PHYSICAL-CHEMICAL CHARACTERIZATION AND HEAVY METALS ASSESSMENT OF WATERS AND SEDIMENTS OF SEBOU WATERSHED (TOP SEBOU, MOROCCO)

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PHYSICAL-CHEMICAL CHARACTERIZATION AND HEAVY METALS ASSESSMENT OF WATERS AND SEDIMENTS OF SEBOU WATERSHED (TOP SEBOU, MOROCCO)

Abstract

The main purpose of this study was to investigate the impact of human activities, geochemical background and seasons on pollutant pathway. Surface water, groundwater and sediments were assessed to highlight and confirm those impacts. Sixteen physico-chemical parameters were measured (T° , pH, O_2 , salinity, conductivity, BOD5, COD, SM, Cl^- , NO_2^- , NO_3^- , NH_4^+ , TAC, TH, SO_4^{2-} and PO_4^{3-}) and twelve metallic trace elements were analyzed (Ar, Cr, Zn, Mn, Ni, Fe, Al, Cd, Cu, Pb, K and Na). Five sampling campaigns were carried out in 18 sampling points for over one year, between July 2018 and July 2019 in the upstream part at the least studied area in Sebou river watershed. The results of our investigations have showed that the water quality of Sebou river varies between medium and good quality depending on the sampling area. The quality is highly impacted by geochemical background and slightly impacted by seasonal change and by surrounding anthropogenic activities. Those conclusions were confirmed by statistical analysis of the data. Strong positive correlations were noted between the elements in waters and sediments, in particular for sulfates, chlorides, iron, copper, zinc, potassium and barium. In addition to that, negative correlation was noted for cobalt, sodium and aluminum contents of surface water and groundwater. In the meantime, we found that these waters are rich in chlorides 62 to 315 ± 9.45 mg/L, in nitrate 0.79 to 7.45 ± 0.52 mg/L, and sulfate 11 to 245 ± 7.35 mg/L, with high contents of iron, aluminum and sodium respectively 69.79 mg/L, 62.23 mg/L and 149.76 mg/L. Toxic chemicals such as arsenic, lead and cadmium were also assessed but their levels testify to the absence of major metal contamination from humane activities.

Keywords

Keywords : Physical-chemical quality, Heavy metals, contaminants, Hydrology, groundwater, Water resources management.

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Cover Page Footnote

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1. Introduction

Aquatic ecosystems are under growing pressure because of excessive use of agricultural inputs, lack of sanitation installations, irrigation, landfills, wastewater discharges, population growth and economic development [1–3]. Aquatic ecosystems are the main outlet for human activities, in most cases without any prior treatment. Currently, countries are making great efforts to improve the fight against pollution [4]. Studies show that human actions introduce into the environment, many contaminants, among these elements we find the most toxic one for the aquatic environment such as As, Cd, Pb, Al and Hg. The fight against these elements is the interest of several studies on an international level [5–7] and their concentrations in water and sediments highly depend on the surrounding conditions.

Morocco is subject to a high level of pollution in some localities, especially in Morocco's largest river "Sebou". Nutrients and toxic elements from agricultural activities, urban and industrial developments [8] and wild dump leachates [9] are the sources of water pollution. Domestic and industrial waters are usually discharged into rivers and spilled on the land untreated [10,11]. Artisanal workers and mining processes are another sources of these contaminants [12,13] in the Sebou river. The city of Fez is worldwide known for its artisanal workers especially for tanning, and numerous studies [14,15] have monitored levels of chromium, lead and cadmium in Oued Fez. Recently, more studies have concentrated their works downstream of the Sebou river near to the city of Kenitra, on the assessment of water quality and pollution monitoring [16,17].

All these studies demonstrate that surface water and groundwater are highly impacted by anthropic activities. Other research has proven that they are also impacted by the geological bed [18,19] but the most of them have focused their work in the middle and lower part of the Sebou river (towards the city of Fez and Kenitra), due to the significant number of industrial activities in these regions.

