{-1} are less well defined, and mineral absorptions from alumino-silicates are sometimes present at 1030 cm^{-1} below 600 cm^{-1} .

5.7.4 Oil shale

The term 'oil shale' can include any sedimentary rock with over 10% organic carbon, but those selected for carving and making ornaments would have been extremely rich in organic material, otherwise they would have disintegrated when they were worked. In Britain, there is one particular organic-rich seam that outcrops in the cliffs at Kimmeridge in Dorset - known as the Kimmeridge shale, or Blackstone. This seam was worked extensively in the past, and concentrations of the working debris are found on Iron Age and Roman sites in the area [26,27].

The Kimmeridge oil shale was formed in anaerobic sediments at the bottom of a shallow marine environment. The organic material is probably derived from marine planktonic organisms which are so degraded that they cannot be distinguished microscopically [5,28]. The Kimmeridge shale appears under the microscope as a groundmass of intermixed clay mineral and organic material together with grains of quartz, calcite and other minerals. The groundmass shows strong fluorescence when illuminated with blue or ultra-violet light, and this fluorescing organic matrix is termed bituminite, and is classified as a liptinite maceral [15].

The infrared spectrum from Kimmeridge shale shows absorptions from aliphatic structures, and sharp absorptions in the free O-H stretching region (figure 4). It also has very strong mineral absorptions at 1420 cm^{-1} from carbonate, and at 1030 cm^{-1} and below 600 cm^{-1} from silicates and alumino-silicates.

The oil shale at Kimmeridge has also been the source of occasional seams of jet. Although the jet does not occur in anything approaching the quantities of the jet at Whitby, it is likely that any jet found at Kimmeridge whilst the surrounding shale was

worked would also have been used for making ornaments.

5.7.5 Kimmeridge jet

Kimmeridge jet has a similar microscopic structure to the Whitby jet - although it does not show the same fluorescence intensity when illuminated with ultra-violet or blue light. The infrared spectrum of the Kimmeridge jet is similar to the spectrum from Whitby jet, except for a sharp absorption at about 1500 cm^{-1} (figure 5). This absorption is assigned to an aromatic C=C stretch, and is present as a characteristic shoulder in the Whitby jet, but it is rarely seen on other coals. A comparison of the IR spectra of Whitby jet, cannel coal, and Kimmeridge jet (figure 6) illustrates the variation in the 1500 cm^{-1} absorption.

FTIR studies comparing jet and other high volatile vitrinites from Spain has demonstrated that the absorption band at 1500 cm^{-1} is an unusual feature of the infrared spectra of these types of coal [19,29]. A sharp absorption at 1500 cm^{-1} was also observed in the spectrum of jet from Utah in the USA.13 Iglesias et al [19] explain this absorption in terms of a different type of substitution on the aromatic ring structures in hydrogen-rich coals such as jet. The intensity and the exact position of the aromatic C=C stretch is dependant on the substitution patterns and the degree of condensation of the aromatic ring. The aromatic structures in these hydrogen-rich coals are thought to have different substitution patterns to other coals, which produces the sharper absorption at 1500 cm^{-1} .

The Whitby jet originates from more mature sediments than the Kimmeridgian sediments, so that the wood structure of Whitby jet has been impregnated by fluid hydrocarbons from the parent shales. A detailed comparison of the organic geochemistry of these two sources of British jet has shown the different chemical nature of the mature Whitby jet using py-GCMS [12]. The variation in the absorption intensity of the 1500 cm^{-1} band presumably reflects this difference in maturity; the immature hydrogen-rich vitrinite analysed by Suárez-Ruiz et al [29] likewise produced a more intense, sharper absorption in the 1500 cm^{-1} region (similar to the spectrum from Kimmeridge jet) than the more mature Spanish jet.

This work on FTIR analyses of geological samples was promising. As in the earlier pilot study by Hunter et al [6], the different materials produced spectra which were distinct from each other, and the differences noted in the IR spectra could be explained by the various organic precursors and depositional environments that were inferred from the microscopic examination. The next stage was to find out whether this information could be usefully applied to identifying the archaeological artefacts from their infrared spectra.

