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Investigation of Trace Metals in Water Samples Taken from Kura River

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Abstract The present study was conducted to investigate the trace metal contents (Al, As, Ba, Cd, Co, Cr, Cu, Mo, Mn, Pb, Ni, Sr, V, U and Th) of water samples, which were collected from 10 different sites along the river Kura in Azerbaijan Republic. Agilent 7700x Series ICP-MS was used for measurements. The mean concentration of studied metals in the water, followed the decreasing order of Sr > Ba > Al > Fe >Mo > V > As > Cu > U > Ni > Cr > Co > Pb. The concentration of Cadmium and Thorium in all samples were less than 0.02 μg/L and 0.01 μg/L respectively. The measured metals content in water samples for all sites has been found to be less than the limit of recommended by the World Health Organization. The analysis of results showed strong concentration correlation (R: 0.8-1) between groups of elements: 1) Al-Cu-Fe-Ni, 2) Cu-Ni-Co, 3) Al-Pb-Fe, 4) Mo-U- Sr and 5) Ba-Cr at p-value < 0.01 level and may indicate same source. A strong negative correlation exists between Fe and Ba R=-0.65 and is an indication of distinct sources for the metals in the river.

Keywords Kura River, Trace metals, Agilent 7700 Series ICP-MS, Azerbaijan

1. Introduction

The river Kura and its main tributary Aras constitute the main waterways of the South Caucasus. Kura and its tributaries receive inputs of water from at least five countries: Armenia, Azerbaijan, Georgia, Iran, and Turkey, before it finally reaches the Caspian Sea. This provides potential for transboundary water pollution within the Kura–Aras watershed. Azerbaijan, situated along the lower stretches of Kura–Aras, is particularly exposed to pollution from countries located further up along the rivers [1, 2]. Figure 1 shows a map of the South Caucasus and surrounding areas, indicating how the Kura–Aras river system may be influenced by ecosystems of different countries.

The Kura river starts in Turkey, flows through the main urbanized areas of Georgia including the capital city Tbilisi, and then continues into Azerbaijan. On its way, Kura also receives contributions from some tributaries originating in Armenian territory, contributing to the Azerbaijani section of the Kura river via Georgian territory or directly. Aras also has its initial sources in Turkey and then follows the Turkey/Armenia, Iran/Armenia, and Iran/Azerbaijan borders until it finally enters Azerbaijan territory and joins Kura at Sabirabad, about 100 km from the outlet in the Caspian Sea. This means that there may be considerable potential for transboundary transfer of water pollutants into Azerbaijan, and finally into the Caspian Sea, from other countries in the region. Water pollution is a complex process that leads to changes in water composition, aquatic flora and fauna, and causes water quality deterioration. The economic and recreational use of such poor-quality water becomes dangerous to human health [3-5].

Recognition of the importance of trace metal concentrations evidence in natural waters and/or environment is growing in pollution monitoring studies. For instance, authors of [6] reported about the determined trace metals in drinking water in Irbid (Jordan), and results showed that the level of most elements (As, Ba, Cd, Pb, Cr, Cu, Fe, Zn, Mn, Ni, and Se) were within the Jordanian standards and WHO standards for drinking water. In [7], the occurrence of heavy metals in water of San Petro River in Mexico was studied. The results confirmed that the San Pedro River is contaminated with heavy metals and other contaminants that might affect human health as well as the health of the ecosystem.

The assessment of heavy metals pollution in surface water in Ganga in West Bengal showed that the dominance of various heavy metals in the surface water of the river Ganga followed the sequence: Fe >Mn > Ni > Cr >Pb > Zn > Cu > Cd [8]. The investigation of different heavy

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metals in surface water from Transylvania/Romania showed that toxic heavy metals were detected in water samples in the range of few ppb [9]. In work [10] authors have analyzed different heavy metals in water samples from Malaysia and results showed that the concentrations of heavy metals in water samples were below the detection limit.

Monitoring and assessment of the water pollution has become a very critical area of study because of direct implications of water pollution on aquatic life and human beings. The contamination of surface water by heavy metals is a serious ecological problem as some of them, like Hg and Pb, are toxic even at low concentrations, are non-degradable and can bio-accumulate through the food chain. Though some metals, like Fe, Cu and Zn, are essential micronutrients, they can be detrimental to the physiology of the living organisms at higher concentrations [11, 12].

