

## Comparison of controlling mechanisms of flocculation processes in estuaries

<sup>1</sup>\*A. Biati; <sup>2</sup>A. R. Karbassi

<sup>1</sup>Department of Environmental Science, Graduate School of the Environment and Energy, Science and Research Branch, Islamic Azad University, Tehran, Iran

<sup>2</sup>Graduate Faculty of the Environment, University of Tehran, P.O.Box 14155-6135, Tehran, Iran

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**ABSTRACT:** During estuarine mixing, dissolved metals come into the particulate phase due to the flocculation processes. Such processes are biologically vital. In the present study, controlling mechanisms of elemental flocculation during estuarine mixing in northern and southern estuaries of Iran in relation to the various physical and chemical parameters of waters have been compared. Except for zinc and lead, for other studied elements in Minab River, water flocculate at higher rates in comparison with the rivers flowing into the Caspian Sea. Redox potential might have negative effect on flocculation process in Minab Estuary. Contrary to rivers flowing into the Caspian Sea, in Minab River elemental flocculation is governed by dissolved organic carbon and it shows a non-linear and conservative behavior during estuarine mixing which implies that dissolved organic carbon originates from terrigenous source. The results also shows that maximum removal of elements occurs in lower salinities (1.5 to 5.8 ‰) for the rivers in North of Iran and 3.3 to 11.4 ‰ for Minab River in South of Iran. Flocculation of studied metal in different rivers results in reduction of overall metal pollution load by various percentages. The initial metal contents on river water and mean discharge of river might lead to higher flocculation rates.

**Key words:** Estuarine processes; Metals; Purification; Removal; River

### INTRODUCTION

An estuary is a semi- enclosed coastal body of water which has a free connection with the open sea and within which sea water is measurably diluted with fresh water derived from land drainage (Pritchard, 1967). It is a very productive biome and supports biogeochemical cycling, maintenance of biological production, movement of nutrients and etc. (Meire, 2005; Samarghandi *et al.*, 2007; Hassani *et al.*, 2008). Information on the variability of the existence forms of microelements in estuarine waters is necessary in order to understand their behavior in passing through the geochemical barrier and their effect on estuarine ecology (Anikiev and Goryachev, 1991; Achterberg *et al.*, 2003; Savchenko, 2009). Flocculation of dissolved metals can significantly affect the chemical mass balance between rivers and seas or lakes. During estuarine mixing, dissolved metals come into the particulate phase due to the flocculation process. Salinity may inhibit the vertical turbulent eddies due to a vertical density gradient and can influence sediment transport through increased flocculation (Eckert and Sholkovitz,

1976; Boyle *et al.*, 1977; Sholkovitz *et al.*, 1977). Some researchers have mainly focused on salinity, pH, colloidal stability, surface properties, humic acids, turbulence and concentration of suspended matters, ionic strength and algal concentration as the main governing factors or flocculation of trace metals during estuarine mixing. (Hunter, 1983; Zhiqing *et al.*, 1987; Shankar and Karbassi 1992; Featherstone and O'Grady, 1997; Matagi *et al.*, 1998). Due to growing concern over the divers effects of heavy metals and their compounds on aquatic ecosystems, many investigations have been carried out on dissolved elemental concentrations and then controlling mechanisms in some rivers flowing in to the Caspian Sea in North of Iran and the Persian Gulf in the South of Iran (Karbassi and Nadjafpour, 1996; Saeedi *et al.*, 2003; Karbassi *et al.*, 2007, Karbassi *et al.*, 2008 a,b; Biati *et al.*, 2010 b). In the present study, controlling mechanisms of elemental flocculation during estuarine mixing in northern and southern estuaries of Iran in relation to the various physical- chemical parameters of water are compared.

