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DETERMINATION OF RADIONUCLIDES AND METALS CONCENTRATION IN CASPIAN SEA SEDIMENTS

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Abstract: The distribution, of heavy metals and naturally occurring radionuclides U-238 (Ra-226), Th-232 (Ra-228) and K-40 in sediment samples Caspian Sea were investigated to obtain information about the sources and degree of contamination. 16 surface sediment samples were collected and analyzed for major elements (Al, Fe), heavy metals (As, Ba, Cd, Co, Cr, Cu,Hg, Mn, Ni, Pb, Zn) and naturally occurring radionuclides. The average concentrations of these heavy metals at the same sampling location followed the order of Al>Fe>Mn>Ba>Cr>Zn>Ni>Cu>Pb>Co>As>U>Hg>Cd.

The chemical contamination in the sediments was evaluated by comparison with the sediment quality guideline proposed by USEPA. The Pearson correlation coefficients analysis applied to determined heavy metal and radionuclide concentrations of investigated surface sediment samples.

Keywords: radionuclides, heavy metals, Caspian Sea, Azerbaijan

1. Introduction

The Caspian is the largest inland body of water in the world, containing some 44% of the globe's inland waters. The Caspian Sea occupies a deep depression on the boundary between Asia and Europe with a water level at present 27 m below sea level. It is approximately 1200 km long with a maximum breadth of 466 km, contains 79,000 km3 of water, and has a total coastline of more than 7000 km. The Caspian is fed by five major rivers or river groups: in the north the Volga (80% of total inflow) and the Ural (5%); in the west the Terek, Sulak and Samur (4-5%) and the Kura (7-8%); and, in the south, the short mountain rivers from the Iranian Alborz range (4-5%). Azerbaijan has more than 800 km of coastline along the Caspian Sea and almost the entire country is part of the Sea's catchment area. Environmental problems of the Caspian Sea are multiple and various in their origin. On one hand, they are caused by the commercial use of the sea; on the other hand, human activity impacts coastal areas, including input from rivers in the Caspian. As the Caspian is an inland water body, anthropogenic (man-caused) impacts on catchment area (about 3.5 million km2) accumulate here. Anthropogenic impact on the Caspian ecosystem occurs concurrently with various natural endogenous and exogenous processes. It is primarily sea level changes, periodical seismic activity, surges and retreats, mud volcanoes and neo-tectonics. Special features of the Caspian include constant alterations of its area, volume, and configuration of the coastline and water column structure. Anthropogenic activity, as well as a natural impact, can have a chronic (long term) or acute (short term) effect. The sources of pollution are industrial, agricultural and accidental discharges and sewage. The main sources of pollution to the Caspian Sea have generally been considered to be offshore oil production and land-based sources, notably the Volga River

The presence of oil in the Caspian Sea region has been known since ancient times and the oil reserves were amongst the first to be exploited in the world. Baku was a centre for oil production at the turn of the 20th century and developed further development during the era of the Soviet Union. Oil production areas cover vast areas of the coastal zone, particularly along the south coast of the Apsheron Peninsula in Azerbaijan. Population growth and industrial development in the Caspian region have generated an immense pollution problem. Ten million people live adjacent to the Caspian Sea and 60 million more live in the Volga River watershed [1-3]. The World Bank estimates that one million cubic meters of untreated industrial wastewater is discharged into the Caspian annually. Soviet oil extraction left behind polluted soil and water, rusty equipment, and well fires that burned for years. Meanwhile, the five countries surrounding the sea are rushing to exploit still untapped oil deposits. Since the Caspian is an enclosed body of water, it has limited carrying capacity compared to larger and more open bodies of water. Pollution entering the Caspian is either biogeochemically altered, or remains in the Sea for years; none escapes and dilution is limited from external buffering waters. Since the Caspian is an enclosed body of water, it has limited carrying capacity compared to larger and more open bodies of water. Pollution entering the Caspian is either biogeochemically altered, or remains in the Sea for years; none escapes and dilution is limited from external buffering waters. In the former Soviet Union, water and sediment quality measurements were taken on a regular basis and with good coverage. In Azerbaijan, the situation reported to be changing, and the number of monitoring surveys has increased. Within the current situation, over the same period the flux of pollutants into the Caspian has changed, with a drastic reduction in industrial and agricultural activity in Turkmenistan, Kazakhstan, the Russian Federation and Azerbaijan [4-7].

In this study heavy metal concentration and naturally occurring radionuclides U-238, Ra-226 and K-40 in sediment samples Caspian Sea were investigated to obtain information about the sources and degree of metal contamination. It is know that the important geochemical phases for metals in marine sediments can be deduced by studying the relationships between the concentration of metals and, Fe and Al.