Heavy metals are widespread pollutants of great environmental concern, as they are non-degradable, toxic, and persistent with serious ecological ramification on aquatic ecology [13].

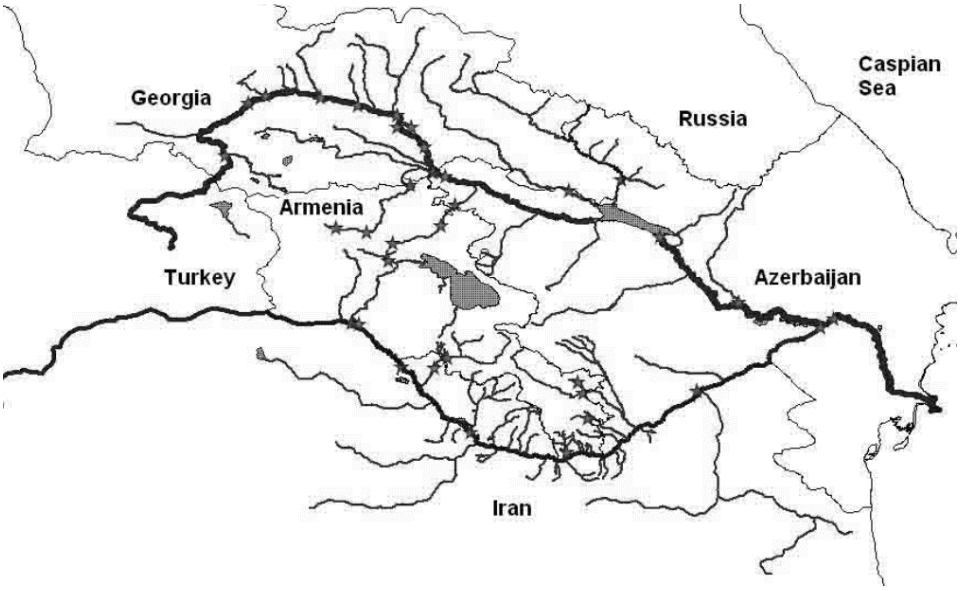

Figure 1. Kura-Aras watershed

2. Materials and Methods

Humans have always depended on aquatic resources for food, medicines and materials as well as recreational and commercial purposes such as fishing and tourism [14]. Determination of natural radio-nuclides such as U and Th in water samples are also important. The determination of uranium and thorium concentrations in geological samples is very important in the exploration of the natural resources of these elements. Of these geological samples, natural waters have special importance as they are indicators of uranium and thorium anomalies.

Uranium concentrations of 0.5-1.0 ppb exist in the waters of South Greenland [15]. In the survey of radioactivity in Boyuk Menderes River, Turkey, detected uranium and radium concentrations of 0.24-17.65 ppb and 0.016- 0.751 Bq/l respectively [16]. Bolivar et al. conducted hydro geochemical and stream-sediment reconnaissance in the Montrose quadrangle (Colorado) and determined that the uranium concentration in waters there varied within the range of 0.02-856 ppb [17].

Uranium concentration in 498 drinking water samples taken from four districts of SW-Punjab India has been found to vary between 0.5–579 μgl^{-1} with an average of 73.5 μgl^{-1} [18]. The concentration of uranium in various rivers of India such as Yamuna (0.09–3.61 ppb) and Chambal (0.2–1.74 ppb) [19], Bhagirathi (2.11–3.96 ppb) and Alakanda (1.86 ppb) [20] have been previously studied. Springs and streams in Himachal Pradesh, India had 0.07 to 4.65 ppb of uranium [21].

The estimated worldwide averages for dissolved uranium in rivers range between 0.3-0.6 ppb [22] and reach to abnormal high values in some cases due to chemical weathering of uraniferous rocks such as in Platte River of the North American High Plains Region where uranium contents reach up to 31.7 ppb [23]. It has been determined the concentrations of dissolved uranium in forty major rivers from around the world have an average concentration of 0.31 ppb [24].

They noted that this value could be biased by the very high levels observed in the Ganges-Brahmaputra and the Yellow River. Excluding these two river systems, the global average of uranium is reduced to 0.19 ppb. Compared to uranium, thorium is a highly particle-reactive element and does not readily occur as a dissolved ion [25].

