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Brassware wastewater treatment optimization in the city of Fez with sequencing batch reactor using activated sludge

Laidi Omar*, Merzouki Mohammed, El Karrach Karima, Benlemlih Mohamed
Laboratory of Biotechnology, Dhar El Mahraz Science Faculty, PB: 1796, Atlas, Fez, Morocco.

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**corresponding author E-mail: laidi.omar3@gmail.com ; Tel: 212 06 66 54 49 08*

Abstract
Brassware wastewater is classified as the most dangerous effluent, in particular when it is directly discharged in the receiving environment. The aim of this work is to study the biological pathway treatment of effluents at low and medium organic load with sequencing batch reactor (SBR). The effluents characterization have shown a high concentration of metallic load such as silver (3.05 mg/L), copper (10.64 mg/L), nickel (150.94 mg/L), and organic load with the an average COD of 4000 mg/L. The SBR has been used in a 24 hours cycle for 22 hours of aeration and 2 hours of tailing. The results have shown a significant reduction in COD and BOD_5 for both low and medium applied organic loads. The COD removal rates present 81 and 74%, while the BOD_5 abatement reaches 86 and 81% at low and medium organic load, respectively. The abatements observed for heavy metals such as Ag, Cu and Ni with low and medium load were 32.87, 41.42, 30.98% and 27.77, 34.63, 26.06% respectively. The efficiency SBR results have been affected by the presence of both high heavy metals concentrations and increased organic load in real effluent brassware.

Keywords: effluent brassware, sequencing batch reactor, heavy metals, organic load.

Introduction
Heavy metals are released at high load into the environment due to continued rapid industrialization and have become a major global concern. In Morocco, cadmium, zinc, copper, nickel, lead, mercury and chromium, are often detected in industrial wastewater that come out of metal plating, mining activities, battery manufacturing, oil refining, paint manufacturing, printing and photography pesticide [1], but also of craft activities such as tanneries and brassware. The latter are much more threatening, as their discharges also contain other toxic elements such as organic acids (boric acid) and cyanides. This heavy metal pollution is represented mainly by nickel and in very low amounts of copper and silver that come out mainly of discharges treatment process and rinsing basins with electrolytic surface treatment. The polluting power of effluent brassware is mainly attributed to their high heavy metal content. Accordingly, the effluent direct discharge into the river of Sebou causes physicochemical and biological damage to the aquatic ecosystem.
Currently, several methods have been suggested for effluents treatment with high heavy metals concentrations. Hence, the used techniques such as activated carbon adsorption [2], natural zeolites adsorption [3], surfactants [4], chemical coagulation [5], reverse osmosis [6], nano-filtration [7], ion exchange [8] and ultrafiltration [9] have been studied. Rather, the majority of these processes and techniques are still at early stages and require a high price for their full-scale implementation. Several authors have mentioned that the chemical adsorption is the most effective technique to eliminate heavy metals [2-3], even at very low concentrations [10]. However, the high adsorbents price (activated carbon and others) is considered the main obstacle to its industrial application. From an economic standpoint, it is not possible to use activated carbon for industrial wastewater treatment with high heavy metals concentrations [11].
For this purpose, efforts have been focused on the research of cheaper and performing effective techniques with low cost and without problems and damaging in industrial scale. Thus, the potential purifying power of microorganisms has been considered as an effective cost in wastewater treatment with high concentrations of heavy metals. Within this framework, several research have been conducted to examine bioadsorption removal of heavy metals using a passive linking process with micro-organisms such as bacteria, fungi and yeasts [12,13-14]. Moreover, several authors have been interested to the removal metals with batch stirred bioreactor, Pb (II) and Cu (II) using *Bacillus cereus* [15], Cr (VI) *Bacillus licheniformis* [16], Ni (III) and Cd (II) *Escherichia coli*

[17], Cr (III), Cr (VI), Cd (III) Zn (II) and Cu (II) *Pseudomonas aeruginosa*, finally Cd (II) and Cr (VI) with *Pseudomonas sp*. [18].
Thus, the aim of this work is the optimization of brassware wastewater treatment with high heavy metals load by an adapted microbial consortium in a sequencing batch reactor (SBR). Tow organic load were applied (low and medium load), in order to obtain a treated effluent that meets the Moroccan rejection standards.