Based on our research, the top Sebou river (area of the present study) has never been characterized before and the novelty of this work is to further enrich the knowledge on this aquatic ecosystem and sheds light on pollutant pathways between all the compartments. The top Sebou present a background site located upstream of two major cities, far from any industrial

activity that could impact the quality of its waters. However, agriculture is mainly developed there and is a source of pollution in the region.

In order to achieve this objective, we have carried out several analysis on three matrices: surface water, groundwater (wells and springs) and sediments. We took under consideration seasonality, presence or absence of external impacts such as agriculture, wastewater, wild dump leachates and industry in the surrounding.

2. Material and methods

2.1. Geographic location of the study area

For the choice of sampling sites, a systematic and grid approach was favoured [20]. The choice of the most upstream point (reference point), was chosen for its accessibility and its location upstream of the confluence between, Mahsser river and Guigou river. This confluence gives birth to M'dez stream which stretches over twenty kilometers, before flowing onto Sebou river. The other points were positioned around every 10 km to the Allal El Fassi Dam (Map N°1). We took into account, the presence of anthropogenic impact such as a construction of a dam on M'dez stream (S4), agricultural lands in Guigou region (S1, S2), a sewage reject, a dump, an agglomeration (S7).

2.2. Hydrogeological and climatic framework

The basin has prevailing Mediterranean climate with an oceanic influence. The average rainfall there reaches 750 mm however there is a large variation between the upper Sebou and the heights of the Rif, 400 mm and 1800 mm respectively. The average annual evaporation is quite strong in the basin and varies from 1600 mm on the coasts to 2000 mm inwards. The temperatures oscillate between 20 and 30 °C and are highest in July and August and lowest in January [10].

The study area is located on the tablecloth of the middle Atlas ridges between the Fez Meknes basin in the north and the Middle Atlas in the South. This aquifer is formed by more or less tabular carbonated terrains of the lias (lower Jurassic). It is indeed the domain of limestone or more precisely, of calcareous carbonate rocks and, dolomites [11].

2.3. Experimental protocol

Five sampling campaigns were carried out in accordance with the seasons between, July 2018 and July 2019. 18 points were sampled (Fig. 1) combining surface water (S1, S2, S4, S7, S9, S10), groundwater (S3, S5, S11), springs water (S6, S8) as well as sediments (S1, S2, S4, S6, S7, S9, S10). The samples were analyzed within the National Laboratory of Studies and Pollution Monitoring.

In-situ parameters were measured in the field and in the laboratory by two Multiparameters (HACHLANGE SensiON-NM374 and a CONSORT C3030 type). Water was collected in polyethylene bottles, and transported to the laboratory in electric coolers at a temperature not exceeding 6 °C. The analyses were carried out according to standardized methods and according to ISO 17025 standards (Table 1).

The sediments were taken from the riverbed into a clean polyethylene bags, each station was sampled at around ten points, and then the samples were put together and homogenized to create a representative sample of about 250g. For the analysis of trace metal elements, the samples were taken in polyethylene

plastic bottles and the pH was immediately brought to ≤ 2 by addition of concentrated nitric acid.

3. Results and discussions

3.1. Physico-chemical parameters of water

3.1.1. Temperature and pH

Water temperature is an ecological factor that brings important ecological responses [21]. It acts on the density, the viscosity and the solubility of the gases in the water, in particular of dissolved oxygen, the dissociation of salts, as well as on chemical and biochemical reactions [22]. The ideal water temperatures is between 6° and 12 °C [23]. The temperature of surface waters of Sebou river in its upstream oscillates between 16 and 25 °C. Statistical analysis shows negative correlation between pH values and temperature, i.e. higher temperature tends to reduce pH [24]. The pH value varies between 6.87 and 8.20 ± 0.24 throughout the year, without exceeding the Moroccan grid, which defines the quality of surface water [25] (see Fig. 2). However, these values have an alkaline predominance, which could be explained by the presence of limestone layers from the lower lias in this

Fig. 1. Sampling points on the hydrological map of the Sebou watershed.

