Determination of Cr, Pb and Ni in water, sludge and plants from settling ponds of a sewage treatment works

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Abstract

Wastewater from a sewage treatment works is channelled through a series of inter-connected settling ponds into a stream. Furthermore, leachates from a domestic and industrial landfill site are disposed into the first pond. From the variety of plants growing in the ponds, *A. sessilis, P. stratiotes, R. steudelii* and *T. capensis* were investigated for their ability to uptake chromium, lead and nickel (these metals are toxic to humans while nickel is also involved in plant growth). The levels of the metals in the water, plants and sludge were determined using an inductively coupled plasma-mass spectrometer (ICP-MS). For the plants, the amounts of the metals in roots, stems and leaves were also measured. In general it was found that the plants accumulated up to 15% of the level of metals in the water and that accumulation depends on the plant species as well as on the organ of the plant. The concentrations of metals in the water in the last pond were found to be well within the limits set by the South African National Water Act of 1998 for discharge of water into rivers. Furthermore, the results of this study, which involved a real system, were compared with those from model studies where conditions of the system could be controlled by the investigator.

Keywords: uptake, heavy metals, plants, wastewater treatment

Introduction

Water is regarded, internationally, as the most fundamental and indispensable of all natural resources (Ashton and Seetal, 2002). The management of water resources is a big challenge for most countries of the world. In Africa, the need to recycle wastewater is increasing due to the dire shortage of freshwater. Large-scale wastewater treatment constitutes a very important part of the management of water resources. In view of this, the reuse of treated water from a sewage treatment plant can make a significant contribution to the water resources of industrialised cities. Many industries do, and will, use recycled water provided that such water will not cause short- or long-term damage to their machinery due to the presence of heavy metals.

Physical and chemical treatment (Chan et al., 2006) of wastewater may involve one or more of the following: ion exchange (Taylor, 2006), reverse osmosis (Qdais, 2004), electrolysis (Fischer et al., 2005), precipitation (Banfalvi, 2006) and reduction (Chang, 2005). All of these processes are relatively expensive. Thus cheaper alternatives which are suitable for large-scale application need to be established. Green plants have been identified as miners of the earth. They are the major accumulators of inorganic nutrients upon which other life forms are directly or indirectly dependent (Baker, 1983). Plants known as 'hyperaccumulators' (Zhang et al., 2002; Odjegba and Fasidi, 2004) have been shown to accumulate hundred or thousand times more metals than 'normal' plants.

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Uptake of metals by terrestrial plants has been studied by several workers some examples being Ghaderian et al. (2007); Wu et al. (2007), Dahmani-Muller et al. (2000) and Van Aardt and Erdmann (2004). The various categories of water plants useful in phytoremediation have been identified by, among others, Outridge and Noeller (1991). Studies on aquatic plants involving wetlands and paddy-fields were done by researchers such as Gaur et al. (1994); Fazeli et al. (1998) and Almela et al. (2002). Investigations on aquatic plants in streams, rivers, water and reservoirs were carried out by e.g. Del Rio et al. (2002); Valitutto et al. (2007); Szsymermanowska et al. (1999); Kassim et al. (1997); Hozhina et al. (2001); Samecka-Cymerman et al. (2002); Arribere et al. (2003) and Cardwell et al. (2002). Work on plants found in saline water has been described in the literature by Quan et al. (2007); Macfarlane and Burchett (2002); Nienhuis (1986); Carter and Ericksen (1992); Catsiki and Panayotidis (1993); Romeo et al. (1995); and Warnau et al. (1995).

In the light of the literature search described above, it was found that very little work has been done on plants in settling ponds serving a sewage treatment facility. In view of this, our study focused on the uptake of heavy metals by aquatic plants growing in the settling ponds of a sewage works. Furthermore this project was motivated by the need to:

- Compare the bioaccumulation of Cr, Pb and Ni by a given plant in the settling ponds (a real system) with that obtained in model systems using a tank
- Determine the distribution of the selected metals among water, plants and sludge
- Ascertain the distribution of the selected metals within the organs of the plants.
- Determine the concentrations of the selected metals in the water being disposed into the Umhlangane River (see Fig. 1).

