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Investigation of the Physico-Chemical Quality of the Wastewater in Fez City (Morocco) Using a Multivariate Statistical Method

Faiçal El Ouadrhiri[1*], Majda Elyemni [2], Abderrazzak Adachi[1], Anouar Hmamou[1], Ahmed Bendaoud[1], Mehdi Chaouch[1], Amal Lahkimi[1]

[1] Laboratory of Engineering, Molecular Organometallic Materials and Environment, Faculty of Sciences Dhar El Mahraz, Sidi Mohamed Ben Abdellah University, Fez, Morocco

[2] Laboratory of Biotechnology, Faculty of Sciences Dhar El Mahraz, Sidi Mohamed Ben Abdellah University, Fez, Morocco

* Corresponding author's e-mail: faical.elouadrhiri@usmba.ac.ma

ABSTRACT

Similar to countries with arid and semi-arid climates, Morocco faces problems of degradation of the quality of its environment and more specifically the quality of groundwater. In this work, we approached the physico-chemical characterization of the raw effluents of three industrial units in Fez city, namely the textile industry (U_1), the copperware industry (U_2), olive oil industry (U_3) chosen for their degree of pollution and their environmental impact, the aim of which is to highlight the degree and nature of the pollution generated by these effluents, and their biodegradability during the winter period January to February of 2018, when the waste water treatment plant (WWTP) is malfunctioning. A set of samplings and measurements of different physico-chemical pollution parameters were carried out such as: temperature, pH, electrical conductivity, turbidity, salinity, chlorides, BOD_5, COD, suspended solids (SS), Ca^{2+}, K^+, as well as heavy metals. The results show that the U_3 effluent is highly loaded in organic matter with high COD (37600 mg O_2/L) and BOD_5 (13000 mg O_2/L), while the U_2 effluent contains very high concentrations of heavy metals (Pb, Ni, Zn, Cu, Cd) 91,8, 71, 55,4, 53, 28 mg/L, respectively. on the other hand, the U_1 effluent is characterized by high SS contents, and concentrations of Ca^{2+}, K^+ exceed Moroccan standards. The COD/BOD_5 ratio shows that U_1 and U_2 effluents are difficult to biodegrade even if their organic loads are low. In the light of these results, it is recommended that these discharges be pre-treated before they are discharged into the liquid sewer system.

Keywords: physico-chemical characterization, wastewater, textile industry, olive mill wastewater (OMW), copperware industry, pollution, COD, BOD_5, heavy metals.

INTRODUCTION

Population growth and the establishment of industries in Morocco have contributed to the deterioration of the quality of aquatic ecosystems (Sebou River) and threaten the health of populations (Koukal et al., 2004; Perrin et al., 2014a). The Sebou Basin, with a surface area of about 40,000 km^2, forms a basin between the Rif in the North, the Middle Atlas and the Meseta in the South, the Fez-Taza corridor in the East and the Atlantic Ocean in the West. One of the most important basins of the kingdom and currently contains a total population of nearly 6 million inhabitants, it is among the most polluted rivers in the entire region according to (ABHS, 2018).

The city of Fez, which has a wastewater treatment plant (WWTP) of the activated sludge type with a medium load with a flow of 155,400 m^3/day and a pollutant load of 64 t/day of BOD_5 designed to minimize the pollutant load of discharges from the sewerage network, includes the effluents of some industrial units that have carried out pre-treatment of their wastewater before discharging it into the sewerage network (RADEEF, 2016). To date, all domestic and industrial discharges transit through the WWTP. However, one phenomenon prevents its proper operation, which is the overload of organic matter (BOD_5 and COD). This situation has a negative impact along the river on the health (problem of waterborne diseases), irrigation, water fertilization, water ingstation and the socio-economic conditions of the inhabitants (Struk-Sokołowska, 2016; Wąsik et al., 2017).

Little research has been carried out in the last decade, revealing that the pollution of the Sebou river is mainly due to industrial effluents. (Perrin et al., 2014b) concluded that the Sebou river is characterized by severe pollution due in particular to tannery effluents from the old Medina of Fez. The vulnerability of the pollution of the Sebou river is due to industrial activities in the region including olive mills, brassware, and textile industries (RADEEF, 2016 and Minister of Energy, Mines and Environment of Morocco 2018). On the other hand, the study (Bounouira et al., 2018) revealed that the sites located near the most urbanized and industrialized areas are severely altered and the main problems are the low dissolved oxygen content, high organic matter content and turbidity as well as the existence of chrome and copper.