ABSORPTION BANDS IN THE IR SPECTRA OF COALS

Wavenumber (cm ⁻¹)	Assignment
3300	hydrogen-bonded OH
3030	aromatic C-H stretching
3000-2700	aliphatic C-H stretching
1700, 1650	C=O stretch
1600	aromatic C=C
	- poss. enhanced by phenolic groups
1500	aromatic C=C
1450	CH ₂ and CH ₃ bending
1420	carbonates
1375	CH ₃ groups
1030	silicates
900-700	aromatic C-H out-of-plane bending
400-600	silicates

Table 1: band assignments

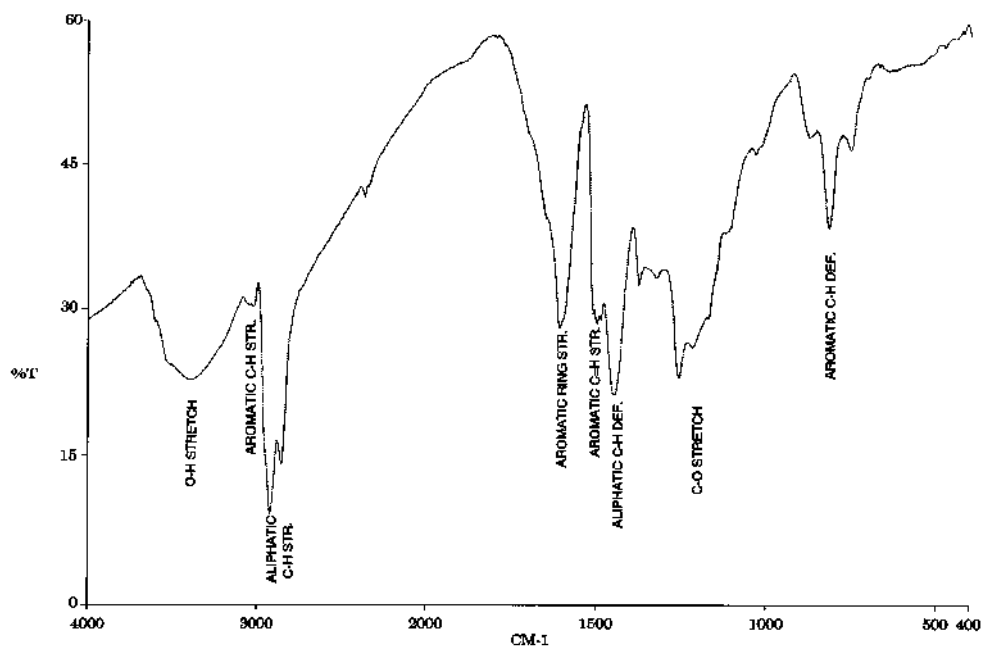


Figure 1: FTIR spectrum of Whitby jet

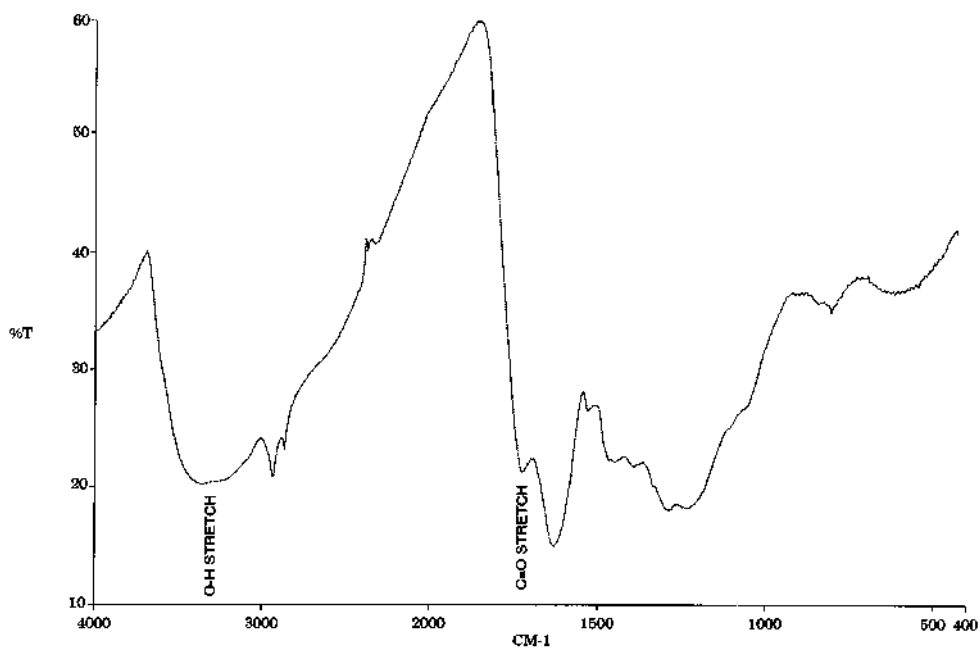


Figure 2: FTIR spectrum of lignite

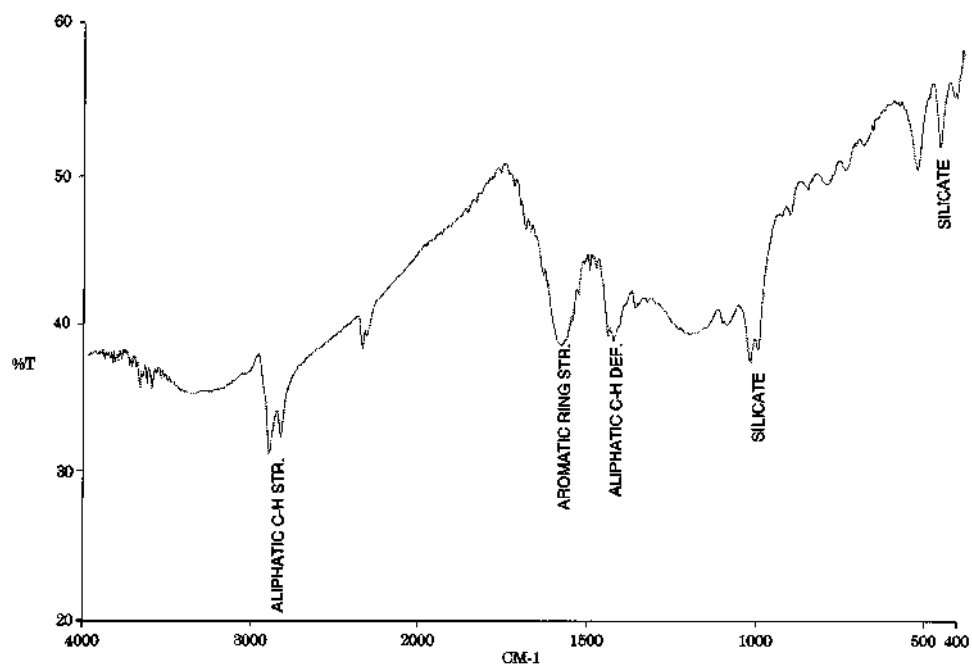


Figure 3: FTIR spectrum of cannel coal

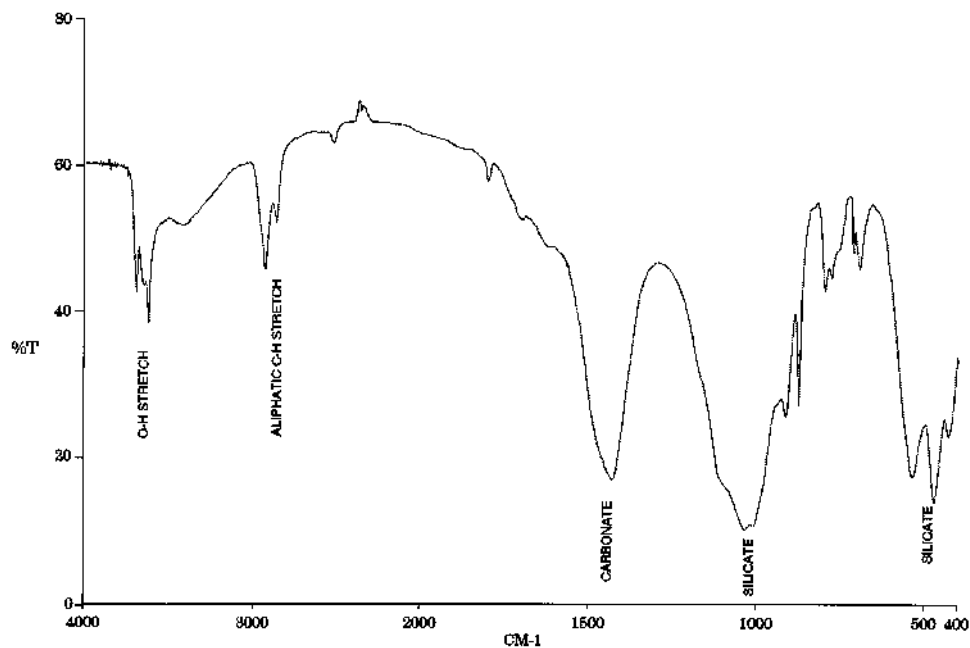


Figure 4: FTIR spectrum of Kimmeridge shale

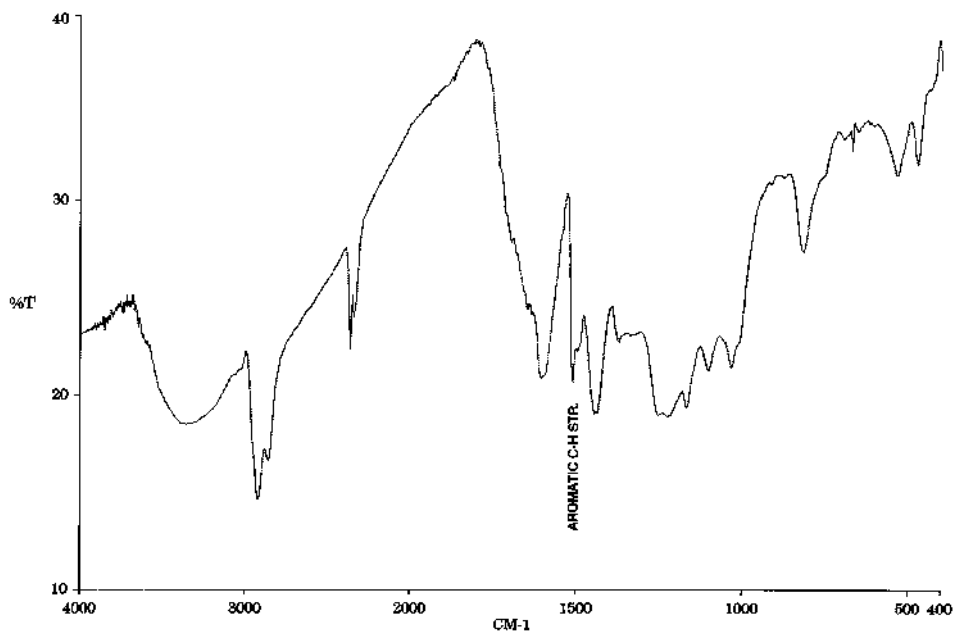


Figure 5: FTIR spectrum of Kimmeridge jet

5.8 FTIR analyses of archaeological assemblages