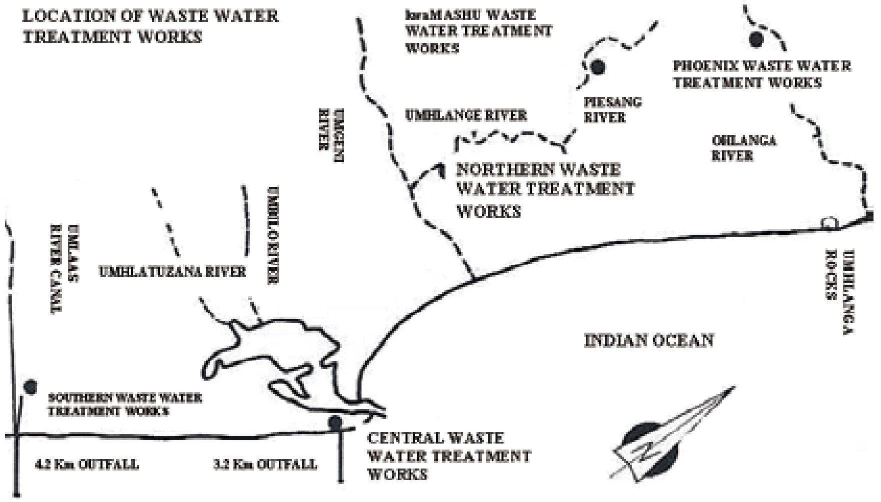

Figure 1
Sketch showing location of the North Durban Treatment Works

Study area

Durban Municipality serves a population of 2 to 2.5 m. people and controls 28 wastewater treatment works, which receive industrial as well as domestic wastewater. Treatment works located in the vicinity of the ocean, subject their waste to 'primary treatment' and then dispose the waste directly into the sea through outfalls. Inland treatment works use 'full treatment' resulting in the separation of solids and liquid (water). While the solid component is sent to landfill sites the water receives further treatment in a series of linked ponds. Our samples were taken from the five inter-linked ponds of the Northern Wastewater Works, the largest wastewater works controlled by Durban Municipality. It receives approximately 50 x 10^6 ℓ of wastewater per day, (about 20% from industries and about 80% from homes). There is also an input of leachate from the nearby (Bisasar Road) landfill site. This landfill site accepts both industrial and domestic solid waste. It was expected that the leachate would contain heavy metals.

Experimental/method

General

Only A Grade glassware was used throughout this study. All glassware was soaked in 10% nitric and washed with milli-Q water prior to use. Nitric acid (70% – Reidel de Haen), perchloric acid (70% – ACE) and hydrochloric acid (32% – BDH) were used as received. Standard solutions (1 000 mg·$ℓ^{-1}$) of Cr, Ni and Pb were purchased from Saarchem and diluted as required with 1 N nitric acid. The certified reference material (SARM No. 51) for sludge was purchased from Industrial and Analytical (Pty) Ltd. Dried plant material was milled using grinder type DCFH 48 supplied by Janke and Kunkel GMBH & Co. Dried sludge was ground in an agate mortar and pestle. Ground materials were passed through a 500 µm sieve to remove larger particles.

Sampling and sample pretreatment

Sampling was done between 2001 and 2002, with preliminary studies taking place in the latter part of 1999. Three compartments of the site were sampled. These were the dredged sediment (hereafter called 'old sludge'), sediment (hereafter referred to as 'new sludge') from the 5 ponds and the water from the 5 ponds. The plants *Pistia stratiotes* and *Alternanthera sessilis* were collected from each of the 5 ponds. *Typha capensis*, *Rumex steudelii* and *Alternanthera sessilis* were sampled from the old sludge. Water samples were stored in a fridge at approximately 4°C. Sludge samples were oven-dried at 60°C for 2 d, then crushed and sieved. Following sampling, the plants were rinsed with deionised water and separated. *A. sessilis* leaves were separated from the stems. *T. capensis* was separated into above- and below-ground components. *R. steudelii* was separated into leaves, roots and stems, and *P. stratiotes* was kept whole. The different portions of plants were placed in brown bags and dried (in an oven) at 60°C for 2 d and milled using the grinder described above. Digestion of plant and sludge samples was effected using modifications of literature methods as exemplified by Rodushkin et al. (1999) and Tinggi et al. (1997).

