

Humic Substances in the Sediments of Lake Edku, Egypt: II – Its Role in the Accumulation of Trace Metals

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ABSTRACT. Humic and fulvic acids extracted from Lake Edku sediments are highly enriched in Cu, Zn and Ni and to small extent in Co when compared to the bulk sediment, but depleted in Fe and Mn. The mean concentrations of Cu, Ni, Zn and Co in the humic acid extract were, 359, 208, 188 and 47 $\mu\text{g g}^{-1}$ while their concentrations in the bulk sediment were 65, 84, 129 and 40 $\mu\text{g g}^{-1}$ respectively. Humic acid showed relatively higher concentrations for all elements except Zn. The presence of humic acid in solution enhances the transfer of Cu, Cd, Ni and Zn into the suspended matter at pH's higher than 6, through the precipitation of metal-humic acid complexes and adsorption on freshly precipitated humic acid. Fe showed another mechanism which, most probably, is the formation of colloidal hydrous oxides.

Introduction

In recent years, the fluxes of many trace elements from terrestrial and atmospheric sources to the aquatic environment have increased (Forstner and Wittmann 1981). A major fraction of trace metals is found associated with bottom sediments as a result of complex physical, chemical and biological processes. The processes and factors controlling the scavenging of trace metals by the sediments must be understood if impact on the environment is to be predicted.

The duality between the dissolved and particulate phases of trace metals in the aquatic environment leads to the complexity in the understanding of their environmental geochemistry (Bourg 1983). Exchange across the water-solid interface occurs in all the aquatic environments. Several mechanisms are sharing in this process of

which adsorption is of significant importance in regulating the concentration of trace metals in natural water (Davis 1984).

The role of organic matter, particularly humic substances in trace metal geochemistry has been stressed by several authors. Hunter (1980), Davis (1982) and Bourg (1983) argued that organic matter should be included in adsorption experiments on model surfaces to represent correctly the natural systems. It has also been shown (Bourg 1986) that the presence of complexing ligand, like humic substances, in natural water may significantly alter the adsorption behaviour of trace metals on solid surfaces. Organic coating on natural particles may also influence the adsorption process (Faguet 1982).

This work aims at investigating the partition of Fe, Mn, Zn, Ni, Cu and Co between the mineral phase and humic substances and the role of humic acid in the adsorption of these metals on lake sediments.

Area of Study

Lake Edku (Fig. 1), one of the shallow coastal lagoons of the Nile Delta, lies to the west of the Rosetta Branch at 30°15'N and 31°15'E. The lake, 115 km² in area, has an average depth of 1 m and communicates with the Mediterranean Sea (Abu-Qir Bay) through a narrow two meter deep channel (Bougaz El-Maadia) situated at its north-western extremity. The lake receives annually 1.0×10^9 m³ of drainage water through the main agricultural drains situated at its north-eastern extremity. Various studies carried out on the Lake have shown that the hydrobiological, chemical and geological characteristics vary greatly due to the direct influence of the drains in the south-eastern part and due to the exchange with the sea at the western extremity. The central part is greatly influenced by the massive growth of floating and rooted macrophytes (El-Sarraf 1976). More detailed description of Lake Edku is given in El Sayed *et al.* (1993).

Material and Methods

Based on the main factors affecting the environmental conditions, the lake was subdivided into three sub-basins. Five sampling stations (Fig. 1) were selected to represent these sub-basins.

Sample collection, treatment and detailed analytical techniques used for the separation and purification of humic acid are given in El Sayed *et al.* (1993).

Trace metals in lake sediments were determined in powdered samples after complete acid dissolution (HNO₃/HClO₄/HF) in digestion bomb at 140°C for 10 hours. Measurements were carried out using Atomic Absorption Spectrophotometer (Varian 125). All acids used were of Merck suprapure grade. Relative standard deviation of six replicates of one of the analyzed samples was less than 5% for Cu, Mn, Co and Ni and 8% for Fe and Zn. For the digestion of humic and fulvic acids the same technique was applied but using a nitric/perchloric mixture (3:1).

