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Assessment of heavy metal contamination in the water of Dayet Er-Roumi Lake (Morocco)

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ABSTRACT

The Dayet Er-Roumi Lake is one of the main Lakes in Morocco (SIBE), it's the only permanent continental Lake of low altitude. In recent decades, a significant deterioration in water quality generated by various pollutant discharges has been identified. The present study aimed to evaluate the metal contamination, assess the pollution degree, and quantify Dayet Er-Roumi's water quality through spatio-temporal monitoring of several metallic trace elements: Fe, Pb, Zn, Mn, Al, Cr, and Ba during 2019 in four seasons. Seven different sampling points were selected along the study area. The results revealed that the heavy metals concentrations in water varied widely and exhibit fluctuations among different metals during the beginning and end of the present study, the order of abundance of these metals Al > Fe > Zn > Mn > Ba > Pb > Cr. According to the correlation analysis, highly strong positive correlations were observed among some heavy metals (between Al, Ba, Zn, and Fe), suggesting common sources and identical behavior during their transport. The calculated average of Heavy metal Pollution Index (HPI), based on the critical pollution index (100), denote that the lake is suffering from serious metal pollution (HPI = 210.665 > 100), the highest level of HPI was recorded during two seasons: spring (HPI = 651.362) and summer (HPI= 159.945). Overall, the results of HPI show a high degree of contamination indicating serious anthropogenic pollution. The obtained results confirmed that the anthropogenic activities, (agricultural and domestic, etc…) have a considerable impact on the water quality characteristics of the lake.

INTRODUCTION

Surface waters (that represent surface aquatic ecosystems) reveal only 0.6% of the freshwater (**Lévêque, 1996**). These aquatic ecosystems play a decisive role in the life of many plant and animal species, including humans. They are considered as a support for aquatic biodiversity (**Lévêque, 1996a**). In recent years, the contamination of the aquatic environment with heavy metals has become a worldwide problem, owing to its environmental toxicity, longevity, persistence, abundance, and also the ability to bioaccumulate in aquatic ecosystems (**Rainbow, 2007**). The potential sources of metal pollution in the aquatic environment are mainly anthropogenic activities, in fact, numerous heavy metals, are released into the environment, due to rapid population growth, intensive domestic activities, and expansion of industrial production (**Islam *et al*., 2015**). Indiscriminate use of fertilizers and pesticides in agriculture leads to serious environmental problems and deterioration of water quality, it's also threatening living beings (**Kar *et al*., 2008**).

Metals behavior in natural water is up to substrate sediment composition, suspended sediment composition, and water chemistry (**Mohiuddin *et al*., 2012**). During transport, metals may undergo many changes in their speciation due to dissolution, precipitation, sorption, complexation, etc., and that affects these metals behavior and their bioavailability in the lake. Although some metals like Cu, Fe, Mn, Ni, and Zn are essential as micronutrients for life processes of plants and microorganisms, they become harmful passing a certain limit, while many other metals such as lead, nickel, cadmium, mercury, chromium, arsenic, etc., are considered dangerous for the environment even with low concentrations due to their toxic nature (**USFDA, 1993; Commission Regulation, 2001**).

Recently, the wetlands of most countries are facing serious problems of contamination with heavy metals, agrochemicals, and household waste, which degrade water quality. In Morocco, Lake Dayet Er-Roumi, is classified as a site of biological and ecological interest, it is the only permanent natural lake in the Khmisset region. With its ichthyological wealth, this lake plays a very important socio-economic and ecological role. However, fluctuations in the water quality are remarkable; they are mainly linked to the neighboring human activities (agricultural, domestic, and tourist discharges) and climate change.

This lake has attracted scientists attention because of its pollution. Till this day, no scientific research regarding the heavy metal problems in the lake has been conducted. Therefore, the objective of the present study is to evaluate the pollution status, assess the degree of heavy metal contamination, and track the abundance of heavy metals in the lake, taking into consideration the spatial and temporal variations.

MATERIALS AND METHODS

1. Description of the study area

The site Dayet Er-Roumi (33°45'N - 06°12'W) is located on the alluvial coastal plain between Rabat to the West and the Middle Atlas Mountains to the East, situated on the territory of three rural communes: Ait Ouribel, Ait Ouahi, and Ait Houderrane. It is characterized by a semi-arid climate with a maximum summer temperature of 38°C and a minimum winter temperature of 7°C and a Mediterranean rainy regime. It has 2 km long and 400m to 700 m wide, with an area of about 90 ha and a maximum depth of 13.5 m in the center (**Mabouhi, 2006**), This lake is fed by groundwater and two tributaries (Fig. 1).