✉ \*Corresponding Author Email: [bayati\\_id@yahoo.com](mailto:bayati_id@yahoo.com)  
Tel./Fax: +98912377164



**MATERIALS AND METHODS**

Minab River has a length of 200 Km and its average annual discharge is 10.99 m<sup>3</sup>/s. Minab River is formed by two main branches named Rudan in western part and Jaghin in eastern part. It lies between longitude of 57° 07' and Latitude of 27° 09'. Annual average of sediment load weights of River in Berentin Station has been estimated at 5920 ton/km<sup>3</sup>/y. Various municipal and industrial wastewater containing heavy metals and organics along with agricultural fertilizers, pesticides and herbicides enter into the Minab River. Freshwater samples were collected from the Minab River (ca. 16 Km upstream) in a 25 L clean polyethylene bucket from the surface. On the same day, it was filtered through 0.45 µm Millipore AP and HA filters (Biati *et al.*, 2010b). About one liter of filtered water was acidified with concentrated HNO<sub>3</sub> to a pH of approximately 1.8 and stored in polyethylene bottles in a refrigerator prior to the analysis of dissolved metals. The rest of filtered waters were also kept in refrigerator. On the same day, Persian Gulf water was collected approximately 20 Km away from the coast to ensure that the sample was not diluted by river water. In order to prevent the contamination of samples, all equipment was acid washed with a mixture of HNO<sub>3</sub> and HCl. Rinsing was done with running Milli-Q water. Filtered river water and sea water were mixed together at room temperature in various proportions yielding 9 different proportions of salinity (3.3-29.9 ‰). They were kept for 24 h with occasional stirring. The resulting flocculants were collected on 2.5 Cm diameter Millipore

membrane filters (type HA, Pore size: 0.45 µm). Millipore filters were digested by 5 mL concentrated HNO<sub>3</sub> overnight. The concentrations of Cu, Zn, Ni, Pb and Mn were determined by ICP (JOBINYVON model JY 138 ULTRACE). Procedural blanks and duplicates were run with the samples in a similar way. Calibration ICP was done by dilution of single concentrated standards purchased from SPEX<sub>CerPrep</sub> Company. The accuracy of the analysis was about ±5 % for all elements in the dissolved and flocculants phases. It should be noted that salinity of fresh water and seawater in sampling station was measured using portable apparatus. Of the existing clustering techniques (Lance and William, 1966; Anderson, 1971; Davis, 1973) the Weighted Pair Group (WPG) method (Davis, 1973) was used in this study.

**RESULTS AND DISCUSSION**

At laboratory scale fresh water with its initial trace metal contents is mixed up with saline water while in the nature fresh water does not mix up with saline water as same as laboratory scale. At very first stage of mixing of river water with saline water, some of dissolved metals ooze out of the fresh water in the form of flocculants. Thus, at the later stage of mixing (i.e. higher salinities) fresh water is impoverished in base metals and fewer flocculants form (Table 1). About 117µg/L of total Zn contents flocculates during estuarine mixing (about 5%). The maximum flocculation of Zn occurs at salinities of 3.3 and 29.9 ‰, respectively (37 and 36 µg/L). In other

Table 1: Mean actual concentration of elements in flocculants along with salinity in Minab River (Biati *et al.*, 2010)

Elements	Zn	Pb	Ni	Cu	Mn	S
Aquarium No.	(µg/L)					(‰)
Minab River water	117	50	88	36	259	0.72
1	37 (31.6)*	5 (10)	55 (62.6)	14 (38.9)	85 (32.8)	3.3
2	0 (0)	14 (28)	1 (1.1)	6 (16.7)	48 (18.5)	8.3
3	9 (7.7)	0 (0)	2 (2.3)	6 (16.7)	0 (0)	9.1
4	6 (5.1)	0 (0)	1 (1.1)	0 (0)	42 (16.2)	11.4
5	0 (0)	0 (0)	14 (15.9)	0 (0)	0 (0)	14.5
6	0 (0)	0 (0)	0 (0)	0 (0)	5 (1.9)	18.5
7	0 (0)	0 (0)	0 (0)	2 (5.5)	0 (0)	26.7
8	0 (0)	0 (0)	0 (0)	2 (5.5)	0 (0)	28.8
9	36 (30.80)	0 (0)	0 (0)	0 (0)	0 (0)	29.9
Total	88 (75.2)	19 (38)	73 (82.9)	30 (83.3)	180 (69.4)	-