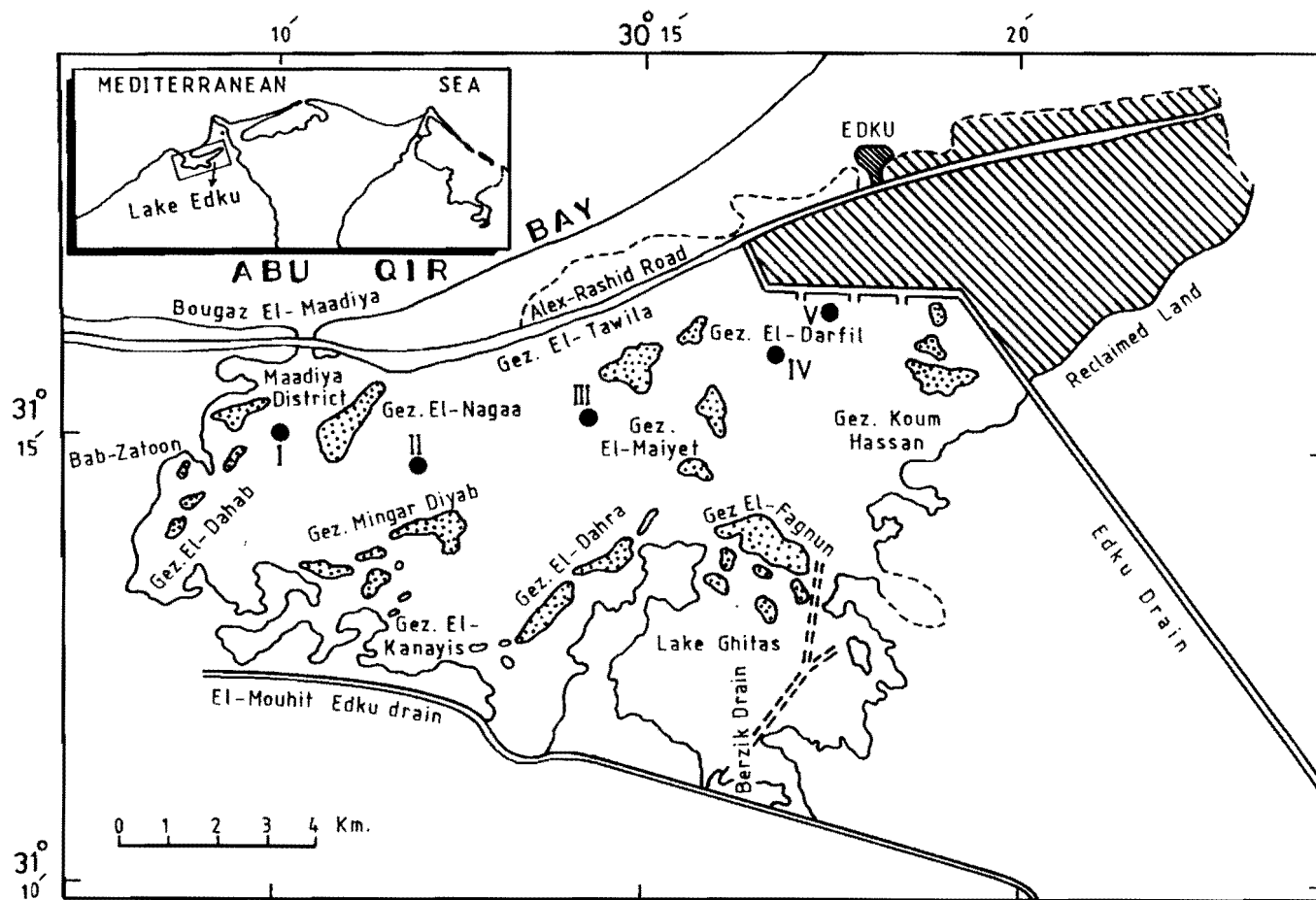


FIG. 1. Area of study and sampling stations.

Experimental

A series of laboratory experiments was done to test the role of humic acid (HA) in the adsorption of Fe, Cd, Zn, Ni and Cd on lake sediments in natural lake water. Lake water was filtered using pre-ashed acid cleaned Whatman GF/C. The water was stripped from humic substances by passage on the resin XAD2, pH was then adjusted to about 7 and the water was stored in pre-cleaned dark glass bottle until use. Sediments ($< 63 \mu\text{m}$) were freed from the loosely bound metals by shaking with 0.1 M HCl for 4 hr, then washed thoroughly with deionized water and dried at 50°C . All experiments were carried out using 100 ml of treated lake water. Sediment concentration was maintained at 200 mg l^{-1} and metal concentration was fixed at 4 mg l^{-1} . Time of equilibration was 1 hr with continuous shaking. For each metal, three series of experiments, covering the pH range 3-9 and a humic acid/metal ratio of 0, 1/2 and 1/1 (W/W) were undertaken. The final pH of the experiment was adjusted after the addition of all the components and it was not controlled during the experiment. After the experimental run, samples were filtered using prewashed GF/C Whatman membranes and humic acid and the trace metals were measured in the solution and the quantity adsorbed was calculated as the difference from the original concentration.