The artefacts analysed were from sites in two major military and civilian centres during the Roman period: Verulamium and York. It was hoped that results from the two different centres of Roman activity would provide a useful contrast for understanding the use of the various black lithic materials. York is close to the major British source of jet at Whitby, and the discovery of partly worked fragments of jet or similar materials has led to the suggestion that it was an important centre for the manufacture of these ornaments [30].

In total, 107 archaeological examples of black lithic materials were analysed using FTIR: 29 from York and 78 from Verulamium. There were several factors to be taken into account when analysing the artefacts, so that interpreting the infrared spectra to identify the different materials proved to be more difficult than the results from geological material had suggested.

5.8.1 Sampling methods

The archaeological material analysed ranged from large unworked and semi-worked fragments, to broken armlets, and small, complete, intricately carved beads. It was not possible to follow the same procedure that had been used for analysing geological samples (i.e. carefully quantified KBr discs prepared from homogenised samples of the raw material). Where possible, a small sample was removed with a scalpel from a broken edge, and ground by hand together with the KBr. Even this method would have been too destructive for many of the complete beads and pins. Some of the smaller artefacts from Verulamium were analysed using an infrared microscope, at the School of Chemistry and Applied Chemistry, University of Wales College of Cardiff. The constraints imposed on the sample preparation by the nature of the artefacts often resulted in spectra with much poorer resolution.