\* The amounts in parentheses are indicated as percentage of removal

salinities, flocculation of Zn is not significant. Amongst studied metals, only Zn flocculates at the highest salinity (29.9 ‰). About 38 % of total Pb content flocculates during estuarine mixing which is equal to 19 µg/L. Flocculation of Pb occurs at salinity of 3.3 and 8.3 ‰ and this behavior makes it exceptional amongst other studied metals. The highest flocculation of Ni (about 55µg/L) is found at salinity of 3.3 ‰. In salinity of 8.3 to 11.4 ‰, flocculation of Ni ranges from 1 to 2 µg/L and increases in salinity of 14.5 ‰. Flocculation of Ni does not occur in salinities higher than 14.5 ‰. Generally, total flocculation of Ni (similar to Cu) during estuarine mixing is about 83 % (73 µg/L). The highest flocculation of Cu (same as Zn and Ni) occurs at salinity of 3.3 ‰. However, the flocculation is found in salinity of 8.3 and 9.1 ‰ and to a lower extent at 26.7 and 28.8 ‰. About 70 % of initial content of Mn flocculates during estuarine mixing and most of it occurs at salinity of 3.3 ‰ (about 83 %). Flocculation of Mn can be also traced in salinity of 8.3 and 11.4 ‰. The concentration of studied metals in Minab River water during estuarine mixing has been compared with the concentration of elements in flocculants of five rivers flowing into the Caspian Sea in North of Iran (Table 2). As can be seen in Table 2, except for Zn and Pb other studied elements in Minab River water flocculate at higher rates in comparison with the river flowing into the Caspian Sea (Karbassi and Nadjafpour, 1996; Saeedi et al., 2003; Karbassi et al., 2007; Karbassi et al., 2008a,b) and this might be due to higher initial metal contents in Minab River water than the northern river of Iran (Biati et al., 2010 b). The lowest flocculation rate in Minab river is associated with Pb (38 %) and in Tadjan, Talar, Babolrud and Haraz (Rivers flowing into the Caspian Sea) Ni flocculates in the lowest rate (7.1 %, 18.8 %, 12.2 % and 11.3 %, respectively). In

Shirud River Zn shows the lowest flocculation rate which is in contrast with the other studied rivers in the North. An almost conservative behavior of Ni in Haraz, Babolrud and Talar estuaries is incompatible with the findings of nearby estuary (Tadjan Estuary). Copper has highest flocculation rate amongst studied metal only in Minab, Babolrud and Shirud Rivers but in the other rivers maximum flocculation rate can be found for Zn. The highest flocculation rate of Zn occurs in Tadjan river (96.6 %). Moreover, the widest range of flocculation rate is found in Tadjan River (from 7 % to 97 %). General pattern of flocculation rate of the seven mentioned rivers is as follows:

Minab: Cu (83.3 %) > Ni (82.9 %) > Zn (75.2 %) > Mn (69.4 %) > Pb (38 %)

Tadjan: Zn (96.6 %) > Mn (51.7 %) > Cu (41.7 %) > Ni (7.1 %)

Talar: Zn (72.5 %) > Mn (52.3 %) > Cu (48 %) > Pb (27.5 %) > Ni (18.8 %)

Babolrud: Cu (77.9 %) > Zn (74.6 %) > Mn (64.97 %) > Pb (38.2 %) > Ni (12.2 %)

Haraz: Zn (85.5 %) > Mn (55.2 %) > Cu (39.6 %) > Pb (33.7 %) > Ni (11.3 %)