2. Materials and methods

Sediment samples from the stations were collected by Van Veen Grab fitted with stainless-steel jaws. At each station for metal analysis a sample of approximately 200g was taken from the surface oxic layer of sediment and stored in a container that was frozen on returning to the laboratory.

Sediments were sampled air dried in the laboratory, disaggregated with a mortar and pestle and passed through a 2 mm by sieve. Particles larger than 2 mm were discarded. Dry material of size less than 2 mm was mixed well and kept in labeled plastic containers for further analysis. To determine the analytical and handling error, two sub-samples were taken from each of these sediment samples, each undergoing an independent digestion procedure. All chemicals were of analytical-reagent grade purchased from Merc and Aldrich. Solutions were prepared in deionized water obtained with a Milli-Q apparatus (Millipore). Calibration standards for AAS were supplied By Aldrich and CPI.

Aliquots of approximately 0.25 g of sediment samples were weighed into acid-cleaned TFM vessels and digested with mixture 6 ml of nitric, 2 ml of hydrochloric and 3 ml hydrofluoric acid in microwave oven (Milestone Ethos plus with HPR –1000/10S high pressure rotor) operated following microwave run program(Table 1):

Table 1. *Microwave heating program*

Step	Time(minutes)	Temperature	Microwave power
1	4	40^oC	Up to 1.000 Watt
2	4.30	90^oC	Up to 1.000 Watt
3	4.30	190^oC	Up to 1.000 Watt
4	7	220^oC	Up to 1.000 Watt
5	20	220^oC	Up to 1.000 Watt

Trace metals were determined by AAS 220 FS+GTA 110+VGA 77 firma Varian and AAS AAnalyst 800 with Zeeman corrector firma PerkinElmer. All chemicals used were of analytical reagent grade. Deionized water was used throughout the experiment. Stock standard solution (1000 ppm) of heavy metals was purchased from Merck, Germany. The working standard solutions were prepared daily by diluting the stock standard solution of each metal. Sediment samples were analyzed for 226Ra, 228Ra, and other radionuclides (K40, Co60, Cs134 and Cs137) via gamma-spectrometry using a Canberra intrinsic germanium detector. All gamma spectrometric analyses were performed in silicone sealed Marinelli beakers after aging for one month to allow for ingrowth of 222Rn and daughters. The photopeaks from the radon daughters 214Pb and 214Bi at 295, 352, and 609 keV were used to quantify 226Ra and the 228Ac peaks at 338 and 911 keV were used for 228Ra [8-10]. For all samples the accumulation time of specters was 4 hour.

3. Results and discussion

Descriptive statistics of the measured parameters for sediment samples including mean, maximum, minimum and also standard deviation all are shown in Table 2. The concentrations of the analyzed elements in Caspian sea sediment samples were as following: Al: ranged between 24690-72400 mg/kg, average concentration-56420 mg/kg, Ba: ranged between 61-743 mg/kg, average concentration-418 mg/kg, As: ranged between 1.84—9.51 mg/kg, average concentration-7.10 mg/kg, Cd: ranged between 0.082—0.29 mg/kg, average concentration-0.12 mg/kg, Co: ranged between 7.78—21.29 mg/kg, average concentration-16.31 mg/kg, Cr: ranged between 27.68—294.3 mg/kg, average concentration-111.2 mg/kg, Cu: ranged between 27.41—62.19 mg/kg, average concentration-46.64 mg/kg, Fe: ranged between 7515-26533 mg/kg, average concentration-22260 mg/kg, Hg: ranged between 0.064—0.198 mg/kg, average concentration-0.13 mg/kg, Ni: ranged between 25.18—60.17 mg/kg, average concentration-49.81 mg/kg, Pb: ranged between 11.87—22.62 mg/kg, average concentration-17.53 mg/kg, Mn: ranged between 372–826 mg/kg, average concentration-705 mg/kg, Zn: ranged between 45.56—93.38 mg/kg, average concentration-78.74 mg/kg, U: ranged between 0.442—2.04 mg/kg, average concentration-1.44 mg/kg, Total activity: ranged between 245-711 Bq/kg, average concentration-525 Bq/kg, Ra-226: ranged between 5.49—25.91 Bq/kg, average concentration-17.90 Bq/kg, K-40: ranged between 234-630 Bq/kg, average concentration-473 Bq/kg, Cs137: ranged between 3-27 Bq/kg, average concentration-11Bq/kg (Table 2). The results showed that the concentrations of these heavy metals in sediments varied from different sampling locations. The average concentrations of these heavy metals at the same sampling location followed the order of Al>Fe>Mn>Ba>Cr>Zn>Ni>Cu>Pb>Co>As>U>Hg>Cd.