Results and Discussion

Humic substances

The results of total organic matter (TOM), humic substances as humic and fulvic acids, mud content and the ratios between total humic substances and OM in the sediments of Lake Edku are given in detail in El Sayed *et al.* (1993). The mean humic substances concentration for the whole Lake is $26 \pm 31 \text{ mg g}^{-1}$. It appeared that TOM and THS components were fairly good correlated with the mud content of the sediments. Meanwhile, sediments close to the drains seems deficient in organic matter and humic substances when normalized to their mud content. However, organic concentration in this area is comparable to values measured in other Egyptian delta lakes, and reflects the OM content of the delta soil. Humic substances in the lake represented $44 \pm 30\%$ of the TOM.

Trace Metals

In lake sediments

Total trace metal concentrations in the bulk sediments of Lake Edku as well as the average concentration of trace metals for recent lake sediments (Forstner and Wittmann 1981) are listed in Table 1. The results obtained in Lake Edku are closely comparable to those found in recent pelitic lake sediments. The most characteristic features of trace metals distribution is the relative general increase of concentrations at the near drain stations. No definite correlation could be identified between trace metal concentrations and both organic matter and mud contents of the parent sediments. The concentrations of iron and manganese are particularly low at stations I

and III characterized by the lowest mud content and the highest organic matter respectively. This may result from the fact that both iron and manganese are mainly present in the residual form in the fine fraction (Gibbs 1977). It could be seen from Table 1 that all the elements show lower concentrations at station III with respect to stations IV and V. Organic matter enrichment in the sediments of the central basin results from the accumulation of fresh macrophyte debris which contained certainly lower amounts of trace metals than the terrigenous sediments. It has also been shown (Faguet 1982) that organic matter may screen the possible sites of adsorption particularly for Zn.

TABLE 1. Organic matter (OM), total humic substances (THS), humic acid (HA), fulvic acid (FA), mud content (< 63 μm) and THS/TOM in the sediments of Lake Edku.

St	OM %	FA mg g^{-1}	HA mg g^{-1}	THS mg g^{-1}	Mud %	THS/TOM %
I	2.18	5.49	0.95	6.44	9.8	29.54
II	4.42	11.88	14.88	26.76	32.8	60.54
III	8.80	47.18	38.36	85.54	79.4	97.20
IV	2.46	3.78	0.95	4.73	50.9	19.23
V	2.65	3.30	1.07	4.37	49.2	16.49
Mean	4.10	14.33	11.24	25.57	44.42	44.60
S.D.	2.48	16.71	14.59	31.14	22.90	30.60

In humic substances

Abundance of trace metals in both humic and fulvic acids is listed in Table 2. It seems that concentrations in the humic extract from stations IV and V are more or less, higher than in the other stations which agrees with the concentrations found in the parent sediments. Evidently, all trace metals except Zn are more abundant in humic acid than in fulvic acid. In this concern, our results agree with other previous studies. Humic acids isolated from Lake Ontario sediments contained higher concentration of Co, Cd, Cu, Zn, Cr, Fe and Mn than the fulvic acids extracted from the same sediments (Nriagu and Coker 1980). However, fulvic acids extracted from the sediments of Mahakam estuary showed higher Cu, Cd, Pb and Zn concentrations than the humic acids extracted from the same samples. Desai and Ganguly (1970) showed that humic acid from marine sediments contained higher Cu and Zn but lower Al and Fe than fulvic acid from the same sediments. From the previous observations, it seems that no fixed order could be attributed to humic and fulvic acids regarding their trace metal contents. Probably, several factors, like origin, modifications during transportation and post depositional alterations may control the trace metal/humic and fulvic acids interactions and define their trace metal contents. The order of relative abundance in humic acid is $\text{Fe} \gg \text{Cu} > \text{Ni} > \text{Zn} > \text{Mn} > \text{Co}$ while in fulvic acid, the order is $\text{Fe} \gg \text{Zn} > \text{Ni} > \text{Cu} > \text{Mn} > \text{Co}$ (Table 2). The order of relative abundance in humic acid agrees with the order of bonding strength of metal ions onto humic acid (Jonasson 1977). The difference between humic and fulvic acids possibly means differing binding sites and/or binding mechanisms.