The present study was conducted to investigate the heavy metal contents (Al, As, Ba, Cd, Co, Cr, Cu, Mo, Mn, Pb, Ni, Sr, V, U and Th) of water samples in ten different sites along the river Kura in Azerbaijan region; the coordinates are shown in Table 1.

Table 1. The geographical coordinates of sampling stations

№	1	2	3	4	5	6	7	8	9	10
ns1:longitude		45.792719	46.154787	46.227926	47.052552	47.038146	47.05838	48.462661	48.472762	49.260171
ns1:latitude		41.075406	40.958492	40.918927	40.770827	40.790342	40.747847	40.022876	40.036002	39.420507

Water samples were collected at the intersections of Kura River with the flowing waters by means of a standard polyethylene water sampler, which was rinsed a few times with river water from the sampling point before representative sampling from 15-30 cm below water surface. Two hundred millilitres of water were filtered through a 0.45 μm membrane filter using a plastic filtration assembly without a pump. A few drops of high-purity nitric acid were added to the filtrate to adjust to pH < 2. The sample was stored at 4 °C during transportations to the laboratory. Between each sampling, the water sampler was soaked with 10% v/v nitric acid and rinsed with ultrapure water. All plastic-ware sample bottles, pipette tips, filtration unit and flasks were soaked in 10% v/v HNO3 for 24 h and rinsed with ultra-pure water before being used. Milli-Q ultra-pure water (resistivity 18.2 MΩ·cm, pH (5.5-6.5)) was used throughout, and in all laboratory operations. In the laboratory, by adding an appropriate volume of nitric acid, the acid concentrations of the samples were adjusted to approximate a 1% (v/v) nitric acid solution. Multi-element calibration working standards solutions were prepared by appropriate dilution of 10 mg /L multi-element stock standard solutions-Environmental Calibration Standard- Part# 5183-4688 in 5% HNO3 in 1% HNO3 correspondingly.

The concentrations of trace metals in water samples taken along the river Kura were measured using inductively coupled plasma mass spectrometry (ICP-MS). The Agilent 7700x Series ICP-MS applied to analyse the water samples. The method is based on the direct introduction of samples into an inductively coupled plasma mass spectrometer (ICP-MS), without any chemical pre-treatment. An Agilent 7700x ICP-MS system was used to measure each sample in helium mode, using standard Agilent-recommended auto tuning for robust tuning conditions (around 1.0 % CeO/Ce).

3. Results and Discussion

The samples were analysed for Al, As, Ba, Cd, Co, Cr, Cu, Mo, Mn, Sr, Pb, Cu, Ni, V, U and Th using an Agilent model 7700x inductively coupled plasma -mass spectrometry. The 1/% ₋ solutions of multi-element calibration standards in HNO3, were prepared by corresponding dilution of from 10 mg/L multi-element stock standard solutions in 5% HNO3 (Part # 8500-6940).

The blank and calibration solutions were measured in optimized conditions. The calibration curve was automatically plotted by the instrument. The linear correlation coefficient (R) in all calibration curves was better than 0.9995. Instrument drift and matrix effects during measurement were corrected by using Sc, Ge, Rh, In, Tb, and Bi, containing internal standards, which were prepared by appropriate dilution from stock ICP-MS Internal Standard (Mix Part# 5188-6525); they were added on-line at the time of analysis using a second channel of the peristaltic pump. For quality control purposes, duplicate samples, matrix-spike samples were analysed. The 7700x ICP-MS Operating Conditions were used for He mode.

CRMs were purchased from the NRCC (National Research Council of Canada) and at the Absolute standard and were analysed to validate our procedure: SLRS-5 (river water) and SRM 1640 a respectively.