Table 1

Parameters, apparatus and methods.

Parameters	Methods	Equipment
Physico-chemical parameters		
Water temperature (C°)	NM 03.7.008	Calibrated Hachlange multi-parameter
pH (pH unity)	NM ISO 10523	
Conductivity (µs/cm)	NM ISO 7888	
Salinity (mg/l)	NM ISO 7888	
COD (mg d'O ₂ /l)	NM 03.7.054	Mineralizer + Titrimetry
BOD 5 (mg d'O ₂ /l)	Internal method	Oxytop and WTW Thermostat Incubator
Dissolved Oxygen	ISO 5814	Multi-parameter Consort
Suspended matter (mg/l)	NM EN 872	Filtration on glass fiber filter + oven
Chlorides (mg/l)	NM ISO 9297	Mohr Method
Nitrites (mg/l)	NM ISO 6777	Molecular absorption spectrometry
Nitrates (mg/l)	NM ISO 7890-1	
Ammonium (mg/l)	NM ISO 7150-1	
Orthophosphates (mg/l)	—	
Calcium hardness (mg/l of CaCO ₃)	—	Titrimetry
Total hardness (mg/l)	—	
Sulfates (mg/l)	NF T 901-040 1986	Analysis kits
Metallic trace elements		
Arsenic, Chromium, Zinc, Manganese, Nickel, Iron, Aluminum, Cadmium, Copper, Lead, Barium, Potassium and Sodium (mg/l)	Internal Methods	Microwave plasma-atomic emission spectrometry MP-AES 4100 Agilent

region [11]. Moreover, values between 6 and 8.5 for pH in the same area were reported before [26].

3.1.2. Suspended matter

The waters of Sebou river are not loaded with suspended particles. Moreover the four campaigns have revealed values between 01 and 81 mg/l ± 11 (Fig. 3), thus classifying the waters of the river of excellent quality [25]. The very high values of suspended matter

in July 2019 campaign, can be explained by the passage of a flood one week before taking samples, which caused the movement of particles from the sediments into the waters.

3.1.3. BOD₅, COD and dissolved oxygen

The values of these three parameters provide information about water oxygenation as well as the presence or absence of organic pollution. It allows the

Fig. 2. Evolution of pH by station and by sampling campaign.

Fig. 3. Evolution of suspended matter by station and by sampling campaign.

evaluation of biodegradable organic matter. Statistical analysis by PCA has shown a negative correlation between, oxygen levels and those of BOD₅, COD as well as, the load of suspended matter in water. Consequently the more turbid the water, the more it is loaded with dissolved organic matter and the lower its oxygen level [22]. The values obtained showed some irregularities, however did not fall out of the thresholds recommended by the Moroccan grid for surface water quality [25].

We noted however that station S4 on Oued M'dez, records the highest rates of BOD₅ and COD, respectively 12 mg of O₂/l $\pm$ 2.4 and 50 mg of O₂/l $\pm$ 8 (Fig. 4). These values can be explained by the construction of M'dez dam. In particular due to the

discharges from the base camp of workers who reside there.

Dissolved oxygen is one of the most useful parameters used to determine water quality. It is very sensitive to pollution and its levels informs us about the degree of self-purification of a water course [24]. Dissolved oxygen values between 4 and 9 mg/l have been reported [7,26] and between 1 and 6 mg/l [11] in Sebou river. For our study, values ranged from 6.05 to 9.04 $\pm$ 0.27, classifying these waters of good quality at all stations and throughout the year (see Fig. 5).

3.1.4. Conductivity, chlorides and sodium

The conductivity measurement makes it possible to evaluate the overall mineralization of water and to

Fig. 4. Evolution of COD by station and by sampling campaign.