As far as we know, no study or investigation in the literature has been made during the period of activity of the olive oil crushing units which lasts 3 to 4 months, which leads to a total dysfunction of the only treatment plant in the city. In this context that, the purpose of our study is to evaluate the physico-chemical quality of three industrial effluents of the city of Fez namely, a textile unit, a copperware unit and an olive oil unit. These units were chosen according to their degree of pollution and their environmental impact (Redouane et al. 2016, J.N. Edokpayi et al., 2017, Jin et al., 2017). To do this, it is essential to first know the physico-chemical characteristics of these effluents, the degree and nature of the pollution generated. Then, a principal component static analysis (PCA) was carried out to reduce the data, which helps us to confirm and present the results in the form of factor maps and finally to establish recommendations for the protection of the Sebou river.

MATERIALS AND METHODS

Selection and presentation of the study area

The history of Fez has always been intimately linked to the presence of water. The city.s wastewater collection network is designed so that the outlet of the WWTP discharges downstream into the River sebou. The study area is located in different industrial districts of the city. Table 1 and Figure 1 illustrate the location, date of sampling and sector of the units studied.

The location of the industrial units that were the subject of this study, was located with Google Maps is shown in Figure 1.

Sampling

In the statistical approach, we followed a simple and instantaneous sampling mode in order to take into account the daily variation of the quality of effluents with the activity of each industrial unit, and to have a good average representativeness of this quality, we proceeded to the realization of the manual and instantaneous samplings at the level of the main collector of the production wastewater which gathers the totality of the raw effluents at the general exit of each plant, during 24 hours of activity and/or each change of color or aspect of effluent and this from 10-02-2018 until 15-02-2018.

Samples were taken using a telescopic cane in 0.5L PET bottles, rinsed well with distilled water and the effluent itself, then sealed tightly without leaving air bubbles. Then, transported quickly to the laboratory and stored at 4 °C to avoid any change in the characteristics of the water (Rodier., 2016).

Analyses and methods

The assessment of the pollution of a raw wastewater is based on the determination of a number of physico-chemical parameters characterizing this wastewater. We were interested in determining in situ the physico-chemical parameters that are indicators of pollution such as: pH, Temperature,

Table 1. The industrial units concerned by the study

Sampling date	Reject unit	Sector	Location
10–02–2018	RU_1	Textile	Industrial district Sidi Brahim – Fès
12–02–2018	RU_2	Copperware	Copperware district Ain Nokbi -Fès
15–02–2018	RU_3	Olive oil unit	Industrial district Doukkarat -Fès