5.8.2 Effect of conservation resins on the IR spectra

Many of the artefacts analysed had been conserved, and impregnated with various consolidants. The conservation materials produce their own characteristic absorption peaks in the IR spectra from the artefacts. One of the consolidants frequently used was easily identified as polyethylene glycol 4000 (figure 7), which is one of the published methods for the treatment of deteriorating shale artefacts [31]. When the consolidant can be identified, it is possible to use spectral subtraction methods to remove the absorption peaks from an artefact's IR spectrum. Figure 8 shows the same spectrum as in figure 7 but with the PEG 4000 spectrum subtracted, leaving an absorption pattern which is much closer to that of an oil shale (figure 4).

Several artefacts showed unusual peaks which were not attributable to any of the conservation materials that are commonly used to treat jet and jet-like artefacts. In particular, many of the Verulamium artefacts had been excavated in the 1930's, 1940's and 1950's, so there was no record of the conservation treatment applied. A search of the library compiled by the Infrared Users Group may help to solve this problem, and identify the more elusive conservation resins.

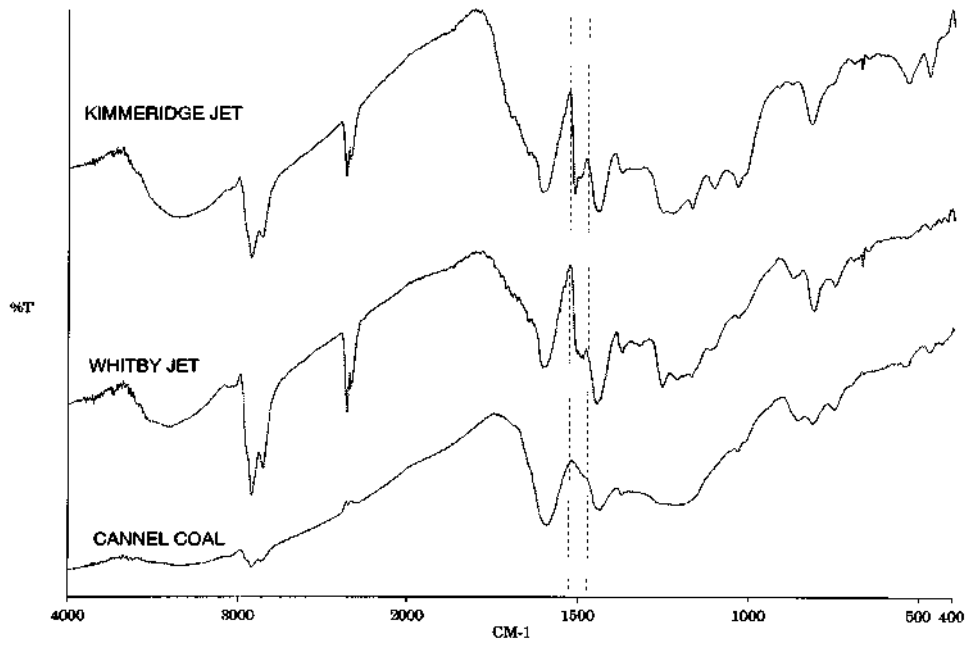


Figure 6: Comparison of the FTIR spectra of Kimmeridge jet, Whitby jet and cannel coal, showing the differences in the 1500 cm⁻¹ absorption