Shirud: Cu (74 %) > Pb (61 %) > Mn (58 %) > Zn (34 %)

Range of salinities in obtained series of mixtures resulting from mixing of river water with the saline water in different proportions in Minab River varies from 3.3 to 29.9 ‰ and in the rivers flowing into the Caspian Sea ranges from about 1.5 to approximately 9.5 ‰ (Table 2). Thus, it can be inferred from data given in Table 2 that rapid flocculation occurs in the earlier stages of mixing of fresh water with the saline water (salinity of 3.3 to 11.4 for Minab River, salinity of 1.5 to 5.5 for Tadjan, Talar, Babolrud and Haraz Rivers and salinity of 3.2 to 5.8 for Shirud River). This is in accordance with the findings of other researchers (Duinker and Nolting, 1978; Bewers et

Table 2: Concentration of elements in flocculants and effective range of salinity in six rivers

River	Elements Concentration of elements in flocculants (µg/L)					Range of salinity in series of mixtures (‰)	Most important salinity ranges flocculation of metals (‰)	Reference
	Zn	Pb	Cu	Mn	Ni			
Minab	88 (75.2)*	19 (38)	30 (83.3)	180 (69.4)	73 (82.9)	3.3-29.9	3.3-11.4	Biati et al. (2010a)
Tadjan	57 (96.9)	ND*	4.7 (41.7)	12.1 (51.7)	2.8 (7.1)	1.52-9.52	1.5-5.5	Saeedi et al. (2003)
Talar	62.4 (72.5)	11.6 (27.5)	14.9 (48)	15.6 (52.3)	4.7 (18.8)	1.50-9.50	1.5-3.5	Karbassi et al. (2007)
Babolrud	63.4 (74.6)	8.42 (38.2)	16.2 (77.9)	12.8 (64.97)	4.90 (12.2)	1.55-9.55	1.5-3.5	Karbassi et al. (2008a)
Haraz	81.29 (85.5)	8.42 (33.7)	14.25 (39.6)	13.8 (55.2)	5.07 (11.3)	1.50-9.50	1.5-4.5	Karbassi et al. (2008b)
Shirud	27.5 (34)	15.8 (61)	20.0 (74)	9.2 (58)	-	3.2-7.4	3.2-5.8	Karbassi and Nadjafpour (1996)

\* Not Detected; \*\* The amounts in parentheses are as indicated percentage of removal



al., 1974); though at relatively higher salinities. Higher removal of heavy metals at greater salinities is reported by Biati *et al* (2010a).

Biati *et al.* (2010b) showed that pH, temperature, DO, alkalinity,  $\text{HCO}_3^-$ ,  $\text{CO}_3^{2-}$ , EC, salinity, Na, Cl, Mg,  $\text{SO}_4^{2-}$  and Ca do not have any role on the flocculation process during mixing of the Minab River water with the Persian Gulf waters. Redox potential might have reverse effect in flocculation process in Minab estuary. Amongst all studied physical-chemical parameters only dissolved organic carbon (DOC) showed high similarity coefficient with Cu, Zn, Ni and Mn. Though, DOC has a little influence on the flocculation of Pb. Therefore, DOC is the main governing factor for the flocculation of Zn, Ni, Cu and Mn in the Minab estuary (Table 3). This finding is in agreement with studies of Sholkovitz (1976), Mantoura and Woodward (1983) and Meyer (1983). In contrast, in studies of rivers flowing into the Caspian Sea DOC does not play an important role in flocculation process. Karbassi *et al.* (2007) and Karbassi *et al.* (2008a) reported that flocculation of Zn, Ni and Cu is controlled by pH and total nitrogen ( $\text{N}_T$ ) during mixing of Talar and Haraz rivers with Caspian Sea water. Statistical analysis reveals that the flocculation of dissolved metals in Tadjan river which is governed by pH has an inverse relationship with TOC, EC and salinity. This suggests that increasing salinity, TOC and EC leads to a decrease of pH and elemental flocculation (Saeedi *et al.*, 2003). Karbassi and Nadjafpour (1996) concluded that in Shirud Estuary, the main governing factor of metal flocculation (Pb and Zn) is salinity. Biati *et al* (2010a) stated that the removal of dissolved metals (Zn, Mn) from waste waters is not influenced by DOC. Electrical conductivity, pH, salinity and Cl show significant role in flocculation of Zn and Mn from wastewaters. Removal of Ni and Pb from wastewaters is controlled by DO, DOC and Eh. Redox potential is of major concern for mobilization of trace metals in estuaries where an oxygen gradient exists in addition to the salinity gradient (Gerringa *et al.*, 2001). Besides, this studies showed that similar to the findings of Biati *et al* (2010b) mechanisms for flocculation of Pb is still unknown and further investigations is needed to find out governing factors in the flocculation of Pb. Moreover, Karbassi *et al* (2008b) stated that pH and  $\text{PO}_4$  control flocculation of Cu in Babolrud estuarine process.