TABLE 2. Trace metal concentrations in humic and fulvic acids and in the bulk sediments (in $\mu\text{g g}^{-1}$).

St	HS	Fe	Cu	Ni	Zn	Mn	Co
I	HA	2834	246	221	228	170	32
	FA	1676	107	185	263	96	33
II	HA	2400	190	160	97	33	39
	FA	1676	91	175	269	250	40
III	HA	2313	259	197	227	73	34
	FA	1147	122	87	244	43	27
IV	HA	2542	479	141	152	147	60
	FA	1940	196	236	313	99	24
V	HA	2522	421	323	324	240	69
	FA	2341	198	262	275	218	38
Mean	HA	2520	359	208	188	153	47
	FA	1760	143	189	294	141	32
Bulk sediment		44400	65	84	129	767	40

This order does not reflect the real participation of humic substances to the trace metals pool in the lake sediments as could be seen from Table 3. The greatest contribution is that for Zn and Cu followed by Ni, Co, Mn and Fe respectively. This contribution may reach as high as 60% for Zn and more than 40% for Cu reflecting the importance of humic substances as a sink for these metals in lake sediments (Fig. 2). Fe and Mn are present at concentrations lower than those found in sediments while Cu, Zn and Ni are present at much higher concentrations. Copper, the metal which forms organic complexes of the highest stability constants (Bourg 1983) showed the highest concentration in the humic substances relative to its concentration in the bulk sediments. For the purpose of comparison, contribution from organic-bound metals to the bulk sediments from different environments are given in Table 4.

TABLE 3. Relative contribution of trace metals in humic substances to the bulk sediments (%).

St	Fe	Mn	Zn	Ni	Cu	Co
I	0.07	2	2	3	2	1
II	0.18	7	7	8	11	3
III	0.79	13	61	25	43	16
IV	0.03	1	1	2	2	5
V	0.03	2	2	2	2	8
Mean	0.22	5	15	8	12	7
S.D.	0.32	5	26	10	18	6