Fig. 5. Evolution of dissolved oxygen by station and by sampling campaign.

follow its evolution. The statistical analysis has demonstrated a positive correlation between the concentration of chlorides, sodium, nitrates and the conductivity. The higher the concentration of dissolved ions, the higher is the electrical conductivity [22]. The waters of Allal El Fassi dam (S9, S10) and springs water (S5, S6) are of excellent quality. Values between 600 and 5291 $\mu\text{S}/\text{cm} \pm 105$ were found in surface water and groundwater, characterizing them of poor quality in stations S2, S3 S4, S8, S10 (see Fig. 6). Previous studies reported values between 900 and 2500 $\mu\text{S}/\text{cm}$ in top Sebou [4], and between 230 and 552 $\mu\text{S}/\text{cm}$ downstream [26].

Sodium levels go hand in hand with chloride with a predominance of sodium in groundwater (S3, S8, S11)

and this is explained by the significant mineralization of water as it infiltrates [24]. Statistical analysis indicates a positive correlation between these two parameters, and the graphs clearly illustrate this relationship in wells, with a maximum chloride value of 315 $\text{mg}/\text{l} \pm 9.45$ and 149.76 $\text{mg}/\text{l} \pm 4.45$ for sodium at station S11 (see Fig. 7).

The levels of chlorides between 62 and 315 $\text{mg}/\text{l} \pm 9.45$ (Fig. 8) can be explained by the presence of limestone layers from the lower lias in this region, as well as the dissolution of the minerals encountered during the infiltration of water in calcareous carbonate rocks [19]. Moreover, values ranging between 180 and 300 mg/l were cited previously [11], and between 35 and 210 mg/l in another study [26]. These values

Fig. 6. Evolution of conductivity by station and by sampling campaign.

Fig. 7. Evolution of sodium levels by station and by sampling campaign.

qualify water of average quality according to the Moroccan quality grid [25].

3.1.5. Nitrites, nitrates and ammonium

Surface waters record higher nitrites values, however the rates still remain very low. Groundwater is more loaded with nitrates with values between 0.79 and 7.45 mg/l ± 0.52 , yet without exceeding the Moroccan quality standards set at 10 mg/l for excellent quality water. The nitrate level is due to leaching of the soil and its infiltration into deep waters [22]. Ammonium levels are below the detection limit and do not exceed 0.05 mg/l.

3.1.6. Sulfates, calcium hardness and total hardness

Sulfate is one of the major elements of the compounds dissolved in rainwater. Some of it has a natural origin coming from dissolution of sedimentary rocks

such as gypsum which is very present in our study area. Sulfates also have an anthropogenic source following the use of chemical fertilizers and laundry products [27]. The values of our waters are between 11 and 245 mg/l ± 7.35 (Fig. 9) and similar studies report values between 14.03 and 237.74 mg/l [26].

Water hardness is an indicator of its mineralization, which corresponds to the concentration of calcium (Ca^{2+}) and magnesium (Mg^{2+}) ions. This hardness is directly linked to the geological nature of the terrain crossed [22]. The calcium hardness values of investigated waters were between 101 and 301 mg/l ± 9.03 qualifying the surface waters (S1, S2, S4) of the study area as being very hard. Spring water (S5, S6), dam water (S9, S10) and groundwater (S3, S8, S11) values oscillate between 101 and 122 mg/l ± 3.66 characterizing them of being moderately hard.

Fig. 8. Evolution of chlorides levels by station and by sampling campaign.

Fig. 9. Evolution of sulfates by station and by sampling campaign.

PCA analysis has demonstrated a strong positive correlation between these three variables, and this is explained by the simple reason that the calcium ion is obtained mainly by its dissolution in the form of sulfates in the presence of a gypsum substrate [28].

3.2. Heavy metals in waters and sediments

3.2.1. Aluminum, iron, Potassium and Sodium

An evaluation of levels of heavy metals in surface waters of Sebou river showed that these waters are very rich in Iron, Aluminum, Potassium and Sodium. These levels are much higher upstream than downstream, especially for Aluminum and iron see Fig. 10.