The chemical contamination in the sediments was evaluated by comparison with the sediment quality guideline proposed by USEPA [14]. These criteria are shown in Table 3. Concentration of As is one site not polluted, in eighth site moderately polluted and seventh site heavily polluted. Cd, Pb and Hg in all stations under investigation belong to unpolluted class. Site 11 and 14 for Cr station 1, 2, 5, 10, 11 and 14 for Cu, and station 4 and 14 for Ni were considered as moderately polluted and other all sites they are heavily polluted. Concentration of Zn except sites 4 and 6 were grate than 90 mg/kg, all sediment samples under the investigation area are not polluted. Ba heavily polluted in all stations and probably related by oil production in Caspian Sea. The concentrations of the U in Caspian Sea sediment were ranged from 0.442 to 2.04 mg/kg with average concentration—1.44 mg/kg, and within the crust abundance (< 5 mg/kg).

According results of analysis, radioactivity observed for Naturally Occurring radionuclides (Ra226, Th232 and K40) characteristically for Sea sediments. Artificial radionuclides Co-60 and Cs-134 in investigated samples were below MDA but radioactivity of Cs-137 change in the range of 0.1-27.1 Bq/kg. It can be explained with quickly dissolving ability of Cs - 137 in water and forming colloids as the ionic form.

Table 2: *Measured concentrations and descriptive statistics of the measured parameters for sediment samples*

-	Al	As	Ba	Cd	Co	Cr	Cu	Fe	Hg	Ni	Pb	Mn	Zn	U	Ra^{226},	K-40	Cs137	Total activity
Cт.	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	Bq/kg	Bq/kg	Bq/kg	Bq/kg
1	66540	5.26	480	0.131	17.09	111.76	49.03	25106	0.14	54.21	20.24	764	81.62	1.36	16.93	466	8.83	517
2	58180	9.51	511	0.129	16.84	109.36	48.12	23512	0.166	51.43	19.81	755	86.79	1.28	15.90	440	5.81	463
3	64250	7.74	236	0.095	18.37	135.61	55.81	24779	0.064	56.19	15.38	788	79.08	1.5	18.61	463	5.57	511
4	67430	9.19	565	0.142	16.76	92.57	52.59	25678	0.176	52.81	22.52	743	90.41	2.04	25.91	629	23.86	708
5	33804	6.35	277	0.103	19.81	197.44	36.22	22765	0.101	51.19	15.29	814	76.43	1.33	16.56	395	6.23	441
6	67080	7.86	553	0.115	16.91	95.52	52.61	26435	0.198	53.78	22.62	737	93.38	1.97	24.44	598	24.1	681
7	57780	8.49	172	0.097	18.53	108.56	34.03	18538	0.071	51.04	13.23	805	63.44	1.12	13.97	379	2.94	413
8	69870	8.83	498	0.111	15.31	88.96	51.41	26533	0.176	52.37	21.94	654	90.39	1.38	17.10	630	27.12	711
9	63750	9.37	534	0.104	16.53	96.48	52.79	24062	0.156	53.44	18.51	719	83.91	1.72	21.35	508	16.27	574
10	35600	6.04	215	0.082	21.49	294.32	36.04	25327	0.082	52.03	13.87	693	80.74	1.38	17.10	329	3.37	367
11	33820	4.45	61	0.096	7.78	27.68	27.41	7515	0.123	25.18	12.82	372	45.56	0.945	11.74	289	11.69	334
12	64620	8.14	743	0.29	17.76	95.84	62.19	26466	0.148	60.17	19.44	826	89.68	1.77	21.96	611	12.21	680
13	61380	7.21	552	0.109	15.55	83.76	52.63	23101	0.144	52.78	17.52	715	78.73	1.57	19.44	523	10.65	579
14	24690	1.84	158	0.148	7.93	33.44	28.58	8593	0.106	27.18	11.87	392	58.25	0.442	5.49	234	5.42	245
15	61530	8.05	558	0.107	18.13	105.28	54.39	25101	0.09	50.02	16.95	787	80.03	1.76	21.84	540	6.82	595
16	72400	5.32	578	0.122	16.23	103.21	52.41	22654	0.173	53.21	18.43	718	81.35	1.46	18.10	532	11.45	590
min	24690	1.84	61	0.082	7.78	27.68	27.41	7515	0.064	25.18	11.87	372	45.56	0.442	5.49	234	3	245
max	72400	9.51	743	0.29	21.49	294.32	62.19	26533	0.198	60.17	22.62	826	93.38	2.04	25.91	630	27	711
mean	56420	7.10	418	0.12	16.31	111.24	46.64	22260	0.13	49.81	17.53	705	78.74	1.44	17.90	473	11	525
st.dev	15218	2.10	198	0.048	3.65	61.64	10.55	5893	0.042	9.53	3.51	134	12.85	0.395	4.96	122	8	140