The values and ranges of element concentrations in the water samples are presented in Tables 2 and 3. The concentrations of the analysed elements in water samples were as following: Al: ranged between 0.487—30.62 ug/L, average concentration-9.814 ug/L, As: ranged between 1.031–2.986 ug/L, average concentration-1.947 ug/L, Ba: ranged between 27.74—57.47 ug/L, average concentration-40.32 ug/L, Cr: ranged between 0.110—1.250 ug/L, average concentration-0.348 ug/L, Co: ranged between 0.024—0.050 ug/L, average concentration-0.035 ug/L, Cu: ranged between 1.186–3.200 ug/L, average concentration-1.940 ug/L, Mn: ranged between 0.264–4.358 ug/L, average concentration-0.930, Mo: ranged between 1.238–8.597 ug/L, average concentration-3.538 ug/L, Ni: ranged between 0.346–0.992 ug/L, average concentration-0.632 ug/L, Fe: ranged between 0.673–16.70 ug/L, average concentration-4.889 ug/L, Pb: ranged between 0.010-0.031 ug/L, average concentration-0.020 ug/L, V:, ranged between 1.792-3.459 ug/L, average concentration 2.631 ug/L, Sr: ranged between 265.4—1060 ug/L, average concentration-564.2 ug/L and U: ranged between 0.697—2.919 ug/L, average concentration-1.434 ug/L respectively (Table 2). The values of cadmium and thorium on all sampling points were less than 0.02 μg/L and 0.01 μg/L respectively.

The mean concentration of studied metals in water followed a decreasing order of Sr > Ba > Al > Fe > Mo > V > As > Cu > U > Ni > Cr > Co > Pb. The measured metals contents in water samples from all sites were found to be less than the limit recommended by the World Health Organization [26]. According to the comparison between the values of the heavy metals and the WHO guidelines for drinking water mentioned in Table 3, the river water can be considered as useful for drinking purposes.

Correlation analysis provides an effective way to reveal the relationships between multiple variables and thus has been helpful in understanding influencing factors as well as the sources of chemical components. The degree of linear association between any two or more of the water quality

parameters were measured by evaluating the Pearson's correlation coefficients R. The values of Pearson's correlation coefficients of heavy metals found in the Kura River water is shown in Table 4.

Below we use the terminology for the correlation coefficient (R) ranges suggested by Evans [27]: range 0.00-0.19: "very weak"; range 0.20-0.39:- "weak"; range 0.40-0.59: "moderate"; range 0.60-0.79: "strong"; range 0.80-1.0: "very strong". Correlation analysis showed very strong correlation (R: 0.8-1) forming between the groups elements: 1) Al-Cu-Fe-Ni, 2) Cu-Ni-Co, 3) Al-Pb-Fe, 4) Mo-U- Sr and 5) Ba-Cr at p-value < 0.01 level may indicate same source. A strong correlation exists between: 1) Al-Co, 2) Ba-U, 3) Co-Fe, 4) Cu-Pb, 5) Mn-U, 6) As-Sr, 7) As-Ba, 8) Mn-Sr, 9) V-Co, 10) V-Ni, 11) Ni-Pb 12) Fe-Co, and 13) Mn-Mo (R: 0.6-0.79 at P<0.01 level) respectively, thereby indicating same source . Similarly: 1) As showed moderately positive correlation with Mn, Mo and U, 2) Ba with Mo, 3) Co with Mn and Pb, 4) Mo with V 4) V with U (0.05 level) and can also be attributed to same origin. The moderately negative correlation has been observed between metals: 1) Al with As, Ba, U and Sr, 2) As with Fe, 3) Ba with Ni and Pb and 4) Fe with U and Sr is an indication of distinctive sources for the metals in the river. A strong negative correlation exists between Fe and Ba R=-0.65 and is also an indication of distinctive sources for the metals in the river. The concentration of Cr showed a weak to very weak correlations with other metals (with the exception of Ba) suggesting that Cr was from different sources than the other metals.

Table 2. The concentrations of the analysed elements in water samples

№	St1	St2	St3	St4	St5	St6	St7	St8	St9	St10
Sr(ug/L)	265.4	271.5	469.7	398.0	520.6	513.8	534.1	758.7	1060	850
Al(ug/L)	25.61	30.61	2.807	6.834	0.487	3.615	0.744	9.884	6.173	11.368
As(ug/L)	1.283	1.322	1.031	1.731	2.137	2.098	2.892	2.033	2.986	1.958
Ba(ug/L)	27.74	30.43	57.47	33.93	39.14	39.55	36.43	44.98	51.23	42.34
Cd(ug/L)	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Cr(ug/L)	0.151	0.136	1.250	0.297	0.219	0.110	0.111	0.353	0.465	0.389
Co(ug/L)	0.044	0.050	0.028	0.039	0.024	0.027	0.031	0.028	0.048	0.032
Cu(ug/L)	2.667	3.200	1.738	1.867	1.186	1.275	1.570	1.609	2.224	2.065
Mn(ug/L)	0.794	1.011	0.321	0.264	0.283	0.637	0.364	0.421	4.358	0.843
Mo(ug/L)	1.238	1.481	1.377	1.936	2.118	2.210	2.281	6.903	8.597	7.240
Ni(ug/L)	0.855	0.992	0.522	0.755	0.346	0.377	0.431	0.569	0.713	0.532
Fe(ug/L)	13.33	16.70	1.547	3.550	0.673	2.789	1.159	2.851	1.764	4.530
Pb(ug/L)	0.029	0.031	0.014	0.015	0.010	0.027	0.012	0.019	0.020	0.024
V(ug/L)	2.706	2.798	2.762	3.459	1.792	1.819	1.867	2.790	3.449	2.867
U(ug/L)	0.697	0.716	1.481	1.161	1.209	1.216	1.211	1.815	2.919	1.914
Th(ug/L)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Table 3. Descriptive statistic results of concentrations of heavy metals compared to WHO guidelines