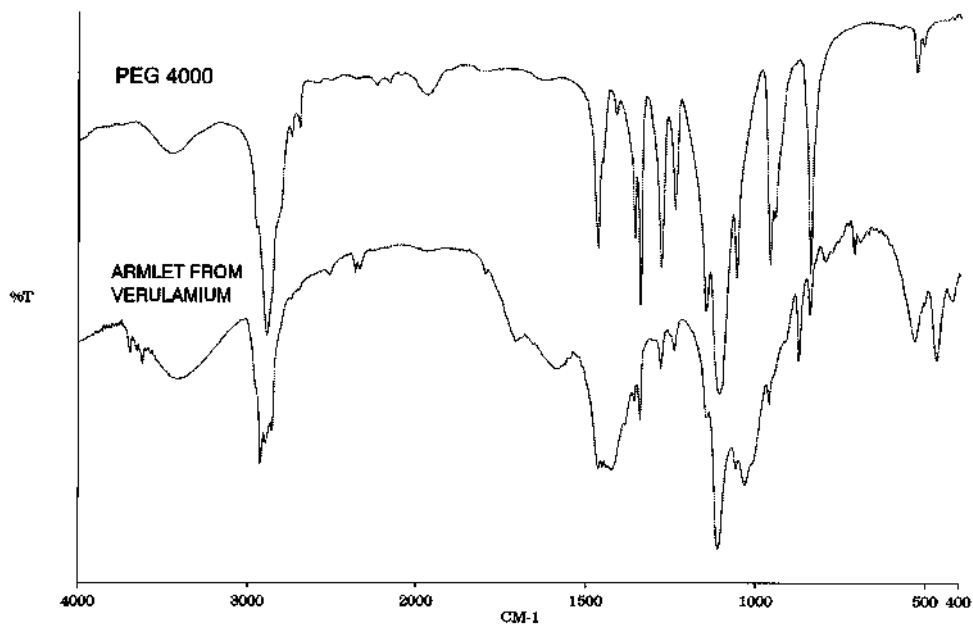


Figure 7: FTIR spectrum of a shale armlet which has been consolidated with PEG 4000

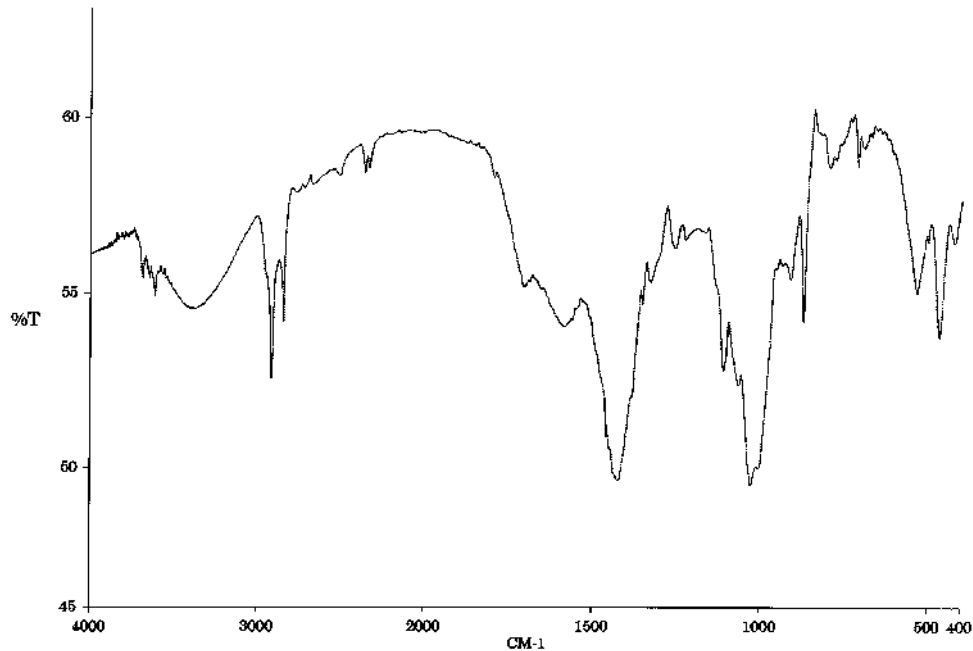


Figure 8: The same spectrum as in figure 7, with the PEG 4000 spectrum subtracted. The spectrum is now almost identical to the FTIR spectrum of an oil shale, shown in figure 4