Table 3: *EPA guidelines for sediments*

Metal (mg/kg)	Not Polluted	Moderately Polluted	Heavily Polluted	Present Study
As	<3	3-8	>8	1.84-9.51
Cd	-	-	>6	0.089-0.29
Co	-	-	-	7.78-21.49
Cr	<25	25-75	>75	27.68-294
Cu	<25	25-50	>50	27.41-62.19
Ni	<20	20-50	>50	25.18-60.17
Pb	<40	40-60	>60	11.87-22.62
Zn	<90	90-200	>200	45.56-93.38
Mn	<300	300-500	>500	372-826
Hg	<1	>-1	>1	0.064-0.198
Ba	<20	20-60	>60	61-743
Fe	<17000	17000-25000	>25000	7515-26533

Correlation analysis provides an effective way to reveal the relationships between multiple variables and thus have been helpful for understanding the 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, as measured by correlation coefficient as called Pearson's correlation coefficients R are shown in Table 4 [12-17].

Correlations between heavy metals may reflect some information of origin and migration about these elements. For example, high correlations between two heavy metals probably mean these two elements share similar pollution sources or they share analogous transformation and migration processes in the certain circumstances. Correlation is an effect size and so we can verbally describe the strength of the correlation using the guide that Evans [15] suggests for the absolute value of R: 0.00-0.19 "very weak", 0.20-0.39 "weak", 0.40-0.59 "moderate", 0.60-0.79 "strong", 0.80-1.0 "very strong".

Correlation analysis showed strong (R: 0.60-0.79) and very strong correlation (R: 0.8-1) forming between of groups elements: 1) Al- Ba-Cu-As-Ni-Pb-Zn-U-A-Ra-K, 2) Fe-Cu-Ni-Zn-U-Ra, 3) Co-Fe- Ni-Mn-Zn, 4) Cr-Pb- Zn 5) Hg-Pb-Zn and Ba-Hg at $P < 0.01$ level may be indicate same source.

A weak and very weak relationship between Cd with other components is demonstrated that number of natural sours for these metals is predominant. Relatively strong and very strong relationships Cu, Ni, Zn U and Ra with Al and Fe indicative that lithogenous input are important geochemical phase for this metal; 4) The strong relationships found between Co, Ni, Mn ,Zn and Fe. Since Fe is relatively significantly regressed with Al, it is likely that these metals are manly associated with detrital minerals.

Table 4. *Pearson correlation matrix between different metals, in the sediment of Caspian Sea.*

	Al	Fe	As	Ba	Cd	Co	Cr	Cu	Hg	Mn	Ni	Pb	Zn	U	Tot(A)	Ra	K-40
Al	1.00																
Fe	0.72	1.00															
As	0.66	0.70	1.00														
Ba	0.76	0.70	0.53	1.00													
Cd	0.18	0.13	0.05	0.53	1.00												
Co	0.38	0.83	0.57	0.33	-0.05	1.00											
Cr	-0.17	0.48	0.70	-0.12	-0.26	0.79	1.00										
Cu	0.86	0.79	0.62	0.88	0.41	0.44	-0.05	1.00									
Hg	0.51	0.29	0.27	0.68	0.23	-0.16	-0.37	0.45	1.00								
Mn	0.61	0.85	0.69	0.55	0.15	0.91	0.47	0.64	0.00	1.00							
Ni	0.73	0.95	0.70	0.67	0.21	0.87	0.46	0.78	0.19	0.93	1.00						
Pb	0.78	0.71	0.60	0.83	0.28	0.28	0.60	0.75	0.82	0.45	0.60	1.00					
Zn	0.70	0.93	0.65	0.81	0.27	0.64	0.85	0.82	0.54	0.70	0.85	0.87	1.00				
U	0.71	0.82	0.71	0.74	0.17	0.59	0.19	0.80	0.43	0.68	0.75	0.73	0.79	1.00			
Tot(A)	0.86	0.77	0.67	0.85	0.32	0.37	-0.11	0.87	0.63	0.56	0.70	0.88	0.81	0.86	1.00		
Ra	0.71	0.81	0.71	0.74	0.17	0.59	0.18	0.79	0.43	0.67	0.75	0.74	0.78	1.00	0.86	1.00	
K-40	0.87	0.78	0.69	0.87	0.34	0.39	-0.11	0.89	0.62	0.58	0.72	0.88	0.83	0.85	1.00	0.85	1.00