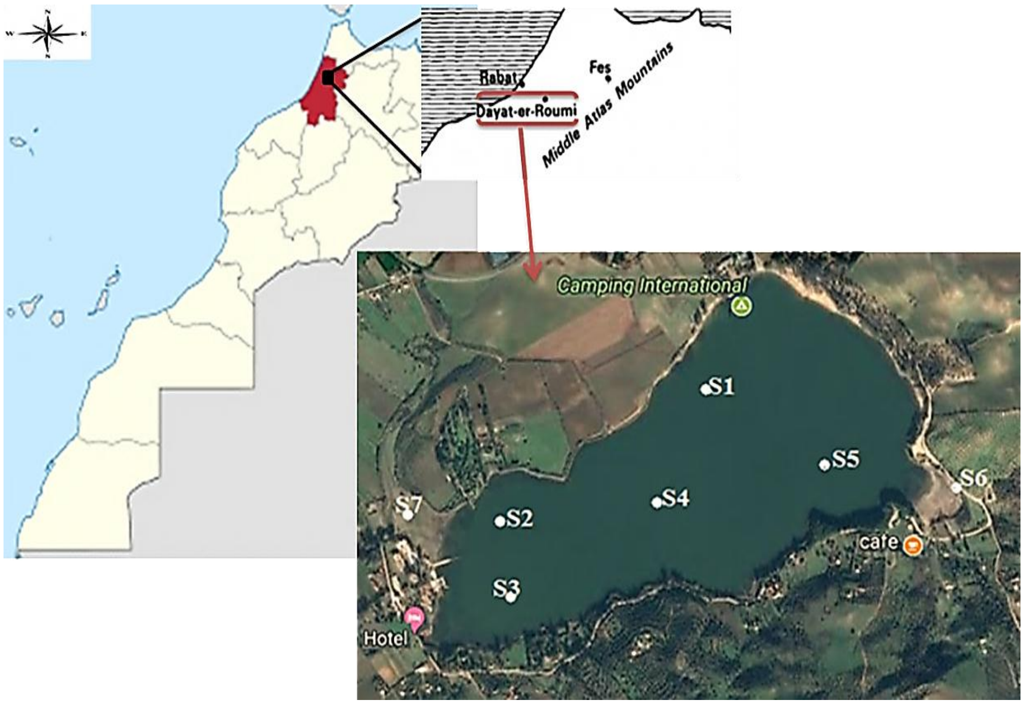

Figure 1. Study area and location of sampling stations.

2. Sample collection and preservation

The choice of sampling sites was made according to the juxtaposed activities; in total, there are seven sampling stations (S1-S7), five points (S1-S5) in the lake representing the areas that are more likely to be affected by human activities (housing, hotel, etc.) and the other two points (S6 and S7) were chosen to represent the lake's tributaries (Fig. 1 and Table 1).

Table 1. Sampling location in the study area.

Stations	Locality	Geographical Coordinates	
		Latitude	Longitude
S1	station near a camping	N 33°45'5.677"	W 6°11'14.119"
S2	station near a house	N 33°44'45.245"	W 6°11'43.570"
S3	station near a hotel	N 33°44'40.149"	W 6°11'44.795"
S4	in the middle of the lake	N 33°44'48.721"	W 6°11'25.165"
S5	station near a cafe	N 33°44'50.127"	W 6°10'55.772"
S6	a tributary	N 33°44'44.434"	W 6°10'48.048"
S7	a tributary	N 33°44'45.018"	W 6°10'44.604"

Water samples were taken by a Van d'Horn bottle at different depths to obtain a composite sample, the sampling was done once a month during 2019. The samples, stored in PET bottles, were fixed in situ by HNO_3 (pH < 2) to preserve the metals and also to avoid their precipitation, sampling bottles were rinsed with distilled water, not to mention that we rinse them at least three times with the sampled water before getting the sampling done, then we transport these labeled samples to the laboratory after placing them in a cooler (**ISO 5667-3, 2012**).

3. Sample preparation and analysis

To avoid the contamination of our samples, we used gloves and lab coats, before starting the manipulation, the Teflon tube and polypropylene containers were cleaned, soaked in 5% HNO_3 for more than 24h, rinsed with ultra-pure water then dried.

For metal analysis, sample mineralization was carried out in a microwave oven, in an acid medium, this digestion step is important because it limits the interference linked to organic materials, it also makes it possible to limit the losses of certain metals by volatilization. To 45 ml of the sample in a Teflon tube, 5 ml of concentrated HNO_3 (65%) was added, and then the tube was introduced in the microwave oven at a temperature of 190 ° C for 10 min (**EPA, 2007**). The mineralized samples were stored in 50 ml polypropylene tubes at a temperature of 4 ° C.

For the determination of heavy metals, samples were analyzed by using a Microwave Plasma Atomic Emission Spectroscopy (MP-AES). It is important to perform the same operation for the blank sample. We rely on internal and external control using an inter-calibration exercise to verify the validity of the analytical methods (**ISO 15587-2, 2014**).

4. Analysis of the results

For a better results interpretation, we relied on statistical analysis SPSS (ANOVA, PCA, and HCA) and calculation of the Heavy metal Pollution Index HPI.

4.1. Statistical analysis

We used a combination of statistical methods to analyze the results. The first statistical approach was based on using Analysis of Variance (ANOVA), the second one was represented by Principal Component Analysis (PCA), while the third was actually represented by Hierarchical Cluster Analysis (HCA).

ANOVA's analysis is used to test if there's a significant difference or not between averages; it's also used to compare these averages and the relative variance between them. Principal Component Analysis (PCA) is a descriptive multidimensional statistical method that can be used as a tool to assist the interpretation of a data matrix (**Travi and Mudry, 1997**). This method makes it possible to evaluate, synthesize and classify a large number of data, to extract the main factors that are the source of the simultaneous evolution of the variables and their proper relationships (**Biémi, 1992**).