2. Material and methods

2.1. Effluent brassware

The brassware wastewater from DAR EL KANAR located in the old medina of Fez city have been collected and studied in this work. Specialized in surface treatment, handling nickel and silver plating of metal objects (the monthly amounts are, Nickel sulfate:8 kg, Chloride Nickel: 4 kg and 1 Kg of boric acid). Three different water basins are recommended for degreasing operation, nickel and silver treatment. Wastewater has been collected once a week, directly from brassware before discharging in the sewer. Water from degreasing, silver plating and nickel plating were separately brought to the laboratory, stored at a temperature of 4°C and mixed with equal amount in the laboratory before their treatment with SBR.

2.2. Brassware effluent characterizations

The pH was measured with a pH-meter, the type of electrode was Senti X 22. Dissolved oxygen has been measured with an oximeter type WTW OXI 315i. The electrical conductivity was measured using conductivity meter ORION based type and the results are obtained at 25°C in mS/cm. Suspended solids (SS), dry volatile matters (DVM), nitrogen ions forms such as nitrates (NO_3^-), nitrites (NO_2^-), ammonium (NH_4^+), and orthophosphates (PO_4^{3-}) were determined by colorimetric assay according to [19] experimental protocols.
Chemical oxygen demand (COD) was determined according to AFNOR T90-101 [19] norms. Biological oxygen demand (BOD_5) was determined by means of an instrumental method using an Oxi Top IS6 [19] BOD meter.
Heavy metals determination has been carried out with atomic absorption spectroscopy in Jobin Yvon Horriba device type [19].

2.3 brassware effluent Treatment through SBR

The SBR system is a Pyrex flat bottom with a capacity of 3 and 4 liters net volume. To ensure the effluent supply and withdrawal, plastic tubes are inserted at the reactor top and connected to peristaltic pumps (7554-95 Master flex L/S). Aeration is provided by means of snorkels TÜBAS FH 255-2050C compressor type. The reactor agitation system was magnetic operating with a succession of 24h cycles: 10min for a supply phase with the effluent, 21h 40min for the reaction phase (aeration and agitation), 2h for decanting phase and finally 10 min for the withdrawal phase.
The SBR supply volumes based on the organic load are shown in Table 1.

Table 1: SBR supply volumes based on the applied organic load.

	Low load	Medium load
Supply volume (ml)	225	525
Mixed liquor volume (ml)	2275	1975

Figure 1 shows the operating principle of the SBR bioreactor cycle and the cycle used. Divided into several stages: supply, biological response, mixed liquor purging, decanting and withdrawal ; the mixed liquor purging occurred before decanting in order to control the sludge age.

3. Results and Discussion

3.1- Physico-chemical and metal characterization of effluent brassware

The brassware effluent characterization has focused on the determination of the average physical, chemical and metal values known as an indicator of water pollution (Table 2 and Table 3).

3.1.1- Physical and chemical characterization

Table 2 shows the analytical results of brassware effluent characterization. Table 2 shows that the brassware effluent does not meet indirect discharge standards, especially in terms of COD, suspended solids (SS) and electrical conductivity. The high observed values for each e parameters, may in fact be the result of chemical additions used in the treatment process of metal objects, particularly in degreasing and nickel plating basin where sodium cyanide (NaCN), anhydrous sodium hydroxide (NaOH), sodium carbonate (Na_2CO_3), nickel chloride hexahydrate ($NiCl_2$. $6H_2O$) and nickel sulfate (SO_4 Ni $(H_2O)_6$) have been used excessively (Table 2).
Indeed, low phosphate content (1.67 mg / L) seems logical as there is no detergents use in the rinsing or degreasing basins.

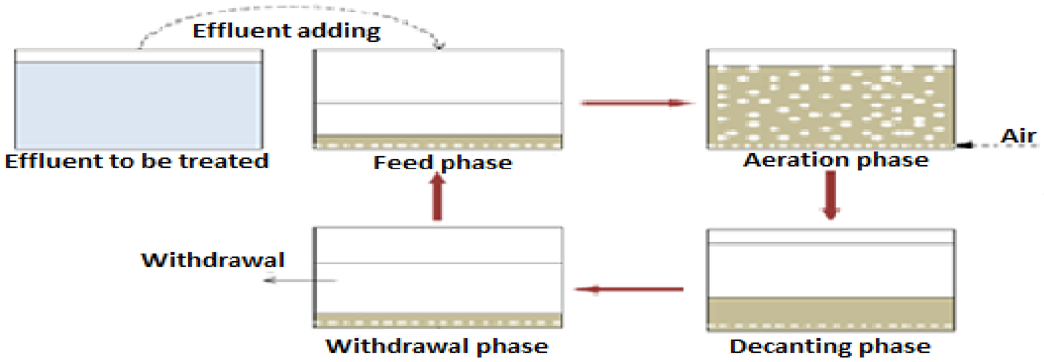


Figure 1: Operating principle of the SBR bioreactor cycle.