4. Conclusions

The chemical contamination in the sediments was evaluated by comparison with the sediment quality guideline proposed by USEPA. The present study showed that, pollution with

As is negligible in one site, moderately polluted in eight site and heavily polluted in seven site. In all investigated stations pollution with Cd, Pb and Hg belong to unpolluted class. Station 11 and 14 for Cr, stations 1, 2, 5, 10, 11 and 14 for Cu, and station 4 and 14 for Ni can be considered as moderately polluted. All other stations are heavily polluted with noted elements. Concentration of Zn in stations 4, 6 and 8 were greater than 90 mg/kg, sediment samples from all other stations can be considered as not polluted. All stations are heavily polluted with Ba possible reason for that could be oil production in Caspian Sea. Correlation analysis showed strong (R: 0.60-0.79) and very strong correlation (R: 0.8-1) between following group elements: 1) Al- Ba-Cu-As-Ni-Pb-Zn-U-A-Ra-K, 2) Fe-Cu-Ni-Zn-U-Ra, 3) Co-Fe- Ni-Mn-Zn, 4) Cr-Pb-Zn 5) Hg-Pb-Zn and Ba-Hg ($P < 0.01$ level may be indication of same source).

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ОПРЕДЕЛЕНИЕ КОНЦЕНТРАЦИИ РАДИОНУКЛИДОВ И МЕТАЛЛОВ В ДОННЫХ ОТЛОЖЕНИЯХ КАСПИЙСКОГО МОРЯ

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Резюме: Для получения информации об источниках и степени загрязнениях, распределение тяжелых металлов и природных радионуклидов U-238 (Ra-226), Th-232 (Ra-228) и K-40 в осадках Каспийского моря было исследовано. Было взято и проанализировано 16 образцов осадков для основных элементов (Al, Fe), тяжелых металлов (As, Ba, Cd, Co, Cr, Cu, Hg, Mn, Ni, Pb, Zn) и для природных радионуклидов. Средние концентрации этих тяжелых металлов в том же месте отбора образцов соответствовали порядку Al> Fe> Mn> Ba> Cr> Zn> Ni> Cu> Pb> Co> As> U> Hg> Cd.

Химическое загрязнение в отложениях оценивали по сравнению с рекомендациями по качеству осадка, предложенными USEPA. Анализ коэффициентов корреляции Пирсона было применено для определенным концентрациям тяжелых металлов и радионуклидов исследуемых образцов поверхностных осадков.

Ключевые слова: радионуклиды, тяжелые металлы, Каспийское море, Азербайджан

XƏZƏR DƏNİZİNDƏN GÖTÜRÜLMÜŞ DİB ÇÖKÜNTÜSÜ NÜMUNƏLƏRİNDƏ RADİONUKLİDLƏRİN VƏ METALLARIN KONSENTRASİYASININ TƏYİNİ

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Xülasə: Xəzər dənizindən götürülmüş dib çöküntüsü nümunələrində çirklənmə dərəcəsi və mənbələri haqqında məlumat əldə etmək üçün ağır metalların və təbii mənşəli radionuklidlərin (U-238 (Ra-226), Th-232 (Ra-228), K-40) paylanması tədqiq edilmişdir. 16 dib çöküntüsü nümunəsi götürülmüş və bu nümunələrdə əsas elementlər (Al, Fe), ağır metallar (As, Ba, Cd, Co, Cr, Cu, Hg, Mn, Ni, Pb, Zn) və təbii mənşəli radionuklidlər təyin edilmişdir. Eyni nümunə götürmə nöqtəsində tədqiq edilən ağır metalların ortalama konsentrasiyaları üçün Al> Fe> Mn> Ba> Cr> Zn> Ni> Cu> Pb> Co> As> U> Hg> Cd düzülüşü ödənilir.

Çöküntülərdəki kimyəvi çirklənmə USEPA tərəfindən təklif olunan çöküntü keyfiyyəti ilə müqayisədə qiymətləndirilmişdir. Araşdırılmış çöküntü nümunələrində müəyyən edilmiş ağır metal və radionuklid konsentrasiyalarına Pearson korrelyasiya əmsalları analizi tətbiq edilmişdir.

Açar sözlər: radionuklidlər, ağir metallar, Xəzər dənizi, Azərbaycan