So the PCA is applied to establish relationships between the stations on the first hand and between the different parameters measured on the other and to identify potential pollution and its characteristic elements.

Cluster analysis (CA) was applied to identify groups of samples with similar heavy metal contents (**Panda *et al*., 2006**). It was formulated according to the Ward-algorithmic method, while the rescaled linkage distance was employed to measure the distance between clusters of similar metal contents. R-mode CA was used to determine the association of different water quality parameters and pollutant sources.

4.2. Calculation of Heavy metal Pollution Index (HPI)

Heavy metal Pollution Index (HPI) is a rating technique that provides the composite influence of individual heavy metal on the overall water quality. This method is based on weighted arithmetic quality (**Prasad and Sangita, 2008; Singh and Rakesh, 2016**). In this indexing, weights (wi) between 0 and 1 were assigned for each metal and the critical pollution index value is 100 (**Reza and Singh, 2010; Bhardwaj *et al*., 2017**). The rating is based on the relative importance of individual quality considerations, and Wi is defined as inversely proportional to the recommended standard (Si) for each parameter. The calculation of HPI involves the following steps.

Firstly, the weightage (Wi) calculation of the i^{th} parameter using the formula below:

$$Wi = k / Si$$

Where k is the proportionality constant and Si is the standard permissible value of the i^{th} parameter (adopted surface water quality standards (**Water quality standards, 2002**)).

Secondly and lastly, the calculation of the quality (Qi) rating for each of the heavy metals:

$$Qi = 100 \times Vi / Si$$

Where Qi is the sub-index of the i^{th} parameter, Vi is the monitored value of the i^{th} parameter in mg/l, and Si is the standard or permissible limit for the i^{th} parameter. After getting the result, we convert the concentration of each pollutant into HPI (**Mohan *et al*., 1996**).

$$HPI= \sum_{i=1}^{n} Wi\ Qi \ / \sum_{i=1}^{n} Wi$$

RESULTS

1. Temporal and Spatial distribution of heavy metals in the water

Figure 2 shows the results of Mn and Ba analyses in waters Lake. Mn values are ranged between 0.1 mg/l at station S7 and 0.31 mg/l at station S6. The higher values are recorded during the winter and spring. Concerning barium's concentration, Figure 2 shows low to medium values, these concentrations vary between a minimum value of 0.05mg/l at station S1 during the summer season and a maximum value of 0.115mg/l at station S6 during the autumn season.

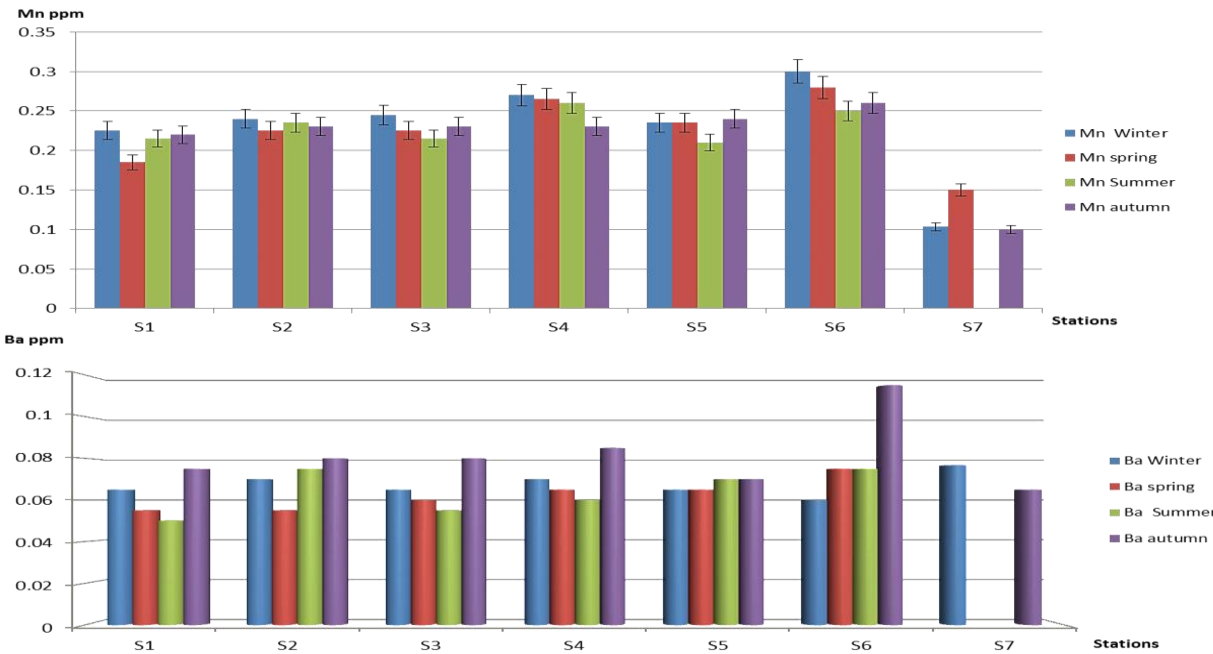

Figure 2. Spatio-temporal variation of manganese and barium.