Sample preparation

Digestion of the plant material and the sludge was done using acids and thermal conditions (heated sand bath). To a 1 g sample (in triplicate) in a 100 mℓ beaker was added concentrated nitric acid (15 mℓ) and left standing at room temperature for 12 h. This was then heated at 150°C for 1 h. After cooling, the samples were treated with concentrated perchloric acid (10 mℓ) and digested at 235°C for 2 h. After cooling, concentrated hydrochloric acid (5 mℓ) was added. This was followed by digestion at 150°C for 20 min. The samples were cooled and transferred to a volumetric flask. The volume was made up to the mark using milli-Q water. These were then analysed using ICP-MS. All the water samples were prepared for analysis by treating a 95.0 mℓ of water with concentrated nitric acid (5.0 mℓ) and filtered through a 0.45 µm filter.

ICP-MS was used to perform rapid and simultaneous analysis of most metals. However, preliminary studies involved the use of both flame AAS and GF-AAS. The choice of this instrument was influenced by the low detection limits of ICP-MS, its high throughput rate and its multi-element analysis capability. The ICP-MS instrument was calibrated using standard solutions. A certified reference material (CRM) for stream sediment was also analysed. Recoveries of 95 to 102% showed that the method was reliable. The analysis data were subjected to statistical treatment using the Microsoft Excel 2000 package. At a confidence level of 95%, $p < 0.05$ was taken to indicate that there was a significant difference between the values being compared.

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Results and discussion

The metals chosen for this study were, nickel (Ni), chromium (Cr) and lead (Pb). Of these metals, Ni is essential, in trace concentrations, for plant growth. The non-essential metals, Cr and Pb were chosen on account of their notoriety in causing cancer and disruption of the central nervous system respectively, in humans.

The order of discussion is concentrations of metals in water, sludge and individual plants, distribution of metals in *A. sessilis* and comparisons.

Water

The concentrations of Pb, Ni and Cr in water are shown in Table 1. In general, it is observed that Pond 1 has a relatively low level of metal concentration, which increases at Pond 2 then exhibits a gradual decrease to Pond 5. The lower values in Pond 1 are rationalised on the basis that this pond contains a high amount of organic matter which binds the metals. At Pond 2, breakdown of organic matter would be expected to release metals into the water and therefore a higher concentration is observed.

Sludge

Sludge samples from the ponds were also analysed for metals and the results are shown in Table 2. The metal concentrations in the sludge are consistent with the expectation of a gradual decrease in metal concentration from Pond 1 to Pond 5. The results for Ni are however out of line with the expected trend. It is noted that values for Ni have been higher than expected throughout this study. A comparison of metal concentration in both compartments (water and sludge) indicates that low levels of metal in the water correspond with high levels in the sludge.

Plants

A. sessilis

A. sessilis is a free-floating plant with fine roots. It can also grow rooted. In an aquatic environment, it draws its nutrients from the water. This plant was collected from both water and the 'old sludge'. For analysis purposes roots and stems were combined while leaves were analysed separately. The results are given in Table 3.

The results (Table 4) show that there are higher levels of metals in the leaves of *A. sessilis* than in the roots and stems. Almela et al. (2002), found that *Amaranthus blitoides,* which is in the same family as *A. sessilis*, is a very good candidate for bioremediation.

P. stratiotes

This is a free-floating, aquatic plant with fine roots. Specimens were collected only from the ponds. Other studies suggest that this plant is effective for removal of dilute concentrations of heavy metals (Klumpp et al., 2002). Studies done by Ingole and Bhole (2003) suggest that the efficiency for the plant to uptake metals is reduced at high concentrations (generally above 10 mg·ℓ$^{-1}$). This could be due to stress induced in the plant. In the studies presented here, metal levels were very low in water. It is noted that due to the very small size of these plants it was not feasible to separate the plants into leaves, stems and roots. Thus the results in Table 5 are for the entire plant.

