Contaminants in Sediments across the Mooi and Vaal Rivers Network in The Vicinity of Potchefstroom

Elvis Fosso-Kankeu, Alusani Manyatshe, Divan van der Berg, Nico Lemmer, Frans Waanders, and Tutu Hlanganani.

Abstract**—**The sequential extraction of heavy metals from the sediments samples based on a four-stage sequential extraction procedure proposed by the European Communities Bureau of Reference (BCR's), was used to assess the sediment contamination in the Mooi and Vaal Rivers network in the vicinity of Potchefstroom. Four fractions of sequential extraction were divided as follows: (i) Acetic acid (exchangeable and specifically absorbed metals), (ii) a reducing agent (bound to Fe/Mn hydroxides), (iii) an oxidizing agent (bind to sediment organic matter and sulphides) and (iv) aqua-regia (bound to mineral structure). The results of the sequential extraction showed that the highest percentages of Fe, Cr and Mg were associated with the residual fraction of the sediment samples from both the Mooi River (83.46%, 27.43% , 88.83%, respectively) and Vaal River (35.21%, 14.1%, 23.93%, respectively), demonstrating that most of this elements were strongly bound to the sediments. However, Ca and Mn were mostly present in the form of exchangeable fraction in the sediment samples from both the Mooi River (72.92% and 33.84%, respectively) and Vaal River (39.53% and 26.06%, respectively), indicating relatively high mobility and bioavailability, meaning that these elements were likely to increase the hardness and toxicity in surface water, respectively. The sediments being composed of oxide minerals were likely to release the metals into the river overtime and therefore affect the biological equilibrium of the aquatic system.

Keywords**—** Mooi and Vaal Rivers, sediments, metals, contamination, recovery, sequential extraction, fractions, mobility and bioavailability.

I. INTRODUCTION

THE effluents of wastewater which flows from the anthropogenic sources such as mining activities, industries, agricultural land and decomposition of organic matter increase the flux of heavy metals in surface water [1; 2; 3]. These sources, more especially mines produce toxic metals which may pose a potential health risk to people living in the vicinity of contaminated area [4]. Mining activities generate a large quantity of tailings and waste rocks, which are deposited in the environment and eventually constitute the source of heavy metals in surface water [5]. When these heavy metals are discharged into the aquatic environment, they are divided between the sediments and water column phases [6]. The contamination of sediments by heavy metals is a serious problem, especially in countries dominated by industries, because of their persistence and non-biodegradability in the environment [7]. Sediments are regarded as an ultimate sink of toxic heavy metals in the aquatic environment [8]. Some of this heavy metals like Ni, Zn and Mn are important for organisms when available in traces while other metals such as Pd and Cd can be extremely detrimental to living organism and human health even at low concentration [9]. The heavy metals in sediments have been widely determined by using the speciation technique for the past two decades [10]. There are many methods for the extraction of heavy metals from sediments which have been studied [11]. In this case, the Mooi and Vaal rivers network situated in the North-West province, South Africa, are studied to evaluate the mobility and bioavailability of trace elements in their sediments. The analysis of total heavy metals contents in sediments does not accurately represent their mobility, bioavailability and toxicity [12], as their toxicity depends especially on chemical form [13]. The speciation of heavy metals can assist in evaluating how strongly they can be retained and how easily they can be released into surface water [14]. The mobility and bioavailability of heavy metals is strongly influenced by their chemical and mineralogical composition in which they occur [15]. The objective of this study was: (i) to evaluate the contamination of sediments along the Mooi and Vaal rivers network in the vicinity of Potchefstroom by using a four-stage sequential extraction procedure proposed by the European Communities Bureau of References (BCR), (ii) to determine the potential mobility and bioavailability of heavy metals in the sediments , (iii) to determine the relationship between the total organic carbon (TOC) in sediments using the Wakley Black titration procedure and (iv) to determine the mineralogical composition and the major components of metals in sediments samples using the XRD and XRF analyses, respectively.