Figure 3 shows that lead is abundant throughout the lake at different levels between the various sampling points. The average values of Pb vary between 0.01 mg/l recorded at station S2 and 0.115 mg/l at station S4. The higher values are recorded during the spring season, whereas the concentrations recorded were equal to zero during winter and autumn. The spatio-temporal evolution of Fe concentrations in Lake Dayet Er-Roumi's water shows the presence of more or less low levels, with average values varying between 0.02 mg/l at station S2 and 4.93 mg/l revealed at station S6 during summer (Fig. 3).

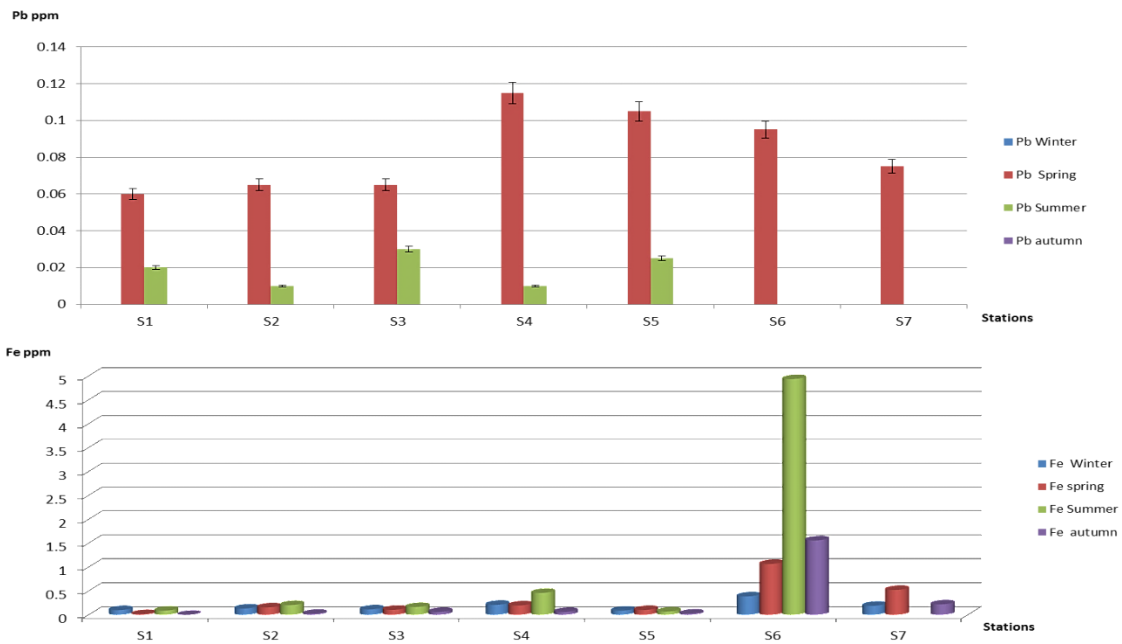

Figure 3. Spatio-temporal variation of lead and iron.

Concerning the concentration of the zinc, it varies between 0.58 mg/ l during spring at station S1 and 1.35mg/l during summer at station S6 (Fig. 4), the most elevated values were recorded during summer and winter (the highest value is represented in the tributary level (S6)). From Figure 4, we noted that the concentrations of aluminum range between 0.085 mg/l at station S5 during autumn and 8.385 mg/l at station S6 during summer.

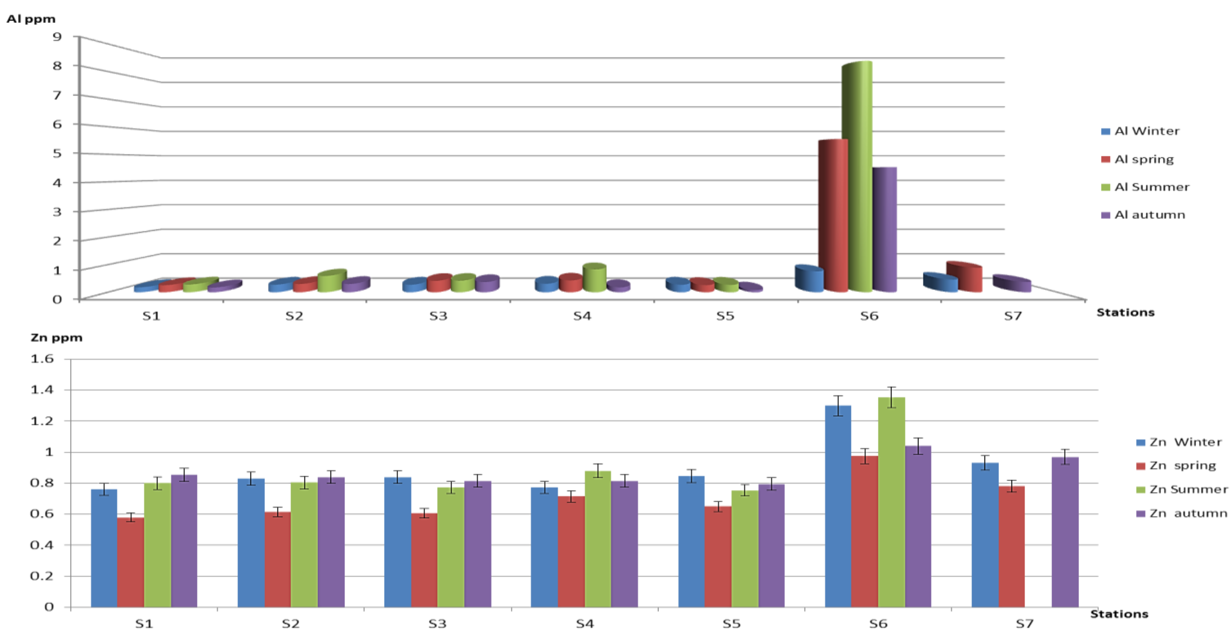

Figure 4. Spatio-temporal variation of aluminum and zinc.