TABLE 1
Heavy metal concentration in water

Sample source	Metal concentration in mg/kg dry weight		
	Pb	Ni	Cr
Pond 1	0.006±0.0001	0.003±0.0001	0.003±0.0001
Pond 2	0.016±0.0001	0.005±0.0001	0.004±0.0001
Pond 3	0.012±0.0002	0.005±0.0001	0.004± 0.0001
Pond 4	0.005±0.0001	0.002±0.0000	0.002± 0.0000
Pond 5	0.005±0.0001	0.005±0.0001	0.002±0.0001

TABLE 2
Heavy metal concentration in sludge

Sample source	Metal concentration in mg/kg dry weight		
	Pb	Ni	Cr
Pond 1	86.0±2.58	60.3±1.81	132±2.0
Pond 2	43.5±0.44	148±1.5	115±1.2
Pond 3	44.5±1.34	216±6.5	109±2.7
Pond 4	43.0±0.86	130±2.6	79.5±1.59
Pond 5	65.0±1.30	118±2.4	83.5±1.67
Old sludge	72.3±0.72	108±1.1	202±2.0
New sludge	112±1.1	142±1.4	395±4.0

TABLE 3
Metal concentration in roots of *A. sessilis*

Sample source	Metal concentration in mg/kg dry weight		
	Pb	Ni	Cr
Pond 1	1.67±0.042	7.00±0.175	2.67±0.067
Pond 2	0.600±0.006	5.00±0.050	2.00±0.020
Pond 3	2.00±0.030	6.00±0.0.090	1.67±0.025
Pond 4	0.750±0.0.015	10.0±0.200	2.00±0.040
Pond 5	0.850±0.0.017	3.00±0.060	0.500±0.010
Old sludge	0.800±0.0.008	4.00±0.040	0.600±0.006

TABLE 4
Metal concentration in stem and leaves of *A. sessilis*

Sample source	Metal concentration in mg/kg dry weight		
	Pb	Ni	Cr
Pond 1	11.5±0.23	6.50±0.130	2.00±0.040
Pond 2	5.00±0.050	9.33±0.093	6.67±0.067
Pond 3	4.67±0.070	4.67±0.070	2.33±0.035
Pond 4	2.67±0.053	8.00±0.160	2.50±0.050
Pond 5	9.00±0.180	7.33±0.147	3.50±0.070
Old sludge	3.67±0.037	7.00±0.070	5.00±0.050

TABLE 5
Levels of metal in *P. stratiotes*

Sample source	Metal concentration in mg/kg dry weight		
	Pb	Ni	Cr
Pond 1	8.50±0.170	24.3±0.49	10.0±0.20
Pond 2	4.33±0.043	16.3±0.16	6.00±0.060
Pond 3	4.33±0.065	14.7±0.22	3.00±0.045
Pond 4	3.00±0.060	22.7±0.45	3.00±0.060
Pond 5	4.00±0.080	17.3±0.35	3.00±0.060

TABLE 6
Levels of metals in *R. steudelii* and in *T. capensis*

Plant name	Plant organ	Metal concentration in mg/kg (dry weight)		
		Pb	Ni	Cr
R. steudelli	Leaves	1.1±0.022	6.0±0.120	4.0±0.080
	Stems	0.80±0.008	4.0±0.040	1.5±0.015
	Roots	3.0±0.045	6.0±0.090	4.0±0.060
T. capensis	Stems and leaves	0.50±0.010	3.0±0.060	1.3±0.026
	Roots	0.40±0.008	6.0±0.150	0.60±0.015

TABLE 7
Distribution of Pb, Ni and Cr in *A. sessilis*

Sample source	Concentration of metals in mg/kg (dry weight)					
	Pb		Ni		Cr	
	Stems & roots	Leaves	Stems & roots	Leaves	Stems & roots	Leaves
Pond 1	1.7±0.034	12±0.180	7.0±0.105	7.0±0.140	3.0±0.075	2.0±0.020
Pond 2	0.60±0.012	5.0±0.075	5.0±0.060	9.0±0.180	2.0±0.050	7.0±0.070
Pond 3	2.0±0.046	5.0±0.080	6.0±0.072	5.0±0.090	2.0±0.054	2.0±0.018
Pond 4	0.80±0.019	3.0±0.051	10±0.130	8.0±0.140	2.0±0.054	3.0±0.033
Pond 5	0.90±0.022	9.0±0.153	3.0±0.039	7.0±0.123	0.50±0.014	4.0±0.044
Sludge	0.80±0.017	4.0±0.068	4.0±0.052	7.0±0.091	0.60±0.016	5.0±0.056