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II. MATERIALS AND METHODS

2.1. Sampling and preparation of sediments samples.

Sediment samples were collected at 10 sampling points along the Mooi and Vaal rivers network in the vicinity of Potchefstroom to investigate their potential mobility and bioavailability in aquatic environment. Most of the areas where these sediments were collected are affected by pollution from anthropogenic sources such as effluents from mining, agricultural and industrial activities. The sediment samples were collected during July 2015, it was a dry season and the water level was low, which make it suitable for sampling. The sediment samples were collected in clean plastic bags and stored at 4ºC prior to analysis. A large amount of sediment samples were collected (about 3 to 4 kg) in order to have enough samples to analyse for sequential extraction, mineralogical and chemical compositions, and organic carbon. In the laboratory, samples were dried up in an oven at the temperature of 100ºC for 24 hours, grinded using the pestle and agate mortar and sieved through 63 µm screen for the analysis. A number of studies focusing on the analysis of heavy metals from sediments have been carried out using this size fraction [7]. The sequential extractions of heavy metals were performed using a four-stage sequential extraction procedure proposed by the European Communities Bureau of Reference (BCR's). The mineralogical composition and major components of metals were analysed using the XRD and XRF spectrometer respectively; and the total organic carbon in sediment samples were determined by using a Wakley Balk titration method. The total analysis of heavy metals contents in sediments samples were analysed using the inductively coupled plasma optical emission spectrometer (ICP-OES).

2.*2. XRD and XRF analyses*

The mineralogical study of the tailings samples were carried out by means of X-ray diffraction (XRD) analysis. The bulk composition was determined using the powder diffraction method, after grinding and homogenization of the samples to 75 µm, nonoriented powders were examined on a Philips X'pert MPD diffractometer, at a power of 1.6 operating at 40KV. Major and trace elements of the tailings were analysed using the X-ray- fluorescence spectrometer (XRF) which was done on the magiX PRO SuperQ Version 4. A rhodium (Rh) anode was utilized in the X-ray tube and operated at 50 kV and current 125 mA; at power level of 4 kW.

2.3. Reagents

Sequential extraction: high quality reagents and deionised water supplied by Oasis were used for all the preparations and analyses. Diluted standard solutions were prepared from the stock standard solutions. A working solution of 0.11 M acetic acid, 0.1 M $NH_2OH.HCL$ (adjusted to pH 2 with 2 M nitric acid), 8.8 M hydrogen peroxide (pH of 2-3), 1.0 M ammonium acetate (adjusted to pH 2 by adding a concentrated HNO_3) and aqua regia (HNO_3 + 3HCL) were prepared using distilled water.

2.4. Apparatus

A platform shaker, centrifuge machine, pH meter, thermometer and a water bath were used throughout all these experiments. A 250 mL flask were used for shaking the sediments sample and 50 mL polypropylene tube was used to centrifuge sediments samples in order to separate the liquid from the solid sediment samples. Trace metals were determined from the sediment samples using the inductively coupled plasma optical emission spectrometer (ICP-OES). A burrette was used for titration procedure proposed by the Walkey Balk for the determination of total organic carbon in sediments samples.

2.5. *Total organic carbon (TOC) procedure.*

The total organic carbon (TOC) contents in sediments samples were determined using the Walkey Balk titration method. The values of total organic carbon contents are presented in percentages in Table 4.

2.6. Sequential extraction method.

To evaluate the binding forms of heavy metals in sediments samples, the sequential extraction proposed by the BCR were applied. All the analysis procedures of sequential extraction used are presented as follows:

First step (exchangeable metals): 40 mL of 0.11 M acetic acid were added to 1.00 g of dry sediment sample. The mixture was shaken at a speed of 250 rpm for 16 h (overnight) at 22 °C. The extract was separated from the solid phase by centrifugation at 3800 rpm for 20 min. The supernatant liquid was decanted into 50 mL polypropylene centrifuge tube and stored in a refrigerator at 4°C prior to analysis. The residue was washed by adding 20 mL of double-distilled water, shaken for 15 min, and then centrifuged again for 20 min at 3800 rpm. The second supernatant liquids were discarded without any loss of residue [11].