2. Correlation study (ANOVA)

Correlation analysis establishes the relationships between the characteristics of heavy metal in water samples, this method can help us reveal the sources and pathways of heavy metals that generated the observed water compositions, and give us the possibility to identify different elements sources (**Azaza *et al.*, 2011; Parizi and Samani, 2013**).

A high correlation coefficient (near 1 or equal to 1) means a good positive relationship between two variables and when the correlation coefficient is around zero we conclude that there's no relationship between these variables at a significant level of $P < 0.05$ (**Varol and Davraz, 2014**). More precisely, we can say that the parameters with $r > 0.7$ are strongly correlated whereas when r is between 0.5 and 0.7 there is a moderate correlation (**Manish *et al*., 2006**). The correlation matrix of the heavy metals is given in Table 2. Pb shows a moderate negative correlation with Mn ($r = -0.524$). Al shows strong positive correlation with Ba ($r = 0.868$, $P < 0.05$), Zn ($r = 0.964$, $P < 0.01$) and Fe ($r = 0.929$, $P < 0.01$). For Ba, it has a strong positive correlation with Zn ($r = 0.908$, $P < 0.01$) and Fe ($r = 0.886$, $P < 0.01$) (Table 2).

Table 2. Correlation of different heavy metals in water of Lake Dayet Er-Roumi.

	Al	Pb	Mn	Ba	Zn	Fe
Al	1					
Pb	-0.152	1				
Mn	0.414	**-0.5246**	1			
Ba	**0.868***	0.1221	0.2947	1		
Zn	**0.964****	0.0532	0.18966	**0.908****	1	
Fe	**0.929****	0.1203	0.0729	**0.886****	**0.993****	1

*. The correlation is significant at the 0.05 level (bilateral).
**. The correlation is significant at the 0.01 level (bilateral).

3. Principal Component Analysis (PCA)

The analytical data were subjected to statistical analysis using SPSS software. Principal component analysis was used to identify the possible sources of heavy metals and also to distinguish and verify the major potential pollution sources in the study area.

The analysis was performed on a matrix of 7 samples (stations) and 6 variables (Al, Pb, Mn, Ba, Zn, and Fe). Table 3 and Figure 5, give the correlations between the variables, the factors, and the variables projection in the space of the axes F1 and F2.

The analysis of the factorial plan F1 and F2 shows that more than 90.235% is expressed. The F1 axis has a variance of 64.295% that is expressed with aluminum, Barium, zinc, and iron. The F2 axis has a variance of 25.940% and consists of the following variables: lead and Manganese (Table 3 and Fig. 5).

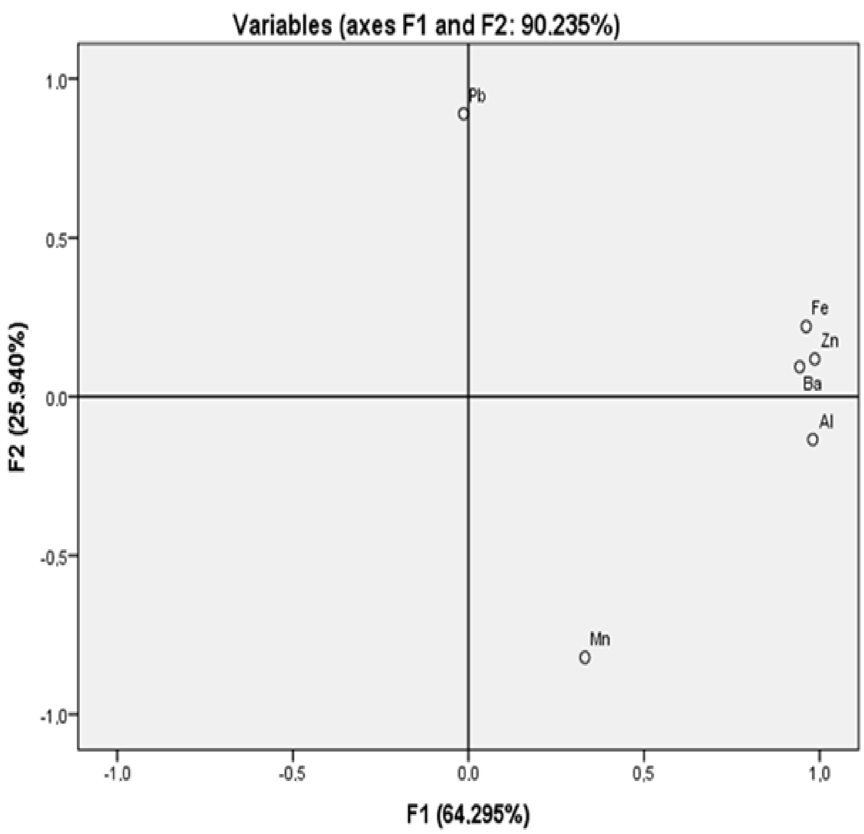

Figure 5. Distribution of metals according to the F1x F2 plan.