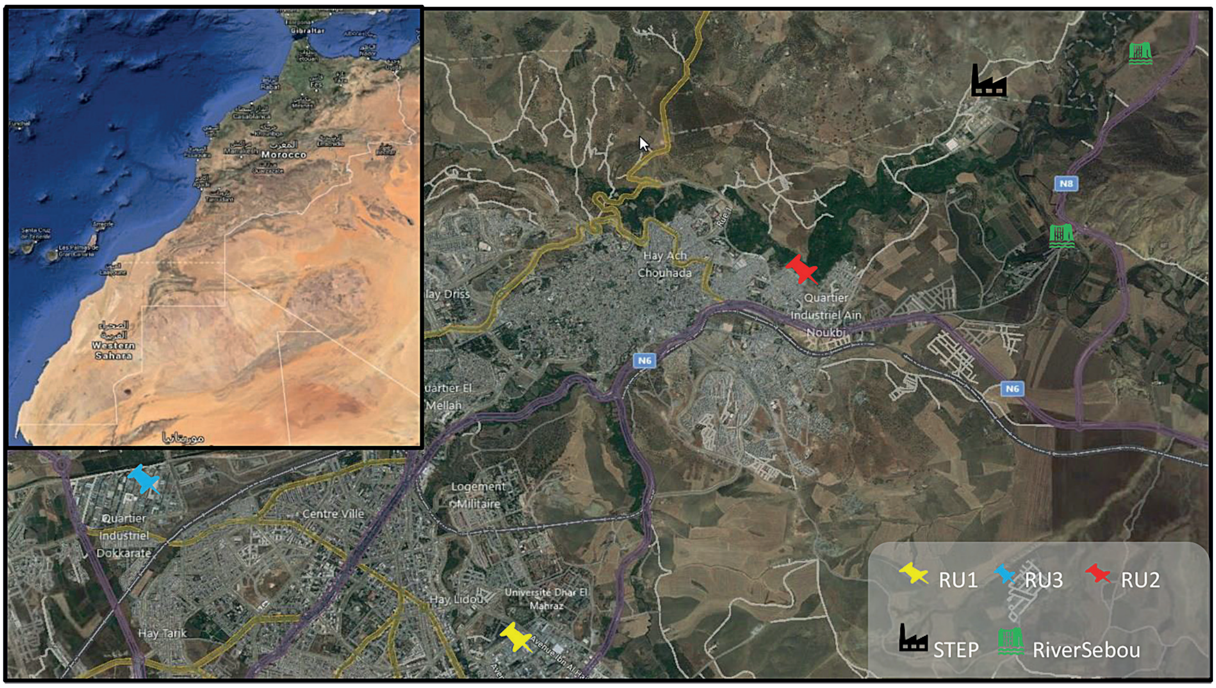

Figure 1. Location of the WWTP and the industrial units concerned by the study

Turbidity, Electrical Conductivity and Salinity. Analyses of heavy metals (Cd, Cr6+, Ag, Pb, Zn, Cu and Ni.), phosphate (PO_4^{3-}), polyphenols, SS, COD, BOD_5, Chlorides, nitrates (NO^{3-}) were also carried out in the laboratory. All the physico-chemical parameters were carried out according to the AFNOR standards decreed by (Rodier., 2016). Inductively Coupled Plasma Atomic Emission Spectrometry (ICP -AES) and/or Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) depending on the concentration range of the samples, at the Fez Research and Innovation Centre (CURIE).

Sample mineralization has been adopted in accordance with the procedures and instructions for sample mineralization for ICP analysis. The analyses were carried out during the period from March to April 2018.

RESULTS AND DISCUSSION

Physico-chemical parameters of effluents

Measurements of pH, temperature, turbidity, electrical conductivity, total suspended solids were recorded over a period of 24 hours and according to the nature and activity of the units concerned are presented in Figure 2.

The pH is a possible indicator of pollution and represents the concentration of hydrogen ions in a solution (Hlavackova, 2005). The Figure 2(a) shows the pH results obtained during 24 hours of continuous activity. The values recorded show significant fluctuations ranging from 3.67 to 10.61 and exceed the standards set by the Moroccan authorities for the discharge of liquid discharges, which are between 5.5 and 8.5 (ABHS, 2018). Indeed, the alkaline pH (10.61) of RU_1 is due to the excessive use of carbonates, H_2O_2 and NaOH in the textile bleaching and washing process. In addition, the acid pH (4.22) of RU_2 is due to the use of acids and metal salts in the washing and treatment of metal surfaces. For RU_3, the very acidic pH (3.79) is due to the acidity of the olive mill wastewater (Rais et al., 2017). These values have been proven by (Benyakhlef et al., 2007) and (Ben Mansour et al., 2011). This fluctuating can have major impacts on aquatic ecosystems. Indeed, they lead to a weakening of the ecosystem by eliminating the species most intolerant to the pH values found (Dupont et al., 2008) and (Benoit-Chabot, 2014). Temperature variations directly influence the biological and biochemical activity of aquatic organisms (Lambert, 2016). Figure 2(b) shows changes in the temperature values of the effluents studied. The values recorded for RU_1, RU_2 do not exceed 30 °C, which is considered as the limit value for direct discharges to the receiving environment (ABHS, 2018) and (RADEEF, 2016). RU_3 has values above the standard with a maximum of 40 °C. These values constitute a thermal pollution risk for the receiving