Surface water and sediments are more loaded with heavy metal than groundwater. The assessment of groundwater shows that, they are rich in sodium only, presenting therefore a cleaner alternative for drinking water in the region. A maximum value was recorded at station S4 for Iron with $69.79 \text{ mg/l} \pm 3.48$. This high concentration in water is highly linked to the nature and composition of the soil because the analysis of sediments has demonstrated the presence of iron in considerable quantities with values reaching $418.74 \text{ mg/l} \pm 20.93$ at S4 (M'dez bridge). Besides, the previous studies which were interested in the metallic trace elements in the downstream part of the river report iron concentrations in the sediments between

Fig. 10. Levels of heavy metals in the surface waters of Sebou river.

1400 and 2900 mg/l [24], and between 33,330 and 44,190 mg/l [29].

Analysis of the samples for Aluminum was carried out from the third sampling campaign. Al levels reaches 62.23 mg/l $\pm$ 3.11 at station S2 and 346.1 mg/kg $\pm$ 24.22 in the sediments of station S9 see Fig. 11. Values between 9400 and 36,000 mg/kg were reported before [15] for sediments, and negligible values in the waters have been reported in another study [28].

3.2.2. Chromium, zinc and manganese

Chromium is entrained in surface water by runoff [22]. Its presence in station S4 at 160 μ g/l $\pm$ 11 exceeds the recommended limit value of 50 μ g/l. This may be due to the passage of water through agricultural areas, which use phosphate fertilizers, containing impurities such as cadmium and chromium [30,31,32]. As for sediments, chromium levels reach a maximum of 0.46 mg/kg $\pm$ 0.03 at Allal El Fassi dam see Fig. 11.

Very known to be used in tanning leather in the city of Fez, a large number of studies were interested in this element downstream of our study area and values going from 30 to 170 mg/l in the waters [12] have been reported and between 77 and 119 mg/kg in sediments [29], or even between 19.6 and 57.4 mg/kg in another study [7].

On the other hand, Zinc levels are higher in waters than in sediments with values reaching 4.5 mg/l $\pm$ 0.31 in station S3. These rates can be derived from fertilizers used in agriculture. Furthermore, studies undertaken on fertilizers have revealed, contents ranging from 0,14 mg/l for nitrogen-sulfur products, to

5,05 mg/l for products with a main phosphate-potassium component [33]. Similar studies carried out on 24 types of fertilizers have revealed levels varying from 0,6–300 mg/l of Zn [34].

Manganese is a chemical element naturally present in soil and relatively abundant in the waters of the Sebou basin. A maximum value of 2,5 mg/l has been reported before [13].

Our results show higher values in surface waters than in wells and dam, with values between 0,24 mg/l and 1,36 mg/l $\pm$ 0.09 at S4. The water quality grid recommends that if the manganese content is greater than 1 mg/l, the water is classified as poor quality.

3.2.3. Copper and barium

Copper rates in waters and sediments are almost identical, reaching maximum value of 0,16 mg/l $\pm$ 0.01 at S4 and qualifying them of medium quality, according to Moroccan grid of surface water quality. The anthropogenic copper is released into water in particulate form and tends to settle, precipitate or adsorb organic matter, hydrated iron and manganese oxides. It is used to treat aerial parts of crops. Copper sulfate is used in phytosanitary products to treat fruit crops against several species of fungi [36].

Concentrations between 9,2 and 15,6 mg/kg were detected downstream of our study area [15], and also between 43 and 112 mg/kg [13]. Next to these values our study area has very low rates with 0,14 mg/kg as a maximum value in sediments of Allal El Fassi dam (see Fig. 11).

Fig. 11. Levels of heavy metals in Sebou river sediments.

Fig. 12. Scatter plot of sampling stations.

Barium values are acceptable and not exceeding the threshold of $0.46 \text{ mg/l} \pm 0.03$ see Fig. 10. However, we note that surface water is more loaded with barium than groundwater. Barium result from anthropogenic contamination by lubricants and thinners used in drilling waters [37], or following the use of barium-containing pesticide and rodenticide for vector control for potatoes and apples [38]. Almost all surface waters contain barium, in varying concentrations depending on local geology and industrial discharges. Levels varying from 20 to 100 mg/l were mentioned downstream of our study area [12].