Second step (metals bound to iron and manganese oxides): Metals bound to iron and manganese oxides were extracted by adding 40 mL of 0.1 M hydroxyl ammonium chloride ($NH_2OH.HCl$) (adjusted to pH 2 with 2 M nitric acid) onto the residue from the first step. After shaking the mixture for 16 h at 22 °C at a speed of 250 rpm, it was centrifuged for 15 min, and then decanted as described in step 1. The residue was washed by adding 20 mL distilled water, centrifuged, and the supernatant discarded as described in the 1st step before proceeding to the next step [11].

Third step (metals bound to organic matter and sulphides): 10 mL of 8.8 M hydrogen peroxide (pH of 2-3) were carefully added in small aliquots to the residue in the centrifuge tube. The tube were covered loosely (to prevent substantial loss of H_2O_2) and digested at room temperature for 1 h with occasional shaking. The tube was continuously digested for 1 h at 85ºC in water bath with occasional shaking for the first 30 min, and the volume was reduced to 2-3 mL by further heating the uncovered tube. An additional 10 mL of H_2O_2 (pH of 2-3) was added to the tube, again the covered tube were heated to 85ºC and digested for 1 h before the volume in the uncovered tube was reduced almost to dryness. After cooling, 50 mL of 1.0 M ammonium acetate (adjusted to pH 2 by adding of concentrated HNO_3) was added to the

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residue and the mixture shaken for 16 h at 22°C at 250 rpm. The extract were separated from solid phase by centrifugation and decantation as described above and stored at 4ºC [11].

Forth step (residual): The residue from the third step was digested using mixture of an aqua regia (HNO_3 + 3HCL) for metals insoluble in the previous steps. For this purpose, first 6 mL of distilled water and then aqua regia solution in a sequence of 15 and 10 mL were introduced into the same residue. After adding each aqua regia solution, the residue was evaporated to near dryness on a water bath. The extract was filtered through filter paper by adding 1 M HNO_3 solution in small amounts on the last residue in the centrifuge tube [16]).

III. Results And Discussion

3.1. *XRD analysis of mineralogical composition*

The results of mineralogical composition are presented in Table 1. Mineralogical analysis shows that the most abundant mineral is quartz, with 43.40 %– 89.19 % and 29 %– 90.89 % determined in sediments samples collected from the Mooi and Vaal rivers with their connected streams respectively, the rest of the minerals such as clinoferrosilite, diopside, welinite, Iron silicide, rutile, clinoenstalite, kyanite, calcium oxide iron and augite were detected in one or more samples. Similar results which showing the dominance of quartz mineral were obtained in Antarctic lake sediments by Bishop *et al*. [17].

Table I
Mineralogical Composition Of Mooi And Vaal Rivers' Sediments (Fraction <63 µm).

Samples	**Quartz**	**Welinite**	**Iron silicide**	**Rutile**	**Kyanite**	**Clinoferrosilite**	**Diopside**	**Calcium aluminium iron**	**Augite**	**Clinoenstalited**
	%	**%**	**%**	**%**	**%**	**%**	**%**	**%**	**%**	**%**
1	86.04	6.29	-	-	-	5.03	-	-	-	-
2	89.19	-	9.82	-	-	-	-	-	-	-
3	73.79	-	-	22.89	-	-	-	-	-	-
4	83.94	-	0.09	-	-	-	-	-	-	11.45
5	72.22	-	-	-	19.67	-	-	-	-	-
6	43.40	-	-	-	-	53.47	-	-	-	-
7	51.81	-	-	-	-	-	33.48	-	-	-
8	89.62	-	-	-	-	-	8.13	-	-	-
9	29.00	-	-	-	-	-	-	71.00	-	-
10	90.89	-	-	-	-	-	-	-	7.74	-

3.2. *XRF analysis of major components*

The results of XRF analysis of major components are illustrated in Table 2, presented as percentages of the corresponding oxides. The following oxides were observed to be dominant at all sampling sites studied: SiO_2 (45.72% – 84.13%), CaO (0.62-17.96%), Fe_2O_3 (5.73-25.51%), MgO (0-2.48%), MnO (0.16%-3.45%) and Cr_2O_3 (0%– 0.25%). Most of this element oxides were observed to be mainly dominant in sample 1 to 8 (Mooi river and connected streams); however, higher concentrations of magnesium were determined in sample 9 and 8 (Vaal river and connected stream). These dominant major elements can be attributed to the type of rock (dolomites), which have covered most part of the study area. The dominant elements such as Si, Ca, Fe, Mg, Mn, and Cr can be potentially toxic to the aquatic environment of binding sites. The nature of sediments determined in this study is similar to the results found by Gashi *et al*. [1], on sediments from the four main river in Kolovo.