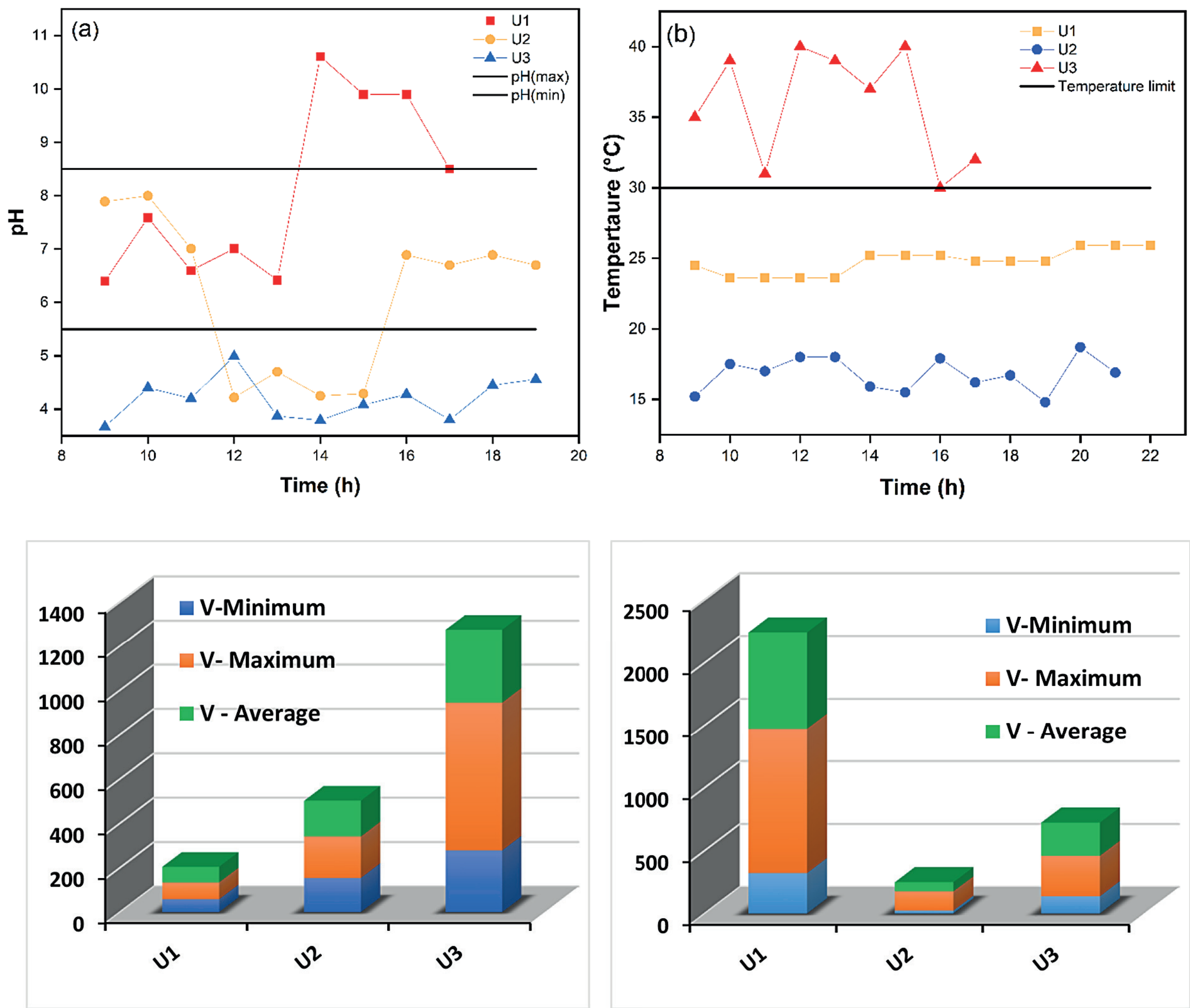

Figure 2. (a) pH variation of the tree industrial units as a function of time; (b) temperature evolution; (c) Average, maximum and minimum turbidity values; (d) Average, maximum and minimum SS values

environment, but are in favor of an acceleration of biological wastewater and sludge treatment processes as they contribute to the increase of degradation kinetics of organic matter (Benyakhlef et al., 2007). Turbidity refers to the content of suspended particles in water that disturb it. In streams it is generally caused by suspended solids and colloidal particles that absorb, scatter or reflect light (Thayer & Boudhraa., 2007). The values found for turbidity range from 70 to 590 NTU (Figure 2(c)). Higher values have been recorded in RU_3 and are due to the small colloidal particles in the copings (Rais et al., 2017). This leads to the conclusion that the studied effluents are moderately turbid and exceed the Moroccan discharge standards. (LeChevallier et al., 1981), (Health Canada, 1997) and (CFPT, 2002) have shown that high turbidity levels are associated with high levels of microorganisms (bacteria, viruses, protozoa), as they bind to particles in the water and can have significant effects on the microbial quality of the water and therefore present a high health risk since this water will be reused in irrigation and others. The analysis of the SS results shows that the studied effluents are characterized by a concentration that varies from 23 mg/L to 804 mg/L. According to these results, it can be noted that the RU_1 largely exceeds the usual standard of Moroccan indirect discharges (600 mg/L). These high concentrations of SS come from the various debris (fibres) contained in the discharge from the textile industry. Our results are close to those found by (Boutayeb et al., 2012), and higher than those found by (Azami Idrissi et al., 2015), (Hachi et al., 2016) and (Kadouche et al., 2018).

