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Humic Substances in the Aqueous Environment: Implications for the Environmental Chemistry of Trace Metals

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Organic compounds are known to be widespread in the hydrosphere and, in many cases, are important contributors to the physical and chemical nature of ground and surface waters (Rashid, 1985). This abundance and importance are manifestations of the intricate inter-relationship between the hydrosphere and all living matter (Hem, 1989). Organic chemicals are introduced into the hydrosphere by natural interactions with the biosphere and the lithosphere and by non-natural, anthropogenic activities.

Since passage of the Safe Drinking Water Act by the U.S. Congress in 1974, a great deal of scientific research has focussed upon characterizing the nature and the distribution of synthetic organic chemicals. During the same time period a great deal of less "glamorous" research has been conducted in the field of naturally occurring water-borne organics. This is a departure from the historic treatment of organic geochemistry as the study of petroleum, coal, and oil shale (Thurman, 1985). Instead, the more recent research has revealed the relative significance of a certain category of aqueous-phase organic compounds collectively termed "dissolved organic carbon."

Dissolved organic carbon (DOC) is a term that describes an enormous library of organically-derived substances that are present in natural waters. An arbitrary definition places the maximum size of "dissolved" organic carbon at .45 microns (Thurman, 1985). DOC may include fatty acids, carbohydrates, hydrocarbons, viruses, hydrophilic acids polysaccharides, proteins, lignin, amino acids, alcohols, terpenes, sterols, heterocyclics, long-chain acids, and other derivatives of plant, animal, and micro-organism decomposition (Thurman, 1985; Dragun, 1988). Many of these substances are readily degraded by the enzyme systems of micro-organisms into simpler monomers that are then re-utilized by plants and other organisms for metabolic activity and growth. In many instances, however, these monomeric organics re-co-polymerize with each other to form humic substances such as humic and fulvic acids (Dragun, 1988). Humic substances represent the residuum of biological breakdown of organic matter (Rashid, 1985) and are widely recognized as significant controls on the chemistry of metals.

Humic substances are defined by Aiken, et. al.(1985) as "a general class of biogenic, refractory, yellow-black organic substances that are ubiquitous, occurring in all terrestrial and aquatic environments." Humic substances are



literally everywhere. Longmire (Pers. Comm., 1989) characterizes humic substances as likely candidates for the most chemically active types of organic carbon and yet they remain the least understood. Because humic substances are the most abundant and geochemically reactive types of DOC (Rashid, 1985), they significantly influence the chemical makeup of ground and surface water.

Humic substances are known to be extremely stable at geological time scales (Dragun, 1988). This stability is one of the more important characteristics of humic substances and is the reason they form the dominant type of organic material in soil profiles (Stevenson, 1985). Humic substances comprise 50 to 70% of total DOC (Thurman, 1985; Longmire and others, 1989). In the absence of oxygen, humic substances can be preserved for 100's of millions of years (Longmire and others, 1989). For instance, humic coal deposits date back to the Carboniferous (Manskaya and Drozdova, 1968). Humic substances have also been isolated from the Archean Onverwacht Chert (Jackson, 1975). As ancient deposits, humic substances provide clues to the development of early life on earth (Rashid, 1985).

Humic substances are divided into three major fractions by Aiken et. al. (1985) based upon their relative aqueous solubilities after isolation. These fractions are: Humin, which is insoluble; Humic Acid, which is soluble above a pH value of 2 and insoluble below a pH of 2, and Fulvic Acid, which is soluble at all values of pH. From the standpoint of importance to aqueous systems, the solubility of humic and fulvic acids in waters of "normal" pH make them abundantly available for chemical interaction. Humic and fulvic acids are significant because they have a strong tendency to form complexes with metallic cations, they participate in weathering reactions for many minerals, they influence the environmental fate of synthetic organic compounds such as chlorinated solvents and pesticides and they react with disinfectant chemicals added to drinking water to form carcinogenic trihalomethanes (Aiken et. al, 1985; Thurman, 1985; Rashid, 1985; Langmuir, 1989).

The following discussion is divided into two major sections: the first is a review of general humic and fulvic acid geochemistry; the second is a scrutiny of the importance of these substances in the transport and deposition of certain metallic ore deposits.