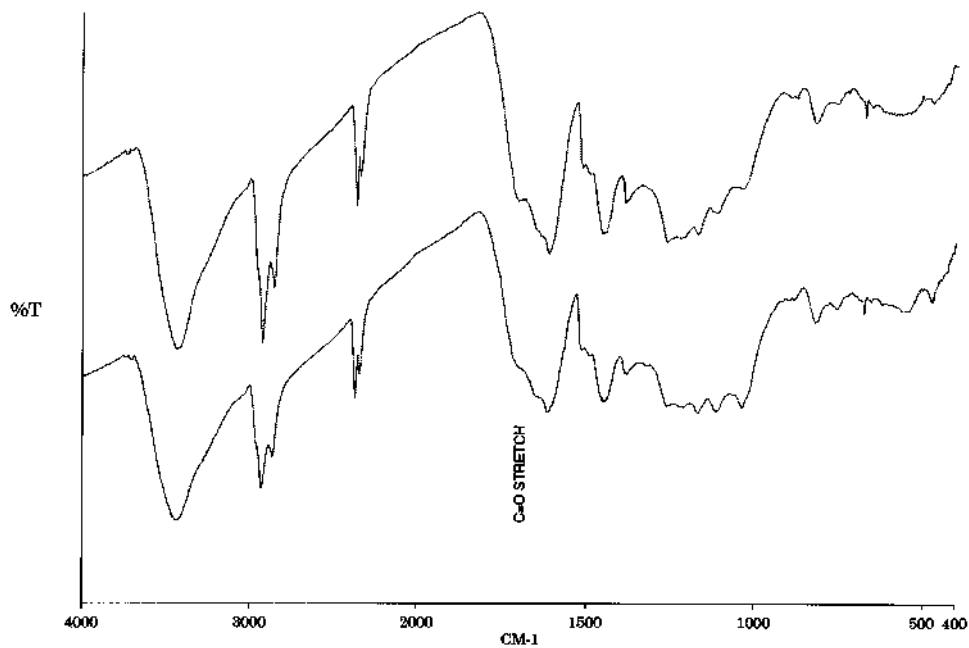


Figure 9: FTIR spectra of two artefacts from Verulamium, showing a C=O stretch at 1700 cm^{-1} , which is probably the result of oxidation of the jet

5.8.3 Oxidation of jet in the burial environment

One problem encountered in analysing archaeological jet-like artefacts has proven more difficult to monitor - and that is the changes to their chemical structure that take place in the burial environment. All the geological samples of Whitby jet analysed produced virtually identical, characteristic infrared spectra. None of the spectra from either the Verulamium or the York artefacts matched the Whitby jet spectra. Some were extremely close - such as the two illustrated in figure 9, which show the same combination of aromatic and aliphatic absorptions, and the characteristic shoulder at 1500 cm^{-1} . However, both spectra also show a marked absorption at 1700 cm^{-1} - a peak which usually appears in the spectra of lower rank coals, and is one of the discriminants used to identify lignite. Carbonyl peaks at around 1700 cm^{-1} also occur in the spectra of more mature coals that have oxidised. Detailed studies of oxidised coals have noted absorptions from carbonyl groups in the spectra of coal samples from near the mouth of a coal adit, as well as in the spectra of coals oxidised in the laboratory [32,33].

The samples taken from archaeological artefacts are necessarily very small, and from the surface of the artefact - which is the area most likely to have oxidised and to show the absorption from carbonyl peaks. These changes have made it difficult to distinguish jet from lignite amongst the archaeological assemblages; this is disappointing, since these two materials are also difficult to distinguish using X-ray fluorescence [7]. It is clear that more work is needed to monitor the changes in the IR spectra of the different materials that occur during burial. Low temperature air oxidation of samples in the laboratory may be one method of determining whether it is possible to distinguish between materials such as jet and lignite amongst the archaeological assemblages.

5.9 Conclusions

The initial FTIR characterisation of the geological reference material was promising, since the spectra could be interpreted in terms of the different geological depositional environments.

FTIR analyses of archaeological assemblages from Verulamium and York has enabled the artefacts to be classified into three groups: jet/lignite, cannel coal and oil shale. Distinguishing between jet and lignite proved to be difficult, partly because limitations in the sample size and preparation methods led to poor resolution, and partly because the jet appears to have oxidised, producing absorptions in the 1700 cm^{-1} region, which are usually characteristic of lignite.

A better understanding of the chemical changes taking place during burial in an archaeological environment may enable us to identify the material types more precisely using FTIR. The problems of poor resolution were improved by using the FTIR microscope; it seems that infrared microscopy is a more suitable technique for the analysis of archaeological artefacts, since the sample size required to obtain good spectra is minimal.

5.10 Acknowledgements

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