R. steudelii

This is a rooted plant with fine roots. Specimens were collected only from the 'old sludge'. The metal concentrations in the plant are low (Table 6), implying that little or no accumulation had taken place.

T. capensis

This is an emergent rooted plant with a well-developed rhizome. It was only sampled from the 'old sludge' due to its availability in that area. The results presented here (Table 6) are consistent with another study, which reported that *Agrostis tenuis* accumulates higher levels of metals in the leaves rather than in the roots (Dahmani-Muller et al., 2000).

Distribution of metals in *A. sessilis*

With regard to distribution of the three metals in different parts of the plants it is noted that this was done for *Alternanthera sessilis* only. As can be seen from Table 7, for Pb and Cr, the concentrations are higher in the leaves than in the roots and stems combined. For Ni, the concentration is higher in leaves for plants taken from Ponds 3 and 4 whereas it is higher in the stems for plants from other sampling locations.

The relatively higher concentration of metals in the plants than in the water suggests that the plants investigated in this study have a capacity to accumulate metals from water. The general trend observed is that the concentration of Ni in the plants is consistently higher than the other metals. As stated earlier this may be attributed to the fact that plants require Ni for physiological needs while the other metals are not necessary for this purpose. This is also supported by studies done by Catsiki et al. (1993) on *Posidonia oceanica*, who found that Ni levels in plants were about seven times higher than other metals.

The question of whether these aquatic plants are accumulators of metals could be answered by considering the levels of metals in water and the levels in the plants. However, it may be useful to first examine how accumulation has been defined. In the studies conducted by Anderson and co workers (1999), hyper-accumulation is expressed in terms of mg of metal per kilogram of dry leaf material. Garbisu and Alkorta (2001) express the degree of hyper-accumulation as a percentage of the dry weight of the material. Essentially these two consider only the levels of metals in the plant leaf. In studies done by Zhang et al. (2002), hyper-accumulation is expressed in terms of the bio-concentration factor (BCF), which is calculated as the ratio of metal concentration in plant biomass to metal concentration in the water/soil. In the present study, *A. sessilis* and *P. stratiotes* can both be classified as accumulators but not as hyper-accumulators of metals from water. This is also in contrast with the more recent findings of Odjegba and Fasidi (2004) and Gratao and co-workers (2005) who used a wide range of heavy metals and came to the conclusion that P. *stratiotes* is a 'natural hyper-accumulator' with potential for water phytoremediation. However, it should be noted that Odjegba and Fasidi (2004) used hydroponic conditions whereas the plants in our study where collected from 'maturation ponds' of a sewage treatment works. Nevertheless the floating nature of *P. stratiotes* means that they can be easily removed. This property coupled to its fast growth rate makes *P. stratiotes* a suitable aquatic plant for phytoremediation of wastewater in sewage treatment works.

The levels of metals in the plants from the 'old sludge' were found to be very low. It is plausible that either the metals had been leached out by rain-water or that the metals were not present in an 'available' form to the plants.

Conclusions

Overall the results from this study suggest that both *A. sessilis* and *P. stratiotes* may be used to monitor metal levels in wastewater and for phytoremediation, where there is a low concentration of metals. However, this cannot be taken to mean that there is a direct correlation between metal levels in water and metal levels in plants since algae and micro-organisms present in the ponds could break down, convert or absorb metals and therefore compete with green plants.

The concentrations of Cr, Pb and Ni (0.002-0.005 $mg \cdot \ell^{-1}$) in the final effluent being discharged into the Umhlangane

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River (Fig. 1) are well below the limit of 5 mg·ℓ$^{-1}$ set by the South African Water Act No 36 of 1998.

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