Table 2: Physical and chemical characterization of the brassware effluent.

	Average values	Indirect discharge standards [29]
pH	7.43 ± 0.17	6.5-8.5
Electrical conductivity (µS/cm)	6702.35 ± 3.75	2700
COD (mg d'O2/L)	3999.35 ± 4.19	1000
BOD_5 (mg d'O2/L)	27.98 ± 3.80	500
NO_2^- (mg/L)	0.37 ± 0.38	-
NO_3^- (mg/L)	25.23 ± 2.45	-
NH_4^+ (mg/L)	4.52 ± 0.17	-
PO_4^{3-} (mg/L)	2.08 ± 0.54	10
TSS (mg/L)	796.81 ± 6.39	600

The ratio of BOD_5 to COD provides an opportunity to judge the effluent biodegradability and assess the accurate treatment system. It is below 0. 2. Hence, the brassware effluent would be considered as untreatable by biological means. Our study aim, however, was making best use of the SBR process to treat such effluent by adapting the microbial flora with the brassware effluent.

3.1.2 - Metal characterization

Quantifying heavy metals by inductively coupled plasma has shown that the brassware effluent is characterized by a fairly significant amount of metal load, which widely exceeded Moroccan discharge standards (Table 3).
The main metal pollutants observed are : silver, copper and nickel. These elements resulting from the use of copper salts, nickel chlorides, sulphate nickel and silver cyanide during copper plating, nickel plating and silver plating operations.

Table 3: Heavy metals average concentrations contaminating the brassware effluent.

Heavy metals	Average values (mg / L)	Moroccan direct discharges Standards (mg / L) [29]
Silver (Ag)	3.05	0.1
Cadmium (Cd)	< 0.01	0.2
Cobalt (Co)	0.05	0.5
Total chromium (Cr)	0.20	2
Cuivre total (Cu)	10.64	0.5
Iron (Fe)	< 0.01	3
Nickel (Ni)	151	0.5
Total lead (Pb)	0.03	0.5
Zinc (Zn)	0.14	5

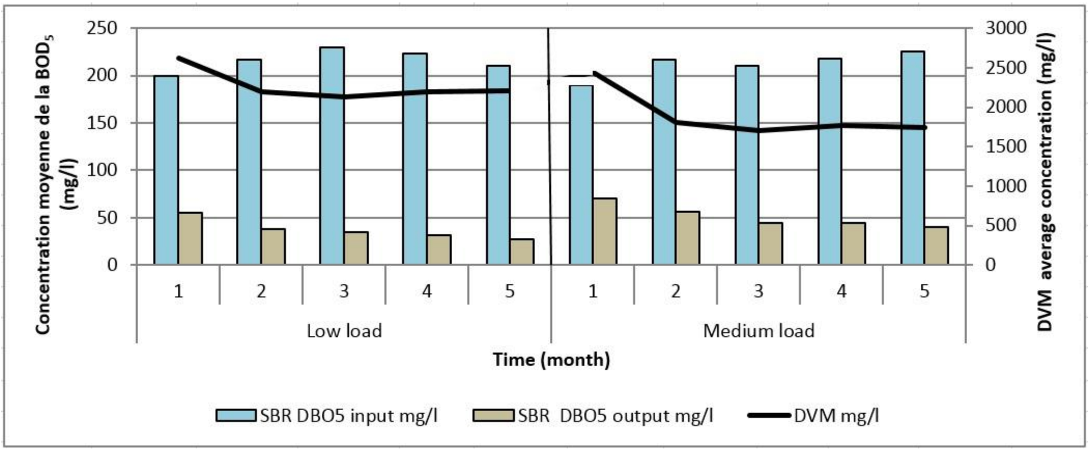
However, the concentrations detected in the effluent are: silver 3.05 mg/L, copper 10.64 mg/L and nickel 150.94 mg/L. These substances are causing a risk for the receiving environment and humans. Indeed, copper is fatal for algae at low concentrations as ppm [20,21-22]. The intake of large doses of copper by man cause severe mucosa irritation, skin corrosion, brain irritation followed by depression ... etc. Almost all nickel chemical forms cause cancer. Long exposure to soluble nickel (1 mg/m^3) can increase the risk of lung cancer [23].