Since salinity is a general term that does not necessarily show the effect of other constituents of the saline water on elemental flocculation in estuaries, other constituents of Sea water might be involved in the flocculation processes. Many studies show that salinity

and pH are the main factors in the flocculation of metals during estuarine mixing (Boyle *et al.*, 1977; Duinker *et al.*, 1983; Hunter, 1983; Zhiqing *et al.*, 1987). Contrary to the findings of Biati *et al* (2010b) that DOC showed a non-linear and conservative behavior during mixing of Minab river water with the Persian Gulf water; conservative DOC behavior is reported for estuarine mixing in Beaulieu estuary (Moore, *et al.*, 1979). Karbassi *et al.* (2008b) reported linear DOC increase for the rivers flowing in to the Caspian Sea. In contrast, a linear DOC decrease over salinity range of 17 to 28 ‰ is reported for Bristol channel (Mantoura and Mann, 1979). Although studies of rivers flowing in to the Caspian Sea indicate that DOC increase with an increases in salinity that could be indicative of non- terrigenous DOC (Karbassi *et al.*, 2007; Karbassi *et al.*, 2008 a,b), study of flocculation process in Minab River in South of Iran showed that DOC originates from terrigenous source and Persian Gulf waters do not contribute to DOC contents of Minab estuary (Biati *et al.*, 2010b). Removal of significant amounts of riverine DOC may occur due to flocculation or precipitation reactions that result in the conversion of DOC to particulate organic carbon and their subsequent settlement on to the sediments (Sholkovitz, 1976). Production of autochthonous DOC in estuaries may also occurs through several processes (Sharp, 1991). Aquatic fulvic acids accounts for 50 to 80 % of the total amount of DOC in coastal waters (Krachler, *et al.*, 2005). The reasons for such increase in DOC concentrations from rivers to the Caspian Sea in North of Iran, which may be related to exudation by micro-algae require further studies.

Table 3 summarizes the governing factors that control flocculation of trace metals during estuarine mixing for Minab, Tadjan, Talar, Babolrud, Haraz and Shirud Rivers. As can be seen in Table 3,  $\text{PO}_4$  is one of the factors that affects flocculation of elements (e.g. Cu) in Babolrud estuarine mixing. It should be pointed out that the processes responsible for removal of dissolved  $\text{PO}_4$  at high concentrations encountered in the Babolrud estuary include precipitation of apatite (calcium phosphate), viviane (ferrous phosphate) and magnesium- ammonium phosphate but these have not been indentified in situ or in laboratory experiments simulating estuarine conditions (Deck, 1981; Saeedi *et al.*, 2003). Considering the mean discharge of the rivers (Table 3) concentrations of dissolved metals in river water and also percentage of elemental removal via estuarine mixing (Table 2), amount of elemental load entering to the Sea and removal of dissolved metals due

Table 3: Controlling parameters of flocculation along with amount of elemental removal via flocculation in six rivers