Humic and fulvic acids are identified as the agents responsible for coloring natural waters (Hem, 1989). Straw or tea colored waters are found to be associated with swamps, bogs, and wetlands (Thurman, 1985). The geochemistry of colored waters has always been associated with the chemistry of transition metals (Malcolm, 1985). One of the dominant geochemical characteristics of humic and fulvic acids dissolved in water is the capacity to form stable complexes with metal cations. This capacity is in large part a result of the large number of functional groups that inhabit various structural positions on the periphery of the humic substance (Perdue, 1985).

Thurman (1985) defines humic substances as "polyelectrolytes of carboxylic, hydroxyl, and phenolic functional groups." Other, less abundant, functional groups include enolic, carbonyl, quinone, and amino groups (Rashid, 1985) as well as ethers, ketones, aldehydes, lactone esters, amines, and amides (Thurman, 1985). The various functional groups are the reactive sites on the humic molecules (Rashid, 1985). Carboxylic functional groups are among the most important in humic substances because they are the most reactive (Rashid, 1985). The chemical properties of carboxylic functional groups are generally characteristic of the bulk of the other functional groups.

According to Thurman (1985), humic substances average 5 to 10 carboxylic functional groups per molecule. Fulvic acids characteristically have a larger number of carboxylic groups than do humic acids (Longmire and others, 1989). Carboxylic functional groups are important for several reasons. One of the more important of these is their contribution to aqueous acidity via the protonation reaction:



Thus carboxylic functional groups dissociate to liberate a proton, thereby creating an anionic charge on the functional group (Longmire and others, 1989). According to Thurman (1985) the dissociation constant:

$$K_a = [R-COO^-][H^+] \\ \log K_a = -4.8 = \text{a strong acid} \\ pK_a = -\log K_a = 4.8$$

(pK_a) is a relative measure of the dissociation strength of an acid. pK_a 's for the functional groups associated with humic substances range from 4.8 to 13 (Thurman, 1985). As the strong pK_a suggests, carboxylic functional groups are considered to contribute substantial numbers of hydrogen ions to equilibrium waters. Antweiler and Drever (1983) studied soil weathering solutions in which organic carbon was the dominant control on solution pH. In fact, a major portion of the total acidity of humic substances is attributed to carboxylic groups (Perdue, 1985). This acidification is an important characteristic in the weathering behavior of humic substances (Thurman, 1985).

The loss of the hydrogen ion as the result of the dissociation leaves the remaining carboxylate with a negative net charge. This anionic charge, when multiplied by the number of dissociated functional groups, yields a large charge imbalance. This strongly ionic character enhances the aqueous solubility of the humic substance (Stevenson, 1985).

Dissociating a carboxylic group increases the solubility of that functional group by four orders of magnitude (Thurman, 1985). This is significant because the dominant chemical activity of humic substances occurs as dissolved substances. Another aspect of increased solubility is that it increases the

effective residence time of the humic substance in the water column (Rashid, 1985). The increased occurrence of carboxylic functional groups within the fulvic acid structure is responsible for its increased solubility relative to humic acid (Longmire and others, 1989). As a result, fulvic acid is retained in solution more effectively than humic acid (Thrumman, 1985). (Recall that solubility as a function of pH is used to define humic and fulvic acids).

Perhaps the most significant aspect of carboxylic dissociation and the attending anionic net charge is the affinity of humic substances for metallic cations. "Because of their high geochemical reactivity, all humic compounds absorb and accumulate large amounts of trace and transition metals" (Rashid, 1985, p. 109). Humic substances form metallo-organic complexes of ranging stability with a large number of metals. As a result, humic substances are significant controlling factors on the solution, precipitation, mobilization, migration, recycling, redistribution, and concentration of many chemical elements that form cations in aqueous solution (Rashid, 1985). A list of the chemical elements that have been implicated in complexation reactions with humic substances includes the following*:

Ac	Ag	Al	As	Au	B	Ba	Bi	Ca	Ce
Cd	Co	Cr	Cs	Cu	Dy	Er	Eu	Fe	Gd
Ge	Ho	K	La	Lu	Mg	Mn	Mo	Na	Nd
Ni	Pb	Pm	Po	Pr	Ra	Rb	Sb	Sm	Sr
Tb	Th	Tm	U	V	Y	Yb	Zn	Zr	

*Sources: Malcolm, (1985); Stevenson, (1985); Raspor and others, (1984); Frimmel and others, (1984); Rashid, (1985); Drever, (1988); Langmuir and others, (1989); Cameron, Schruben & Boudette, (1986) and Szalay (1964).