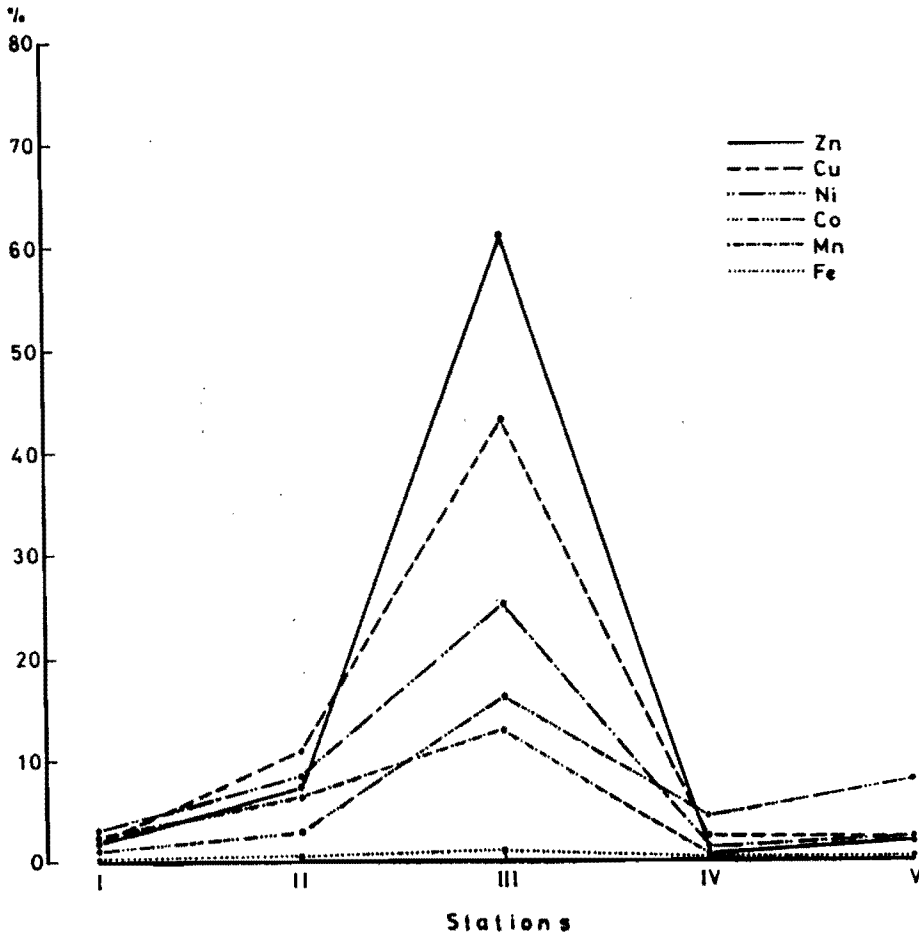


FIG. 2. The contribution of humic substances to the trace metal content in the bulk sediment.

TABLE 4. Contribution of organic-bound elements in sediment and suspended matter from different localities (%).

Locality	Fe	Mn	Zn	Cu	Ni	Co	Reference
Amazon River (SM)	7	5	-	6	13	19	Gibbs, 1977
Yukon River (SM)	11	6	-	3	16	13	Gibbs, 1977
Kara River estuary	-	1-11	9-15	4-29	13-31	21-34	Prohic, 1987
Yamaska and St. Francois River Quebec (SM)	3.8-4.8	1.4-1.7	5-6	31-52	4.4-6.2	12-13	Tessier <i>et al.</i> , 1980

(SM) Suspended matter.

Adsorption of Trace Metals on Lake Sediments

The adsorption behaviour of Fe, Cu, Zn, Ni and Cd as function of pH in the presence of varying proportions of sedimentary humic acid in natural lake water, stripped of its original dissolved HS is shown in Fig. 3.

1. In the absence of humic acid

Only Fe, Zn and Cu showed appreciable adsorption (> 10%) at pH 8. Iron disappears rapidly from solution, more than 90% were adsorbed at pH 6. At pH 8, almost all iron disappeared from solution. Fe (III) has a very low solubility at the natural pH (8.2). In natural water, it is mainly found in a colloidal form rather than in true solution (Sholkovitz *et al.*, 1978). Coagulation of iron in estuarine water has been partly attributed to its presence in the colloidal form as hydrous oxides. Fe (III) has very high partition coefficient (K_D), in natural water systems, more than 98% of its concentration is found in the particulate form (Gibbs 1977; El-Sayed 1988). Kendroff and Schnitzer (1980) stated that insoluble iron hydroxide may contribute to the adsorbed iron fraction at pH higher than 5.

It was therefore concluded that precipitation of iron oxyhydroxide as well as adsorption are sharing in the transfer of iron from the dissolved to the particulate form, precipitation increases with increasing pH as a result of the decreasing solubility of Fe (III). On the other hand, Cd which shows the lowest adsorption within the tested metals, is the metal with the smallest surface complex formation constants. The stability constants of its inorganic complexes, particularly chlorocomplexes are very high (Bourg 1983). Cd is not as competitive, for surface sites, as the alkaline earth elements like Na. These reasons have been advanced to explain Cd desorption in the lower part of estuaries (Valenta *et al.*, 1986).

Adsorption of Cu increased slowly between pH 3 and pH 6 (12-22%). Cu showed the adsorption edge at pH 6-7 in agreement with the results of Cu adsorption on hydrous oxides (Davis and Leckie 1978). At pH 8, adsorbed Cu reached 43%, but equilibrium did not attained after one hour of interaction.

Adsorbed Zn showed also some increase between pH 3 and 6 (9-20%) and its adsorption edge was shown at the same pH as Cu. At pH 8, it still shows lower adsorption than Cu (only 35%). This is a typical behaviour for the adsorption of transition metals ions on hydrous oxides which have the same surface properties as natural sediments. It should however be reminded that the pH region of rapid adsorption increase varies with the amount of sediment and total metal concentration (Davis and Leckie 1979; Benjamin and Leckie 1981; Davis 1984).

2. In the presence of humic acid

In the presence of humic acid, all metals showed enhanced reduction in the dissolved portion. The high the humic acid/metal ratio, the higher was the amount of metal disappeared from solution (Fig. 3).