		Min	Mean	Max	STDEV	WHO
Sr	ug/L	265	564	1060	254	
Al	ug/L	0.487	9.814	30.61	10.35	100
As	ug/L	1.031	1.947	2.986	0.646	10
Ba	ug/L	27.74	40.32	57.47	9.149	700
Cd	ug/L	<0.02				3
Cr	ug/L	0.110	0.348	1.250	0.341	50
Co	ug/L	0.024	0.035	0.050	0.009	
Cu	ug/L	1.186	1.940	3.200	0.625	2000
Mn	ug/L	0.264	0.930	4.358	1.233	50
Mo	ug/L	1.238	3.538	8.597	2.843	70
Ni	ug/L	0.346	0.609	0.992	0.213	70
Fe	ug/L	0.673	4.889	16.70	5.515	100
Pb	ug/L	0.010	0.020	0.031	0.007	10
V	ug/L	1.792	2.631	3.459	0.618	
U	ug/L	0.697	1.434	2.919	0.655	30
Th	ug/L	<0.01				

Table 4. Pearson correlation matrix, between different metals in the water of Kura River

	Al	As	Ba	Cr	Co	Cu	Mn	Mo	Ni	Fe	Pb	V	U	Sr
Al	1	-0.52	-0.58	-0.29	0.72	0.91	0.05	-0	0.86	0.98	0.81	0.32	-0.41	-0.42
As		1	0.15	-0.36	-0.07	-0.38	0.50	0.55	-0.41	-0.55	-0.35	-0.16	0.59	0.67
Ba			1	0.82	-0.35	-0.39	0.32	0.46	-0.42	-0.65	-0.41	0.18	0.73	0.63
Cr				1	0.2	-0.11	0.04	0.06	-0.11	-0.33	-0.33	0.32	0.34	0.18
Co					1	0.89	0.59	0.08	0.92	0.70	0.56	0.64	0	-0.08
Cu						1	0.32	-0	0.93	0.89	0.70	0.53	-0.19	-0.25
Mn							1	0.63	0.28	0	0.20	0.50	0.74	0.64
Mo								1	-0.07	-0.32	0.00	0.42	0.89	0.94
Ni									1	0.83	0.60	0.67	-0.20	-0.31
Fe										1	0.80	0.2	-0.55	-0.56
Pb											1	0.17	-0.22	-0.2
V												1	0.42	0.27
U													1	0.96
Sr														1

4. Conclusions

Within the presented project, we studied the concentration of trace metals in the Azerbaijani region of the Kura River. The mean concentration of studied metals in water followed a decreasing order of Sr > Ba > Al > Fe > Mo > V > As > Cu > U > Ni > Cr > Co > Pb. The measured metals content in water samples in all sites has been found to be less than the limit recommended by the World Health Organization. Correlation analysis provides an effective way to reveal the relationships between multiple variables and thus has been helpful for understanding influencing factors as well as the sources of chemical components. Significant correlation relationships between heavy metals pairs were reported, suggesting existence of a common source of these metals in the water. For estimation the origin of observed metals in samples, the additional investigations are continued in laboratory. Based on the results of our studies, it is recommended: (1) to regularly monitor water quality at the intersections of Kura with the flowing waters; (1) within the country, to build water treatment plant facilities on the drainage waters poured into the Kura, (2) to build water reservoirs over the rivers flowing into Kura from the neighboring countries.

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