

Comparison of the structures of humic substances from aquatic and terrestrial sources by pyrolysis gas chromatography-mass spectrometry

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(Received July 14, 1982; accepted in revised form December 3, 1982)

Abstract—Four humic extracts isolated from terrestrial, marine, plankton and freshwater sources have been investigated by pyrolysis-gas chromatography-mass spectrometry. The results show that substantial quantities of phenols (some not derived from lignin), pyrroles and nitriles (derived from proteins, nucleic acids or porphyrins) are decomposition products. Small amounts of furans (derived from carbohydrates) and unsaturated ketones (derived from polycarboxylic aliphatic acids) are also present. The results illustrate how parent material, transport factors and biological activity affect the composition of humic extracts.

INTRODUCTION

NATURALLY occurring humic substances are a complex mixture of decayed plant and soil materials and the polymeric matrices produced by the random combinations of these materials with other organic substrates. The humic materials of soils and sediments have been studied for over 200 years; yet it is only in the last 40 years that similar studies have been carried out on aquatic humic substances.

It is desirable that humic substances from a wide variety of sources be investigated so that the factors influencing their formation can be elucidated. The nature of the parent material may be important in determining structure since, if the decomposing plant life contains a larger proportion of one class of chemical compounds than another, then the organic matter will contain different degradation products. Secondly, geographical factors can also be important since humic substances may be transported from one environment to another by water. A third factor influencing structure is the degree of microbiological activity in the environment. Biodegradable material will be rapidly transported in an environment rich in saprophytic microorganisms. Climate will affect the type of vegetation and microbiological activity in a particular environment, and through them the degree of chemical transformation. High rainfall will leach many humic precursors such as polyphenols and transport them from one environment to another.

Over the last ten years knowledge of the structure of humic substances has advanced mainly due to the development of two important techniques, namely, pyrolysis-gas chromatography-mass spectroscopy (py-GC-MS) and Fourier transform nuclear magnetic resonance spectroscopy.

Py-GC-MS has been used to characterize a number of geochemical samples from a variety of sources in recent years. This has been due to the development of micro-scale pyrolysis techniques and also high res-

olution capillary columns capable of separating a large number of the components obtained in the very complex mixtures of pyrolysis products from geochemical samples. A description of recent applications of py-GC and py-GC-MS to fossil fuel studies has been given elsewhere (PILB, 1982).

The ¹³C-n.m.r. spectra of humic substances derived from terrestrial (NEWMAN *et al.*, 1980), freshwater (WILSON *et al.*, 1981a), marine water and planktonic sources (GILLAM and WILSON, 1982; WILSON *et al.*, 1983b) have been published recently. Similar studies have been completed by Hatcher and his group (HATCHER *et al.*, 1980a,b, 1981a,b.). The results from these studies showed that the various humic substances contained different carbohydrate contents and different amounts of aromatic carbon. Similarities in the structures of freshwater and terrestrial humic materials were identified (indicating that the freshwater material was of terrestrial origin), and between marine humic material and planktonic extract. In this work we investigate the structure of the same humic materials using (py-GC-MS) and make comparisons between the two techniques and between humic materials from different sources.

EXPERIMENTAL

Extraction and preparation of humic materials

The methods of extraction, preparation and separation of the humic materials have been described (NEWMAN *et al.*, 1980; WILSON *et al.*, 1981a; GILLAM and WILSON, 1982; WILSON *et al.*, 1983b). Details of origin, type and elemental composition have also been published with the extraction methods but are repeated here in Table 1 for completeness. The types and forms of carbon determined by ¹³C-n.m.r. are also shown.

Pyrolysis-gas chromatography-mass spectrometry

Py-GC-MS analyses of the humic materials were performed using a Pye Curie-Point Pyrolysis system interfaced directly to a Finnigan 4023 GC-MS and INOCOS data system.

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