Electrical conductivity and salinity

Figure 3 (a, b) shows the results of the electrical conductivity and salinity of the discharges during 24 hours of continuous operation.

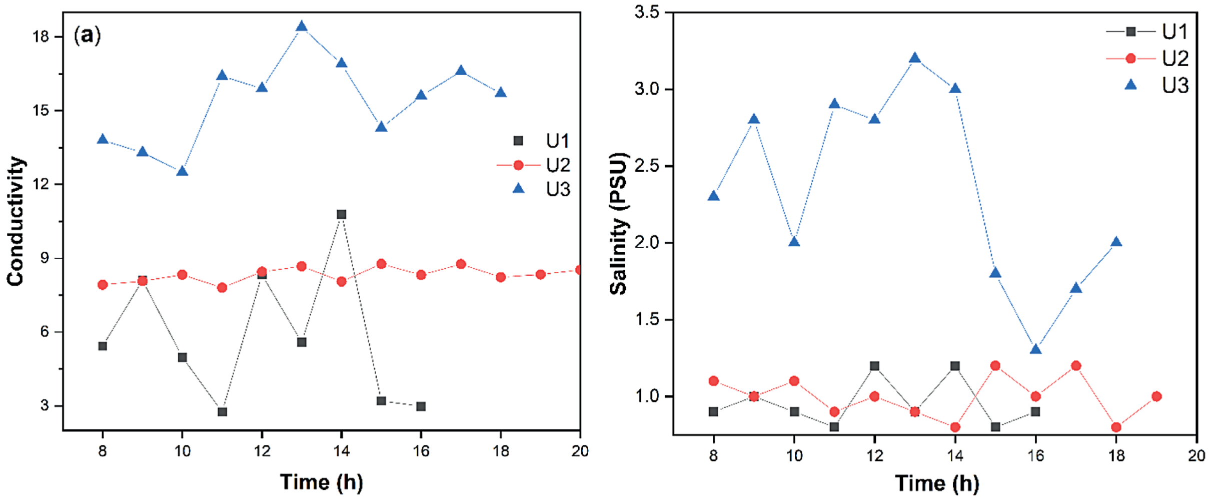

Figure 3. (a) the average, maximum and minimum values of conductivity; (b) salinity

Electrical conductivity is one of the simplest parameters for the diagnosis of water pollution it provides information on the concentration of dissolved salts in a solution (De Sousa et al., 2014). It is a numerical expression of the capacity of water to conduct an electric current. The conductivity of our effluents records average values ranging from 2.8 mS/cm to 18.4 mS/cm. These results show a significant mineralization of these effluents and show an intensive use of salts. The strong mineralization of the effluents recorded at the level of RU_3 with a maximum of 18.6 mS/cm. These values are higher than the 2.7 mS/cm considered as the limit value for discharges. The variation in salinity follows the same evolution as that of conductivity. It varies between 0.8 PSU and 39.7 PSU with a maximum of 51 PSU recorded at RU_3. This favours soil salinization (Raine et al., 2007), (Aragüe et al., 2015) and consequently makes the use of this wastewater undesirable in agriculture. (US salinity laboratory, 1955).

COD, BOD_5, chloride, Ca^{2+}, K^+ and heavy metals analysis

COD represents the amount of oxygen consumed, in mg/L, by the chemically oxidizable materials contained in an effluent. The Table 2 show that the COD content of the three effluents studied recorded dramatically high concentrations of

Table 2. Physico-chemical parameters, major ions, and heavy metals in the effluents studied

Parameter	U1	U2	U3	MAV*
COD (mg d'O_2/L)	1200	1220	37600	1200
BOD_5 (mg d'O_2/L)	240	31	13000	500
Chlorure (mg/L)	150	211	5968	1500
Ca^{2+} (mg/L)	10300	230	539	-
K^+ (mg/L)	1920	94	2578	-
Cd (mg/L)	0.09	28	0.25	3
Cr^{6+} (mg/L)	0.03	0.1	0.19	0,1
Cu (mg/L)	0.05	53	0.31	2
Ni (mg/L)	0.07	71	0.11	2
Ag (mg/L)	0.41	1.98	0.28	0,5
Pb (mg/L)	0.09	91.8	0.43	1
Zn (mg/L)	0.32	55.4	3.56	2
Polyphénols (mg/L)	1.1	3.2	7821	-
Phosphate (PO_4^{3-}) (mg/L)	4.6	1.9	871	-
Nitrates (NO_3^-) (mg/L)	2.9	132	82.7	-

Note: *MAV: maximum admissible value.