3.2.4. Arsenic, cadmium, lead, cobalt and nickel

The region around Fez city, contain 60 mg/kg of Pb [30] and between 14 and 167 mg/kg according to other

studies [35]. Our investigations revealed values below the detection limit, namely 0.0044 mg/l, testifying to the absence of natural or anthropogenic contamination by lead in this area. Also Arsenic and Cadmium contents do not exceed the detection limits namely 0.0016 mg/l and 0.0013 mg/l respectively. Cobalt and nickel were also detected at much lower levels in waters and sediments. The latter do not exceeds 0.24 mg/l in waters for Cobalt and 0.02 mg/l for Nickel.

3.3. Statistical analysis by main component

The data that we analyzed describes the content of surface water, spring water, groundwater and sediments in the Sebou river. Through several quantitative

Fig. 13. Correlation circle for physico-chemical parameters in surface water and in groundwater.

Fig. 14. Correlation circle for metallic elements in water and sediment.

variables grouped into physico-chemical factors and trace metal elements in the waters and sediments. By analyzing some characteristics of central tendency and dispersion of said variables. Thereafter we determined the eigenvalues by using the elbow method in order to carry out the histogram of eigenvalues which will allow us to choose the number of axes providing maximum information (we chose the first 3 axes) (see Fig. 11).

By analyzing the correlations of the physico-chemical and metallic parameters with respect to the sampling stations (Fig. 12) made it possible to highlight two distinct scatter plots, the first one being in

dimension 1, which groups together the stations where the water surface was sampled namely (S1, S2, S4, S7, S9, S10) while the other one located on dimension 2 regroups the stations where groundwater was sampled in particular (S3, S5, S6, S8, S11). This distinction was made following the chemical nature of each of the matrices. This statistically confirms the distinction that exists between surface water and groundwater according to the physico-chemical and metallic parameters that characterize them.

The correlation circles represented above (Fig. 15) highlight the relationships between the variables and their representativeness in the analyzed matrices.

Fig. 15. Circle of correlations of individuals and variables.

Therefore, we notice the close relationship between some physico-chemical parameters in the surface water and groundwater (Fig. 13), as well as the relationships between the presence of heavy metals in water and in sediments (Fig. 14). On the other hand, scatter plot of sampling stations demonstrates the representativeness of the variables compared to the sampling stations. We note that BOD₅, COD, SM and Sulfates are more characteristic of surface waters, while chlorides, nitrates, zinc and sodium are more characteristic of groundwater. Also Mg, K, Cu, Al, Cr and Fe are found in greater quantities in sediments than in waters.

4. Conclusion

In this work, the quality of surface water, groundwater and sediments was assessed in the upstream of Sebou river, area not studied before. In light of this study, which looked at an area never studied before (top Sebou), and in comparison, with the Moroccan grid of surface water quality, the waters of this area extend from medium to good quality with very poor values for certain parameters, in particular for conductivity, iron and Aluminum. The sediments analyzed also revealed significant levels of iron and Aluminum, without exceeding the rates reported by other studies carried out downstream of our study area [3–8].

Principal component analysis highlighted the relations between the parameters analyzed as well as their dispersions compared to the nature of sampled matrices. Thus, positive correlations between surface and groundwater for chlorides, sulfates as well as Fe, Cu, Zn, K and Ba levels have been discovered. Negative correlations were found for Na, Co and Al.

These very close relationships existing between the surrounding conditions, the geochemical backgrounds and the physico-chemical and metallic variables can be used with the aim of highlighting the groups of variables which have the most effect on water quality. However, this will be used in the next step to create a model able to predict the quality of water over time. Accordingly minimizing the costs relating to the number of analysis performed and maximizing the data generated.

Declaration of competing interest

There are no conflicts to declare.

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