Table II
Selected major and trace components of sediments (fraction < 63 µm)

Samples	**SiO_2**	**Fe_2O_3**	**CaO**	**MnO**	**MgO**	**Cr_2O_3**
	%	**%**	**%**	**%**	**%**	**%**
1	61.24	20.06	1.67	1.47	0.67	0.25
2	84.13	5.73	0.62	3.00	-	-
3	67.42	6.44	2.29	0.65	0.88	0.09
4	62.19	7.16	15.01	0.23	1.83	-
5	45.72	25.51	0.52	3.45	0.52	0.09
6	64.98	8.54	7.15	0.27	1.57	0.17
7	52.02	10.87	16.11	0.32	2.48	0.08
8	58.54	7.54	16.51	0.23	1.88	0.12
9	51.21	9.93	17.96	0.40	2.89	0.06
10	71.19	5.87	4.01	0.16	1.67	0.07

3.3. *Metal speciation.*

The concentration in percentages of Fe, Ca, Mn, Mg and Cr obtained at each extraction step in sediments samples are illustrated in Table 3. Elements such as calcium and manganese were mostly recovered in fraction 1 of the sediment samples collected from the Mooi and Vaal Rivers network. The rest of the metals were found to be strongly bound to the crystalline structure of sediments. Mobility and bioavailability of metals is related to the solubility; therefore, bioavailability decreases in order of exchangeable> reducible> oxidizable> residual. The sediments samples collected in Mooi and Vaal Rivers, showed the mobility and bioavailability of heavy metals decreasing in the following order: Ca> Mn> Mg> Fe> Cr and Ca> Mn> Cr> Fe> Mg, respectively. In this study, high concentration of Fe in sediments samples collected from the Mooi and Vaal Rivers were in the form of residual fraction (83.46% and 35.21%, respectively). Its contribution to exchangeable fraction, reducible fraction and oxidizable fraction were (1.65% and 0.76%), (5.89% and 3.79%) and (5.16% and 93.92%), respectively. The mobility and bioavailable will be very low due to less percentages bound to exchangeable fraction, and this demonstrates relatively lower risk of toxicity will be posed by this element into the surface water. These results were in agreement with the studies performed by Yuan *et al*. [12] who found that more than 90 % of the total Fe were bound to the residual fraction.

Calcium and magnesium were obtained in all four fractions of BCR sequential extraction. In sediments collected from both the Mooi and Vaal Rivers, high recoveries percentages of magnesium in the form of residual were 88.59 % and 88.83 %, respectively. Its contribution to exchangeable, reducible and oxidizable were (9.64 % and 1.44 %), (28.79 % and 1.46 %) and (16.39 % and 1.48 %), respectively. Whereas, the distribution of calcium in both Mooi and Vaal Rivers, exchangeable fraction, reducible fraction, oxidizable fraction and residual fraction were as follows: (72.92 % and 39.53 %), (83.29 % and 0.30 %), (45.01 % and 0.18 %) and (10.54 % and 88.59 %), respectively. High percentages of Ca associated with the exchangeable fraction, show that the mobility and bioavailability of this element will be high, and this is likely to increase the hardness of water, which can result to the increase of pH in surface water. These high values of Mg and Ca extracted from sediments can be attributed to the underline rock of dolomites; however, in contrast to Ca, most of the magnesium was bound to the residual fraction, which indicates that only a slight amount of Mg could be released into water.
Chromium was mainly found in the form of residual fraction, with the highest concentration of 27.43 % and 14.1 % extracted in sediments samples from the Mooi and Vaal Rivers, respectively. Its contribution to exchangeable, reducible and oxidizable, were 1.27 % - 2.20 % - 7.75 % and 15.92 % - 12.01 % - 4.02 %, respectively. It has been shown from the other studies that chromium strongly bind to the crystalline structure of the sediments [7, 18]. These indicate the lower mobility and bioavailability of Cr from the sediments.