37600 mg of O_2/L observed at the U3 unit, i.e. 31 times more than the maximum admissible value set in the specifications of the (RADEEF, 2018), and exceed the authorized limits for discharges from the agri-food industry. (BO, 2018). The values recorded in BOD_5 are also well above 500 mg of O_2/L considered as the limit value. These extremely high concentrations in terms of COD and BOD_5 could be explained by the abundance of organic matter, confirming the existence of serious organic pollution according to the pollution standards recommended by (WHO, 2017).

Chlorides also reach extremely high values 5968 mg/L. This is mainly due to the use of salt in the olive oil extraction process. Equivalent results have been obtained by (Esmail et al., 2014).

Watercourses may be contaminated by wastewater and other nitrate-rich wastes (Sirajudeen and Mubashir, 2013). The maximum concentration was 132 mg/L recorded in RU_2, while the minimum concentration of 2.9 mg/L is recorded in RU_1. These high nitrate concentrations that exceed too far the pollution standards recommended by (WHO, 2017) are due to the excessive use of metal salts from pickling, degreasing, treatment or rinsing tanks (Valérie Laforest, 1999).

Several heavy metals are present in wastewater and industrial discharges depending on their origin. Even at low concentrations, their ecological and health impact can be significant (Bélanger, 2009). The results obtained show that the RU_1 and RU_3 do not exceed the admissible limits for heavy metals, however, the RU_2 of copper, lead, zinc, copper and cadmium are highly charged. This release can constitute a real threat to the environment and the receiving environment according to (Aranguren, 2008) and (Keumean et al, 2013) because of the accumulation and mobility of metals. Our results are slightly lower than those found by (Hayzoun, 2015).

Principal component analysis (PCA)

Principal Component Analysis (PCA) is an extremely powerful tool for synthesizing information, which is very useful when there is a large amount of quantitative data to be processed and interpreted (Guerrien, 2003), (Jollife & Cadima, 2016).

In order to understand the interactions between the 24 physico-chemical parameters in pairs, we studied their correlation through the use of covariance or Pearson correlation. The value of a Pearson correlation can range from 0.00 (no correlation) to 1.00 (perfect correlation). Specifically, it can be said that parameters showing r>0.7 are considered highly correlated, whereas when r has a value between 0.5 and 0.7 a moderate value of (Helena et al. 2000).

In this study, the physico-chemical data (pH, temperature, COD, BOD_5, Turbidity, electrical conductivity, salinity, chloride, polyphenols, PO_4^{3-}, NO^{3-} Ca, Na, K, Ag, Pb, Cr, Cd, Cu, Ni, Zn) were apprehended as gradients synthesizing several parameters and not as separate parameters, which justifies the use of multivariate analysis.

The link between all the variables taken in pairs and the correlation coefficients between these different variables gives the correlation matrix, calculated by the SPSS IBM Statistic v25 software and the interpretation is made according to the order of appearance of the results.

Kaiser's criterion (Kaiser, 1960) leads us to select two components, explaining 100.00% of the total inertia of the cloud. The table 3 show that the two components taken into consideration to describe the correlations between the chemical variables and the units concerned, hold 100% of the total inertia with respectively 63.60% for the F1 axis, and 36.40% for the F2 axis which is significant.

Examination of the data relating to the correlation matrix (Table S4) using the classical orthogonal rotation method Varimax (Rakotomalala, 2012) with Kaiser normalization between the physico-chemical variables and the correlation circle of the F1-F2 plane (Figures 4a, b)

Revealed that:

- Salinity, electrical conductivity, chloride, turbidity, COD, BOD_5 and polyphenol are strongly positively correlated with the F1 axis. This axis by its positive pole includes the unit U3. Also, the pH and the calcium content show a strong correlation towards the negative pole of the F1 axis which justifies that RU_3 is well characterized by organic pollution.
- The F2 axis is well correlated with the elements Pb, Ni, Zn, Cd, Ag, Cu and nitrate towards its positive pole. The latter includes U_2 which is characterized by mineral pollution parameters.