Table 3. Correlation of different heavy metals in water of the lake.

	components	
	F1	F2
Al	**0.254**	-0.087
Pb	-0.004	**0.572**
Mn	0.086	**-0.527**
Ba	**0.244**	0.060
Zn	**0.256**	0.076
Fe	**0.249**	0.142

PCA was applied on water samples to assess the spatial distribution of heavy metals, which allows us to differentiate between three groups (Fig. 6). Each group contains a set of stations with a metallic characteristic in each variety.

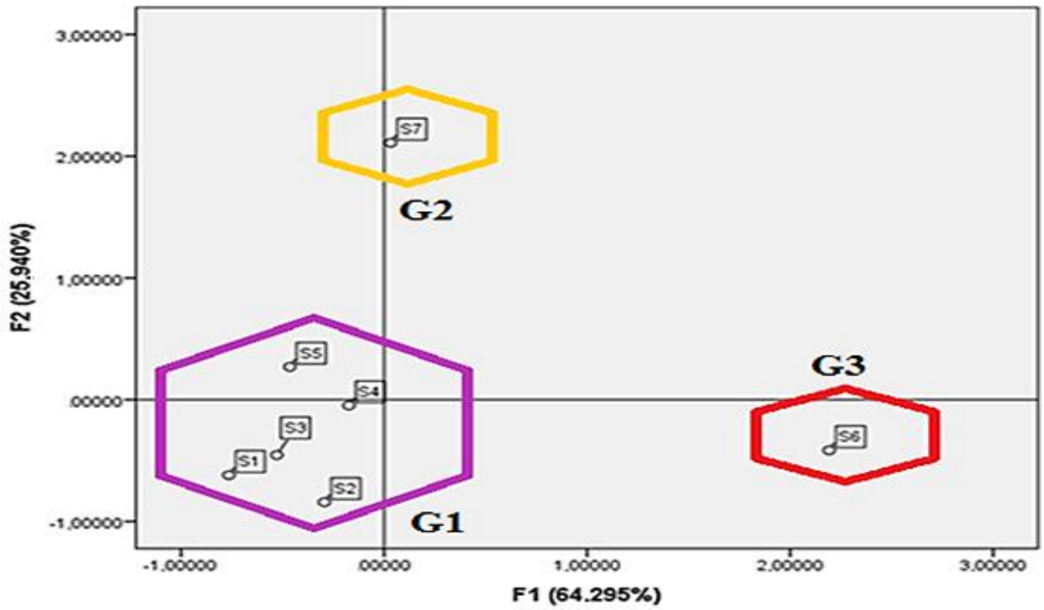

Figure 6. Distribution of stations according to axes F1 and F2.

4. Hierarchical Cluster Analysis

The R-mode HCA was used to determine the relationship among the various heavy metals using Ward's method (Squared Euclidean distance as a measure of similarity). Cluster analysis (CA) grouped the heavy metals into clusters based on similarities within a group and dissimilarities between different groups. Parameters belonging to the same cluster are likely to have originated from a common source. The R-mode CA performed on the samples produced tree clusters based on spatial similarities and dissimilarities (Fig. 7). Cluster 1 includes Al; cluster 2 consists of Fe and Zn, and cluster 3 contains Ba, Mn, and Pb.