The distribution pattern of manganese in sediments collected across the rivers, showed that it was mainly bound to the oxidizable or organic matter and sulphides; with the highest percentage (64.19 %) found in sediments from a stream connected to Vaal River. The concentration of manganese in other fractions was 33.84 % bound to exchangeable (at Mooi River), 24.94 % bound to Fe and Mn (at stream connected to Mooi River) and 43.82 % bound to residual (at Mooi River). Manganese released in the form of exchangeable and reducible fractions in these sediment samples will be bioavailable and this can be more harmful to the biota. The distribution of Mn in sediment fractions as observed in this study is in agreement with previous studies carried out using the four-step (BCR) sequential extraction procedure in sediments samples from Tokat, Turkey, [19], which showed that manganese was mainly bound to the organic matter fraction.

TABLE III
CONCENTRATION OF HEAVY METALS (%) IN THE SEQUENTIAL FRACTIONS OF THE SEDIMENTS FROM THE MOOI AND VAAL RIVERS

Sample1	**Fe**	**Ca**	**Mn**	**Mg**	**Cr**
Step1	0.01	37.40	2.55	18.74	0.52
Step2	1.12	2.39	24.94	3.09	0.56
Step 3	0.68	1.61	6.64	2.99	2.11
Step 4	24.94	6.84	5.48	14.52	16.57
Sample2					
Step1	0.04	72.92	19.16	0.00	0.00
Step2	5.89	21.25	1.03	0.00	0.00
Step 3	5.16	5.82	0.21	0.00	0.00
Step 4	69.15	10.54	0.79	0.00	0.00
Sample3					
Step1	1.65	37.85	33.84	13.09	0.12
Step2	0.04	17.38	1.99	5.43	0.17
Step 3	3.71	8.76	1.27	9.68	0.58
Step 4	43.85	3.26	1.14	29.09	4.15

Sample4					
Step1	0.00	30.59	22.47	17.97	0.00
Step2	5.19	0.41	0.00	1.59	0.00
Step 3	3.21	0.26	64.19	1.72	0.00
Step 4	83.46	0.51	43.82	6.61	0.00
Sample5					
Step1	0.00	60.22	0.78	28.79	1.27
Step2	0.62	83.29	0.48	9.64	1.07
Step 3	1.09	24.47	0.15	16.39	12.01
Step 4	19.54	1.28	0.58	88.83	27.43
Sample6					
Step1	0.03	15.55	13.24	9.19	0.60
Step2	2.87	11.56	17.41	4.09	0.63
Step 3	4.21	45.01	6.23	8.39	3.79
Step 4	64.39	24.07	5.85	29.34	1.68
Sample7					
Step1	0 .01	38.17	20.85	21.11	1.76
Step2	3.03	3.11	2.73	1.56	1.28
Step 3	0.86	0.89	0.00	0.26	11.06
Step 4	23.03	0.87	1.88	11.05	15.19
Sample 8					
Step1	0.02	32.99	20.07	18.31	1.10
Step2	0.82	0.01	1.31	2.27	1.35
Step 3	4.64	1.49	4.74	8.45	3.67
Step 4	41.82	0.77	3.72	21.76	9.99
Sample9					
Step1	0.00	39.53	26.06	1.44	2.20
Step2	3.79	0.30	1.46	1.46	7.75
Step 3	7.25	0.18	0.38	1.48	4.02
Step 4	35.21	1.64	4.36	23.93	14.10
Sample10					
Step1	0.76	27.32	21.03	6.88	1.60
Step2	1.54	0.88	15.77	1.31	15.92
Step 3	93.92	2.13	63.27	10.29	16.11
Step 4	0.26	88.59	32.34	12.82	4.15

IV. CONCLUSION

The sediments from the Mooi River and Vaal are dominated by oxide minerals and contain mainly Fe, Cr, Mg, Mn and Ca. According to the modified BCR sequential extraction results, it can be concluded that most of the elements except for Ca and Mn were mostly bound to the residual fraction, which presages more to the increase of water hardness. However, elements such as Fe and Cr being associated with the exchangeable and reducible fractions, shows that these elements can be progressively release at a speed that will vary depending on the environmental conditions and could therefore degrade the quality of water.

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