Table 3. Initial eigenvalues and percentage of variance for the main components

Total variance explained			
Component	Initial eigen values		
	Total	% de la variance	% cumulé
C1	12,721	63.605	63.605
C2	7,279	36.395	100.00

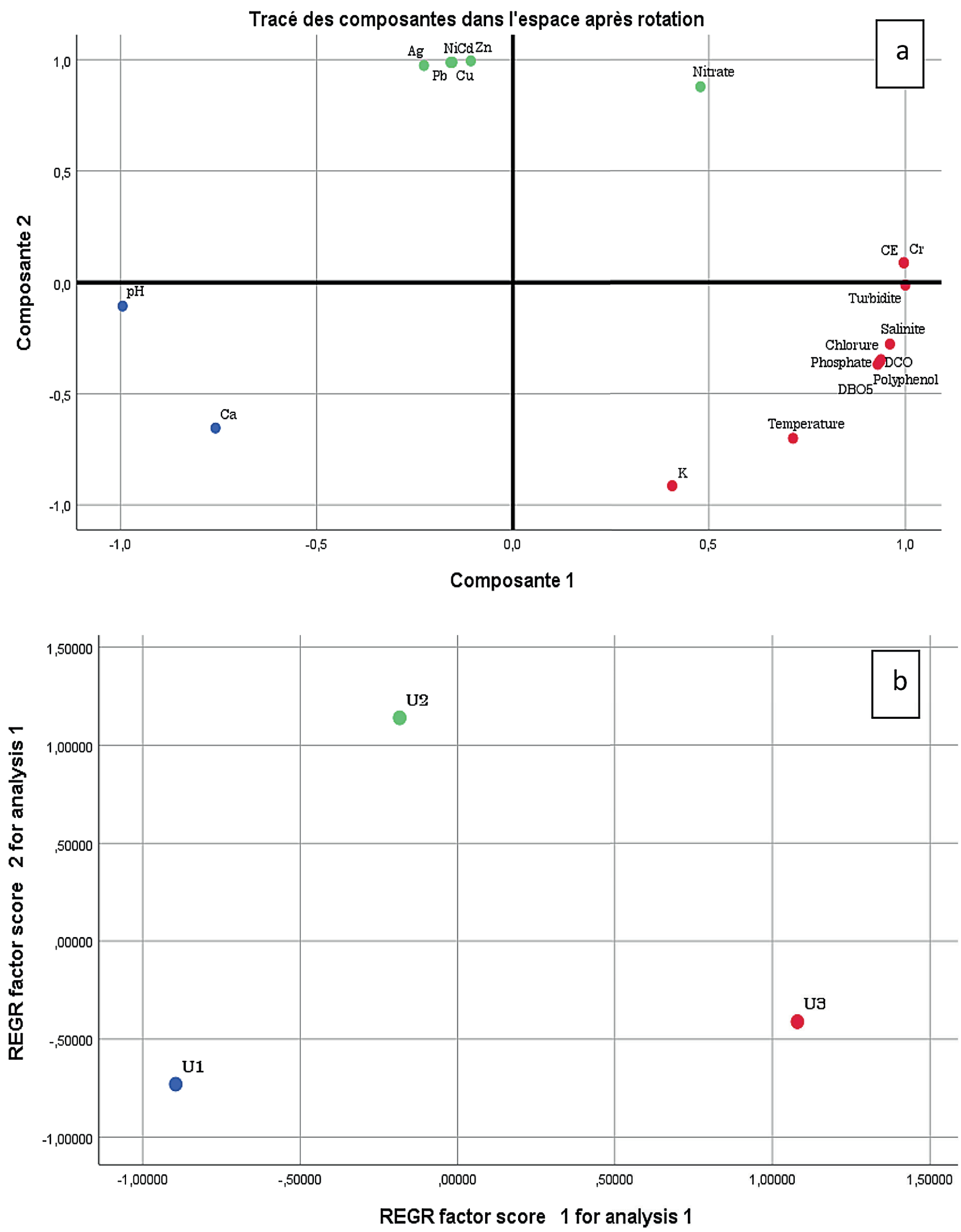

Figure 4. (a) correlation circle of the F1-F2 plane; (b) Factor map of units on the plane (F1×F2)

In conclusion, the PCA has allowed us to compress a considerable amount of information in order to extract the essential information, i.e. U3 is well correlated with organic pollution parameters, however and U_2 and U_1 correlate well with mineral pollution parameters.