River	Controlling parameters of flocculation						EC	Removal via flocculation (Ton/y)	Dissolved elemental load entering to the sea (Ton/y)	Mean discharge of river (m <sup>3</sup> /y)
	pH	Eh	S (‰)	DOC	PO <sub>4</sub>	Total nitrogen (N <sub>T</sub> )				
Minab	○	-	○	+	×	×	○	135.2	55.7	347×10 <sup>6</sup>
Tadjan	+	×	-	×	×	×	-	37.4	10.727	246×10 <sup>6</sup>
Talar	+	×	○	○	×	+	○	54.1	49.63	440×10 <sup>6</sup>
Babolrud	+	×	○	○	+	○	○	27.3	45.9	560×10 <sup>6</sup>
Haraz	+	×	○	○	○	+	○	122.6	97.02	940×10 <sup>6</sup>
Shirud	×	×	+	×	×	×	×	15.9	10.6	127×10 <sup>6</sup>

+: Governs; -: Inhibits; ○: No effect; ×: No information

to flocculation process are estimated. The highest elemental removal during estuarine mixing can be found in Minab River where approximately 135 ton/y of dissolved metals are flocculated and the lowest removal via flocculation processes occurs in Babolrud River (27.3 ton/y). Therefore, it can be concluded that higher initial concentration of elements in river water and mean discharge of river may lead to higher flocculation rates. Removal of dissolved elements in the 6 mentioned river is in the following order:

Minab (135.2 ton/y) > Haraz (122.6 ton/y) > Talar (54.1 ton/y) > Tadjan (41.5 ton/y) > Babolrud (27.3 ton/y) > Shirud (10.91 ton/y).

The highest and the lowest dissolved elemental load flow to the Caspian Sea via Haraz river (97.02 ton/y) and Shirud (10.6 ton/y), respectively. General pattern for dissolved loads of elements flowing into the Caspian Sea and Persian Gulf is as follow: Haraz (97.02 ton/y) > Minab (55.7 ton/y) > Talar (49.63 ton/y) > Babolrud (45.9 ton/y) > Tadjan (10.73 ton/y) > Shirud (8.57 ton/y).

## CONCLUSION

Flocculation process of dissolved Zn, Cu, Mn, Ni and Pb during estuarine mixing of Minab River flowing in to the Persian Gulf and five rivers flowing in to the Caspian Sea have been compared with one another. The results showed that maximum removal of elements occurs in lower salinities (1.5 to 5.8 ‰) for the rivers in North of Iran and 3.3 to 11.4 ‰ for Minab River in South of Iran. The main governing factor that plays significant role in flocculation of dissolved metals in Minab River estuarine zone is DOC (except for Pb) but in the other rivers flocculation is mainly controlled by pH and total nitrogen (N<sub>T</sub>) and also PO<sub>4</sub> in Babolrud and salinity in Shirud estuaries. Other physical- chemical parameters of water does not play important role in flocculation process. Contrary to the northern coastal waters of Iran source of DOC in Minab River (in southern part of Iran) is

terrigenous. Further investigation is suggested on more and detailed role of other constituents of seawater in flocculation of trace metals during estuarine mixing. Flocculation of studied metal in different rivers shows that the overall metal pollution load can be reduced by various percentages. The initial metal contents on river water and mean discharge of river might lead to higher flocculation rates.

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**AUTHOR (S) BIOSKETCHES**

**Biati, A.**, Ph.D. Scholar, Department of Environmental Science, Graduate School of the Environment and Energy, Science and Research Branch, Islamic Azad University, Tehran, Iran. Email: [biati\\_id@yahoo.com](mailto:biati_id@yahoo.com)

**Karbassi, A. R.**, Ph.D., Assistant Professor, Department of Environmental Engineering, University of Tehran, Tehran, Iran. Email: [akarbassi@ut.ac.ir](mailto:akarbassi@ut.ac.ir)

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