The concentration mechanism of many of these elements have been the study of economic geologists for decades. Until the middle of the century these geologists focussed their attention upon the inorganic chemistry of metals (Rashid, 1985). Much work has been done since then on the association of trace, transition, and heavy metals in organically derived sedimentary materials such as crude oil, oil shales, coal, kerogen, etc. (Manskaya & Drozdova, 1968). It is now recognized that the occurrence of metals above crustal abundance in association with most carbonaceous material is the result of the action of humic substances (Rashid, 1985). Humic substances are responsible for many economically important mineral concentrations (Turner-Petersen, 1985).

This action may be illustrated by comparing experimental solubility data against calculated solubility data for various metals. Since aqueous solubility is the dominant factor controlling metal mobility, migration and redistribution, changes in solubility must also change the geochemical behavior of the metal (Rashid 1985). Complexation with humic substances can often lead to unexpectedly high concentrations of metals in solution (Drever, 1988). According to Siever (1971), the calculated solubility of iron in most natural waters at "normal" pH values and redox conditions is 108 to 109 times less than

observed concentrations. A plausible explanation for this increase is the presence of humic substances which react with iron to form soluble iron-humates (Rashid, 1985).

A positive linear correlation has been shown to exist between the humic substance content of soil and the concentration of the trace metals Cu, Co, Mn, Ni, and Zn (Manskaya & Drozdova, 1968). These same five trace metals have been found to be enriched in the burrow walls of polychaete worms in coastal marine waters by the action of humic substance deposition (Over, 1990). Humic peat deposits are known to geochemically enrich uranium by 4 orders of magnitude over abundances in water (Szalay, 1964). Enrichment coefficients for vanadium, nickel, lead, and silver exceed 103 (Rashid, 1985). Polyvalent cations have greater enrichment factors than do monovalent cations (Szalay, 1964).

Humic substance participation in metal complexation reactions is controlled by the ionization of functional groups. Functional group ionization is determined by the acid dissociation constant, K_a , of each functional group. As a result of functional group dissociation, humic substance solubility increases. Dissociation also creates a more acidified aqueous environment by liberating hydrogen ions. Increasing the hydrogen ion concentration (decreasing pH) also increases the inorganic solubility of most metals. Thus, at low pH's, humic acid carboxylic functional groups will be more completely dissociated, resulting in a larger net molecular charge imbalance. This charge imbalance is offset by a larger mass of metallic cations inorganically dissolved by the increased hydrogen ion concentration. Therefore, the humic substances set up a "positive feedback" for maximizing the geochemical concentration of metals via metallo-organic complexation.

The degree to which humic substances influence the geochemical behavior of other chemical species is disproportionate to their relative abundance in water (Rashid, 1985). One implication of the influence humic substances have upon metal chemistry is the concentration of these metals into economically significant ore deposits. In the past two decades geologists and geochemists have recognized numerous instances in which humic substances have been the critical environmental factor culminating in economic concentration of ore metal (Turner-Peterson 1985).

Perhaps the most well documented associated between humic substances and an economic ore is the example of uranium. Uranium is almost always found in concentrations associated with organic matter (Manskaya and Drozdova, 1968; Nakishima and others, 1984). In a general sense, the association of uranium with organic matter has traditionally been considered to be a result of the negative redox condition that attends partially decaying organic matter. An important aspect of humic complexation, however, is that uranium reduction is not necessary for complexation (Turner-Peterson, 1985; Cameron and others, 1986). In oxidizing fresh waters, uranium exists as the soluble hexavalent form.



At low pH's the uranyl cation $(\text{UO}_2)^{2+}$ predominates. At a pH above 5 the double uranyl dicarbonate complex $\text{UO}_2(\text{CO}_3)_2(2\text{H}_2\text{O})^{2-}$ is dominant. With increasing pH the soluble uranyl tricarbonate complex $\text{UO}_2(\text{CO}_3)_3^{4-}$ increases in abundance (Langmuir, 1978). These dicarbonate and tricarbonate complexes are readily fixed by humic substances (Turner-Peterson, 1985).

Primary uranium ore deposits in the Grants Uranium Region of New Mexico are considered to be the direct result of humic acid uranium concentration (Turner-Peterson, 1985). These deposits accumulated in upper Jurassic sandstones along the southern margin of the San Juan Basin. The significance of the humate in the deposit is such that the ore-bodies should really be considered as humate masses with associated uranium (Squyres, 1970 in Turner-Peterson, 1985). The significance of metallo-organic concentration of uranium is brought into perspective when considering that the Grants uranium region has produced 50% of the uranium supply of the United States as of 1985 (Turner-Peterson, 1985).