For Cu, Zn, Ni and Cd, adsorption isotherms were almost flat or showed a rela-

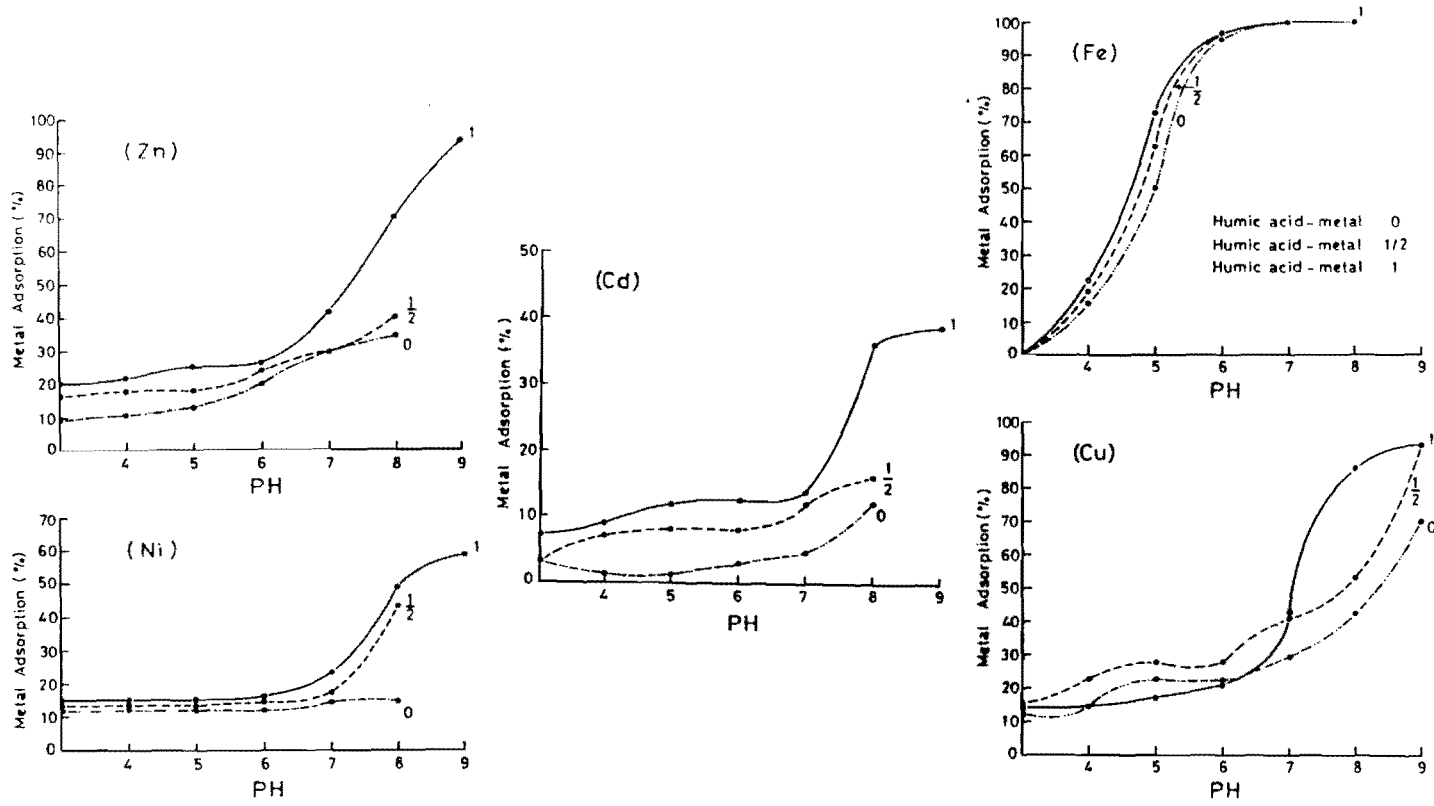


FIG. 3. Influence of pH on the adsorption of trace metals on suspended lake sediments ($< 63 \mu\text{m}$) in the presence of varying proportion of humic acid (— · · · —, no humic acid added; - - - , humic acid to metal: 1/2; — humic acid to metal: 1).

tively little increase (Cu and Zn) in the pH range 3-6 then adsorption increased more or less abruptly to reach, at pH 8 and for a humic acid/metal ratio 1, 86% for Cu, 70% for Zn, 49% for Ni and only 36% for Cd.

At pH 9, the adsorbed Cu and Zn were more than 90%, adsorbed Ni increases to 58% while adsorbed Cd was almost invariable (39%).