The evaluation of the organic load

The calculation of the ratios BOD_5/CDO, SS/BOD_5, the biodegradability index (COD/BOD_5) and the estimation of the Oxidizable Matter (OM) is essential to have an idea of the degree of pollution and the biodegradability of the organic matter of the industrial effluents studied in order to propose a suitable treatment or recovery method. The index of biodegradability is expressed by a coefficient K such that, K = COD/BOD_5, is most often used to get an idea of the biodegradability of the effluent, it depends on the nature and origin of the effluent, which requires different treatments according to Metcalf and Eddy Inc. 2003. This ratio is generally less than 2 for an easily biodegradable effluent, between 2 and 4 for a moderately biodegradable effluent, and when the COD/BOD_5 ratio is greater than 4, the effluent is hardly or definitively biodegradable (Alain, 2007). The empirical relationship between oxidizable matter (OM), BOD_5 and COD was calculated according to (Servais & Billen, 2007):

Table 4. Reports of the global parameters of the effluent pollution of the city of Fez

Parameter	U1	U2	U3
(BOD_5/COD)	0.200	0.025	0.346
(MES/BOD_5)	3.192	2.323	0.020
(COD/ BOD_5)	5.0	39.4	2.9
Oxydable matter (mg/L)	560	427	21200

$$\mathrm{OM} = (2 \times \mathrm{BOD5} + \mathrm{COD})\ /\ 3 \quad (1)$$

Table 4 shows the ratios of the overall pollution parameters generated by the three units. It can clearly be observed that the degree of biochemical degradation (BOD_5/COD) is relatively high and varies from one unit to another, with a maximum value of (0.346) recorded in the unit U_3. The estimate of the oxidizable matter which is of the order of 21,200 mg/L and an average SS/BOD_5 ratio of 0.02 confirm that RU_3 is highly charged in organic matter. This load makes these effluents rather unstable, i.e. they will quickly evolve towards digested forms with release of odours. Our results are in agreement with those reported by (Belghiti et al., 2009), (Hachi et al., 2016) and (Khyati et al., 2004).

The index of biodegradability of the unit U_3 is between 1 and 3, so we can say that the load of organic matter in this effluent is easily biodegradable whereas the units U_1 and U_2 record values much higher than 3, are then very difficult to biodegrade confirmed by the low contents of oxidizable matter. All the parameters analyzed highlight a situation that is certainly worrying even if the evaluation of their contributions would require a rigorous monitoring programme with a higher sampling frequency. In view of the results found, questions remain in relation to the volume of discharges on the one hand and the bioaccumulation of heavy metals in living organisms (fish and plants) on the other. As part of a long-term control of the risk of pollution, it will be necessary to continue investigations into these discharges, supplementing them by measuring other metals and monitoring the impacts on living organisms in the marine environment.

CONCLUSIONS

This work was carried out with the aim of establishing a diagnosis of the physico-chemical state of three industrial discharges of the city of Fez, namely: Textile discharge (RU_1), copperware industry discharge (RU_2) and olive oil industry discharge (RU_3), during the period when the wastewater treatment plant is malfunctioning.

At the end of this physico-chemical evaluation, samples of effluent from the main wastewater collector of each unit were taken and from these samples several parameters (pH, temperature, COD, BOD_5, turbidity, electrical conductivity, salinity, chloride, polyphenols, PO_4^{3-}, NO^{3-} Ca, Na, K, Ag, Pb, Cr, Cd, Cu, Ni, Zn) were analyzed to predict the physico-chemical quality of these effluents. Based on the analyses, it is concluded that most of the parameters analyzed are extremely high compared to the Moroccan liquid discharge standards and the RADEEF specifications. The highest concentrations in terms of COD and BOD_5 were recorded in the discharge RU_3, however the dinner works effluent RU_2 is highly loaded in heavy metals: Ag, Pb, Cd, Cu, Ni, Zn. These concentrations far exceed the MACs set by the Moroccan authorities.

The direct discharge of the discharge without any treatment in the liquid sewerage network of the city of Fez by the RU_3 olive growing units is probably responsible for the malfunctioning of the WWTP. Therefore, all discharges, whether domestic or industrial, discharged into the RiverSebou during this period without any prior treatment is most likely responsible for the dramatic pollution of this watercourse, even if the evaluation of their respective contributions required a rigorous monitoring programme with a higher sampling frequency.

Within this framework, the collection and treatment of these discharges appear to be quite mandatory, complemented by a control and monitoring programme, in order to minimize the environmental risks associated with the discharge of this wastewater in its raw state into the Sebou river.

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