In the Grants uranium region, the humate-uranium ore was formed by introduction of humic acid rich groundwaters into previously deposited sands. The source of humic acid was indigenous organic detritus deposited with fresh water mudflat sediments. During burial compaction, basinal fluids were expelled and flushed through the mudflat facies into the adjacent sands. Uranium complexed humic acids in the basinal fluids reacted with magnetite, ilmenite, and feldspar in the sands. This chemical weathering resulted in cation loading of the functional groups on the humic substances thereby decreasing solubility and resulting in precipitation of humate.

Peat deposits have been well documented to enrich a variety of metals. The "Flodelle Creek deposit" described by Johnson, Otton and Macke (1987), records the Holocene accumulation of uranium and a host of other metals by organic matter controlled adsorption and ion exchange. The Flodelle Creek deposit was discovered to contain a large concentration of precipitated uranium that was virtually non-radioactive because secular equilibrium with shorter half-lived progeny had not had time to develop.

Since the time of the Flodelle Creek discovery, peatland and wetland deposits have been subjected to more detailed scrutiny with regard to humic and metals chemistry (Otton, 1991; Owen, 1991). Cameron, Schruben and Boudette (1987) analyzed the contents of a peat deposit near Fern Lake in Leicester, Vermont and found unexpectedly elevated concentrations of metals and uranium. The uranium was found to be present at levels equivalent to the lowest ore grade of the Flodelle Creek deposit: some 467 parts per million. The uranium anomaly was surprising because of the absence of a logical bedrock source. Other wetland and peatland areas of New England were also shown by these workers to significantly enhance the concentration of metals. It is probable that many deposits of this type can be found in other areas of Vermont.

One aspect of the concentration of metals that is worthy of consideration is the potential liberation of metals when peatlands or wetlands are drained or otherwise disturbed. In Colorado, peat mining was found to allow oxidizing meteoric waters to penetrate the peat mass. Unlike other metals, uranium is soluble in oxidizing waters. The exposure of the peat led to the liberation of a large mass of uranium to the environment (Otton, 1991).

To summarize, humic substances comprise the dominant form of dissolved organic matter in natural systems. There are few chemical reactions that take place in natural waters or soils that are not influenced by these substances. They play a significant role in the geochemical evolution of soils and sedimentary deposits and the fluids that flow through them. They are a dominant factor in the environmental fate of metallic cations and are responsible for the economic concentration of metallic ores. They influence the bioavailability of metals and may also alter metal toxicokinetics. In spite of their ubiquity and geochemical significance, humic substances remain poorly understood and little known to the geological community. Given the well documented importance of humic substances in many hydrogeochemical reactions, it is interesting that environmental sampling does not attempt to quantify humic substance activities, especially when the chemistry of metals is concerned. Perhaps as research continues, a greater understanding of humic substance interactions will be achieved by the geological community.

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In October of 1991 the Center for Disease Control (CDC) lowered the blood concentration at which an individual would be considered to lead poisoned. The level was reduced from 25 micrograms per deciliter ($\mu\text{g}/\text{dl}$) to 10 $\mu\text{g}/\text{dl}$. This change was based upon research indicating that a small amount of lead in a child's blood dramatically increases the risk of irreversible central nervous system damage leading to learning disabilities, behavioral disorders and impaired musculo-skeletal coordination. A child is more susceptible to lead poisoning because a child absorbs approximately 50% of the lead ingested (compared to 5% to 8% absorption for adults). A child's hand to mouth behavior also results in a greater ingestion of lead per unit bodyweight.

In response to the new standards from the CDC, the Vermont Department of Health lowered the blood lead level at which a child is considered to be poisoned to 10 $\mu\text{g}/\text{dl}$ and required physicians to report incidences of poisoning to the Health Department. In addition, the Department directed the new Toxicology and Risk Assessment Program to work in developing a lead program. Currently the Lead Program is involved in:

- Lead inspection of homes with children having blood lead in excess of 20 $\mu\text{g}/\text{dl}$ or greater
- Working with a group of State officials to draft legislation that would regulate lead abatement and establish a lead screening program.
- Public education regarding lead hazards and the development of informational materials.
- Working with the City of Burlington to address lead issues that affect Burlington residents.

Efforts to implement lead program initiatives are ongoing. Please call the Toxicology and Risk Assessment Program at 1-800-439-8550 or 863-7220 if you have any questions regarding lead issues.