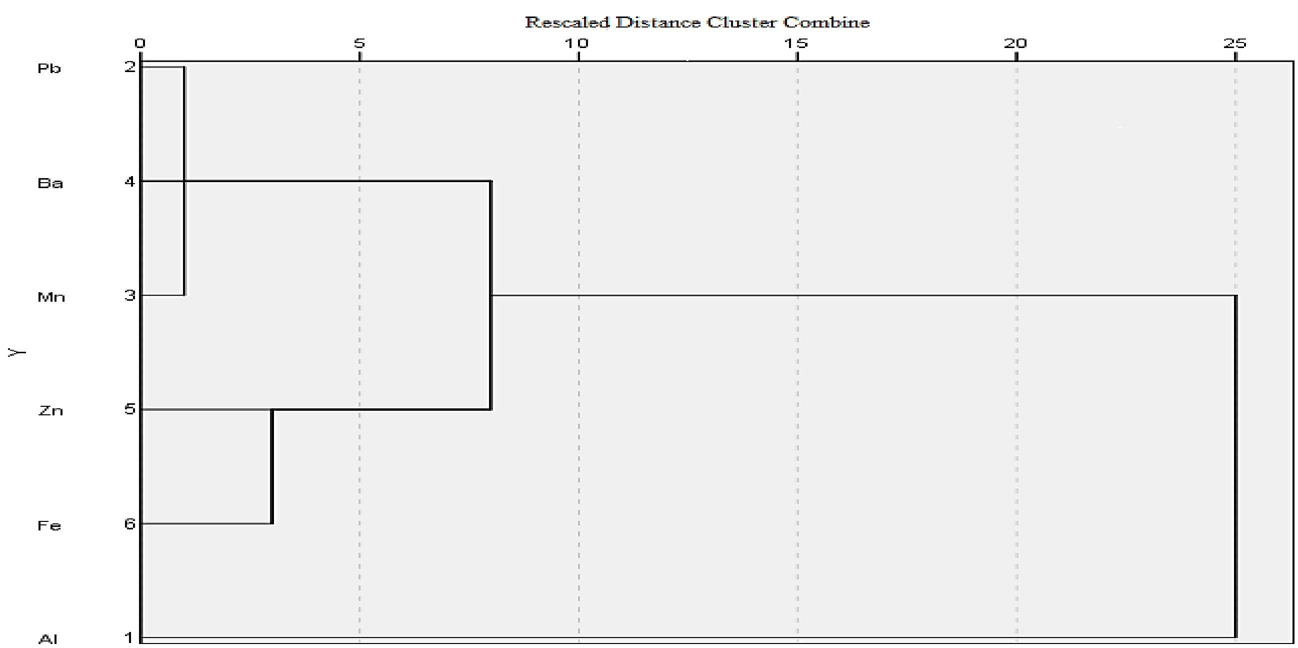

Figure 7. Dendrogram of selected metals in water samples using ward's method.

5. Calculation of Heavy metals Pollution Index

For the heavy metal contamination assessment, the practical indices used is the Heavy metal Pollution Index (HPI), it might also be a very useful tool to evaluate the overall water pollution when it comes to heavy metals. The HPI calculated for water samples we took from Dayet Er-Roumi Lake, was based on the average concentrations of each and every selected metal for all the samplings points in the 4 seasons (Table 4).

The HPI result, 210.665, was way higher than the critical pollution index value that is 100, HPI= 210.665 > 100.

Table 4. Heavy metal pollution calculation for the surface water during the 4 seasons.

Heavy Metals	**Mean concentration (Vi)(mg/L)**				**Highest Permitted values for water(Si) (ppm)**	**Unit weight-age (Wi)**	**Sub index (Qi)**			
	Winter	**Spring**	**Summer**	**Autumn**			**Winter**	**Spring**	**Summer**	**Autumn**
Mn	0.2312	0.2236	0.2309	0.2158	0.5	2	46.239	44.715	46.17	43.143
Al	0.3625	1.1579	1.7959	0.8586	0.2	5	181.07	578.93	897.92	429.286
Pb	0	0.0829	0.0159	0	0.01	100	0	828.58	158.34	0
Ba	0.0674	0.0636	0.0642	0.0815	0.7	1.4288	9.626	9.082	9.17	11.633
Cr	0	0	0	0	0.05	20	0	0	0	0
Zn	0.8972	0.7026	0.895	0.8758	0.5	2	179.43	140.58	179	175.143
Fe	0.1749	0.3093	0.9875	0.2789	0.5	2	34.97	61.85	197.5	55.772
HPI	10.8767	**651.3626**	**159.9455**	20.4727	-	-	-	-	-	-

DISCUSSION

1. Spatio-temporal variation of heavy metal contents

The spatio-temporal evolution of the manganese in Lake Dayet Er-Roumi shows that Mn abounds in all the lake at varying levels between various sampling points (Fig. 2). The higher values are recorded during winter and spring at station S6, this station represents a tributary that crosses agricultural land, and these high values justify the leaching of agricultural soils. However, Mn values in most stations far exceed the recommended standards **(Water quality standards, 2002; Fish water quality standards, 2003)**. These results show that there is a very high level of Mn in the lake due to human activities, not to mention that it causes adverse effects on fish fauna. For barium concentrations in Dayet Er-Roumi Lake, the results (Fig. 2) show more or less low values, however, the highest value was recorded at station S6 (a tributary of the lake) during the autumn season because of the juxtaposed agricultural activities. Barium levels at most stations are below the recommended standard in the four seasons, while a maximum level is located at station S6 (above recommended levels) **(Water quality standards, 2002; Fish water quality standards, 2003)**, these results indicate that Lake Dayet Er-Roumi's tributary (S6) is a source of pollution for fauna as well as flora .

The results show that there is a high concentration of lead in the waters during spring, this augmentation is due to the intensive use of pesticides and fertilizers in the season mentioned before (Fig. 3), while there was a low lead concentration in the summer and an absence of Pb during winter and autumn, that returns to the dilution of Pb concentration. These results are well above the recommended standards **(Water quality standards, 2002; Fish water quality standards, 2003)**. The high levels of lead can be caused by the leaching soils that contain a large amount of fertilizers, also the presence of dissolved lead depends on pH values that are sandwiched between 6 and 8, these values indicate the existence of two soluble Pb species: Pb^{2+} and $Pb(OH)^{+}$ with a dominance of the latter at pH 8 **(Hem and Durum, 1973)**, which is the case in our study area. The results (Figure 3) show that iron concentrations are at most stations below 0.5 mg/l during the four seasons and do not exceed the recommended standards **(Water quality standards, 2002; Fish water quality standards, 2003)**. In addition to this, a maximum concentration has been recorded at station S6 in the summer, at this station (tributary that crosses an agricultural land) we recorded many values that largely exceed the standards **(Water quality standards, 2002; Fish water quality standards, 2003)**, however, this accidental contamination might be caused by the important agricultural activities juxtaposed to the lake, the leaching of agricultural soils can enrich Lake Dayet Er-Roumi's water with iron.

Zn concentrations are very high in all the sampling stations (Fig. 4), they exceed the recommended standards (**Water quality standards, 2002; Fish water quality standards, 2003**).