In the pH range 3-6, adsorbed Zn, Ni and Cd increased slightly (5-10%) when the HA/metal ratio was increased from zero to one. On the opposite, while the absolute fraction of the adsorbed Cu increases (with pH increase), the adsorbed Cu at zero HA/Cu ratio was still higher than that when the ratio was unity. When the ratio was equal to 1/2, adsorbed Cu was the highest. In the case of Zn, Cd and Ni complexation and/or adsorption then coprecipitation with humic acid may be advanced to explain the relative increase of the adsorbed fraction of these metals in the presence of humic acid (Sholkovitz 1976; Sholkovitz *et al.*, 1978; Sholkovitz and Copland 1981).

The behaviour of Cu is hardly to explain, however, it is probable that Cu-humic acid complexes formed under different proportion of dissolved humic acid may have different adsorptive properties. In this respect, when the HA/Cu ratio was 1/2, the formed complexes seem having higher affinity to the solid surface sites than both ionic Cu and Cu-humic complexes formed when the ratio was unity, this was in the pH range 3-6.

In order to understand the great increase of the adsorbed fraction metals at pH 8 and 9, it was necessary to understand the behaviour of humic acid in the following three systems at these pH values :

- 1 – Humic acid + lake water
- 2 – Humic acid + lake water + sediment
- 3 – Humic acid + lake water + sediment + trace metal.

In the first case, after one hour shaking, 31 and 40% of humic acid were separated by filtration at pH 8 and 9 respectively. In the second system, the quantity of humic acid that was filtered out at pH 8.8 was 63%, which means that approximately 23% of the dissolved HA were adsorbed on particles surface. In the third case, the proportion of humic acid that was filtered out increased significantly and varied according to the metal used in the system. About 97% of the humic acid was filtered out with Cu at pH 8 and 9, while relatively lower quantities were separated in the case of Zn (72 and 95%) and with Cd only 49% and 82% of humic acid was filtered out. This massive precipitation of humic acid coincide with the sudden disappearance of the metals from solution.

It seems therefore that, the massive disappearance of Cu, Zn and Ni from solution at pH 8 and 9 is the presence of the precipitation of the humic complexes of these metals and adsorption of these metals on the freshly formed particulate humic acid which has been proven to be an efficient adsorbing surface (Faguet 1982).

On the other hand, the results indicate that the behaviour of iron is mostly controlled by the solubility of the Fe (III) hydroxides. The proportion which disappears

from solution increases slightly in the presence of humic acids, however, its behaviour seems independent from the behaviour of HA (Fig. 3).

Several authors have shown the parallel disappearance of dissolved iron and humic acid (organic matter) from water during estuaries mixing, suggesting that the exact nature of this associations include complexation, colloidal association or both (Shapiro 1964; Sholkovitz *et al.*, 1978; Moore *et al.*, 1979).

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المواد الهيومية في رواسب بحيرة إدكو - مصر : ٢ - دورها في تراكم العناصر النادرة

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المستخلص . تتميز الأحماض الهيومية الفولفية المستخلصة من رواسب بحيرة إدكو باغتنائها بعناصر النحاس والزنك والنيكل ونسبة أقل بعنصر الكوبلت وذلك مقارنة بالرواسب التي استخلصت منها ، ولكنها فقيرة في كل من الحديد والمنجنيز . وبلغت التركيزات المتوسطة في الأحماض الهيومية لكل من النحاس والنيكل والزنك والكوبلت ٣١٩ ، ٢٠٨ ، ١٨٨ ، ٤٧ ميكرو جرام / جرام على التوالي بينما كانت تركيزاتها في الرواسب ذاتها ٦٥ ، ٨٤ ، ١٢٩ ، ٤٠ ميكرو جرام / جرام . وقد تميزت الأحماض الهيومية بارتفاع تركيزات جميع العناصر بها فيما عدا الزنك وذلك مقارنة بالأحماض الفولفية .

أبرزت التجارب المعملية أن تواجد الأحماض الهيومية في مياه البحيرة يؤدي إلى الإسراع من انتقال عناصر النحاس والكاديوم والنيكل والزنك من المحلول إلى أسطح المواد العالقة من خلال ترسيب معقدات هذه المعادن مع الأحماض الهيومية وكذا من خلال الادمصاص على أسطح حبيبات هذه الأحماض حديثة التكوين . وقد ظهر أن انتقال الحديد من المحلول إلى الرواسب يعتمد على آليات أخرى يحتمل أن تكون أكاسيد الحديد المائية شحيحة الذوبان .