The reasons of these observed concentrations during the four seasons are the runoff from agricultural land, the detergents, the household waste, and the excessive and uncontrolled use of chemical fertilizers, insecticides, and fungicides, etc. These results clearly show that the Lake Dayet Er-Roumi is under pressure from the local population. For Al, average concentrations were recorded during the four seasons (fig. 4) with a maximum value in summer at station S6 (tributary). This significant concentration of Al shows that the tributary is polluted which subsequently contaminates the waters of the lake. Aluminum monitoring reveals exceeding concentrations for the recommended standards **(Water quality standards, 2002; Fish water quality standards, 2003)**, these high concentrations are due to significant anthropogenic activities around the lake.

We had to mention that during the sampling, the tributary (station S7) was dry during the summer. The chromium analysis shows the absence of this metal during the four seasons in lake water and its tributaries.

2. Correlation between different heavy metals in water of Lake Dayet Er-Roumi

Zn shows a strong positive correlation with Fe ($r = 0.993$, $P < 0.01$), Ba shows a strong positive correlation with Zn ($r = 0.908$, $P < 0.01$) and Al shows a strong positive correlation with Ba ($r = 0.868$, $P < 0.05$). While Mn does not show any significant correlation with other heavy metals (Table 2), which indicates that the distributions of the metals above were not controlled by the same factor. The strong correlations between some heavy metals show the same input sources and a similar geochemical behavior **(Lu *et al*., 2010; Saeedi *et al*., 2012; Pereira *et al*., 2017)**. Therefore, the associations of metals indicate that the water of Dayet Er-Roumi has assimilated various contaminants from chemical fertilizers, pesticides, domestic sewage, and agricultural runoff areas.

The analysis of the factorial plan F1 and F2 (Table 3 and Fig. 5), shows the main pollutants that dominate and interact with each other in the waters of the Lake Dayet Er-Roumi, these pollutants are Aluminum, Barium, Zinc, and Iron.

Applying the ACP to different samples (Fig. 6), we found that there are 3 varieties (groups):

The first group (G1) is represented by 5 stations: S1, S2, S3, S4, and S5 located in the lake. A high concentration of Al, Zn, Fe, and Ba characterizes this variety; this group indicates very loaded water with metallic elements, which may have harmful effects on fauna and flora as well.

The second group (G2) is represented by the seventh station (a tributary), this group is characterized by an average concentration of lead metal. This station is vulnerable to pollution, which probably returns to the uncontrolled use of fertilizers and pesticides because this tributary crosses agricultural land.

The third group (G3) is represented by station S6 (a tributary), this group is characterized by an average concentration of manganese metal; it indicates that the water is heavily charged with Mn. This station is vulnerable to pollution.

According to the ACP, we conclude that the waters of Lake Dayet Er-Roumi and its tributaries are very polluted by heavy metals. This pollution is anthropogenic and caused by domestic waste, phytosanitary products used in agriculture, and also animal excrements in the lake edges...

Concerning the Hierarchical Cluster Analysis (Fig. 7), Cluster 1 contains only Al, this metal appeared as the only member of this cluster because it has the highest number of samples with a concentration that surpasses the maximum standard limit more than any other metal that we analyzed. Al's sources were both lithogenic and anthropogenic. The second cluster includes Zn and Fe, which were derived from anthropogenic input. Cluster 3 contains Mn, Ba, and Pb, which is the weakest of the three clusters and the one with the least concentrations of heavy metals in it.

3. Evaluation of Heavy metal Pollution Index

The calculation of Heavy metal Pollution Index HPI gives a very high value (HPI = 210.665), which was much higher than the critical pollution index value of 100, when a certain value passes 100, the overall pollution level should be considered unacceptable (table 4). In the two seasons, spring (HPI = 651.362) and summer (HPI = 159.945), the HPI calculated is far exceeding the critical index limit of 100. This indicates that the water is critically polluted with heavy metals. This HPI value reflects direct influences of anthropogenic inputs, intensive and uncontrolled use of phytosanitary products, wastewater, and domestic discharges in the lake. The presence of these undesirable elements constitutes a potential danger to human-beings health and the aquatic biodiversity of this area.

CONCLUSION

The Lake Dayet Er-Roumi (Site of Biological and Ecological Interest) has a socioeconomic importance for Morocco in general and the Khmisset region particularly, but this wetland is subject to various sources of anthropogenic pollution. The study carried out on the water quality of the Lake Dayet Er-Roumi during the four seasons of 2019, has revealed a high concentration of different metals: Pb, Zn, Mn, and Al during all seasons (Winter, Spring, Summer, and Autumn), with an average level of Fe and Ba. These concentrations are largely exceeding the recommended standards. The calculation of Heavy metal Pollution Index (HPI) proves the contamination of the Lake; therefore, Dayet Er-Roumi is threatened by pollution and has a good site to accumulate and store the metal pollutants.

This study reflects the direct influences of anthropogenic inputs, intensive and uncontrolled use of phytosanitary products, wastewater, and domestic discharges, etc. On the whole, it seems that the pollution level shows a significant enrichment of these heavy metals which can be critical or even dangerous since they affect the ecological balance of the ecosystem. The Lake Dayet Er-Roumi requires a comprehensive diagnosis for the current situation and rigorous monitoring of its evolution to conserve and maintain this SIBE.

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