3-2. Brassware effluent treatment by SBR at low and medium organic load.

The brassware effluent treatment has been done by SBR at low and medium load. The treatment efficacy has been assessed by parameters and indicators of pollution monitoring for 5 months.

The COD, BOD_5, heavy metals and other parameters averages show that the abatement rate has been reduced significantly by increasing the organic load. The results of COD and BOD_5 removing are summarized in figure 3.

(a)



(b)

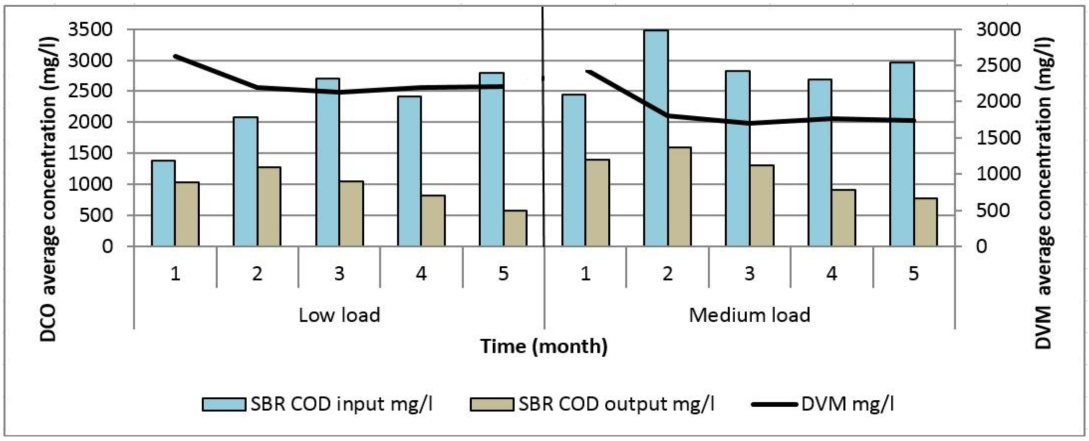


Figure 3: Variations in average total COD and BOD_5 recorded in the input and output SBR cycle with low and medium organic load, (a) BOD_5 and (b) COD.

Average values shown in (Figure 3) obtained during treatment with low load (0.3kg of COD $m^{-3}.d^{-1}$) have shown that there was an important decrease in COD and BOD_5 at the SBR output from the 1th and the 3th month respectively. The concentrations recorded at this point were about 1044 mg/l and 55 mg/l for COD_t and BOD_5 respectively. These values (Figure 3) were obtained when the DVM concentration in the reactor has stabilized around 2.20 g/l ± 0.03 g/l. However, we have found that COD and BOD_5 at the SBR output start decreasing until the 2th and the 3th month when medium load (0.7kg of COD m^{-3} d^{-1}) is applied. The concentrations obtained at the output were 909 m /l for COD and 56 mg/l for BOD_5, with a DVM value around 1.81 g/l± 0.02 g/l. Shifting from low to medium organic load, the COD and BOD_5 abatement rate has successively experienced 80-74% and 87-82% decrease. This decrease was accompanied by a 18% decrease of the concentration of the volatile Matters after an increase in the organic load [24], which explains the COD and BOD_5 decrease. This could be due to the presence of inhibitors, in high concentration in cases of medium load, which may affect the

biomass treatment performance, such as the non-biodegradable heavy metals [25,27-28]. The heterotrophic microorganisms from sludge for COD degradation and assimilation are inhibited by the presence of high concentrations of heavy metals contained in the brassware effluent [Table 3]. Nevertheless, the concentrations obtained in terms of COD and BOD_5 removal have shown that the treated effluent meets the discharge standards without any environmental impact [29].
Evolution of NH_4^+, NO_2^-, NO_3^- concentrations based on DVM recorded at the input and the output of each SBR cycle at low and medium load is shown in Figure 4.

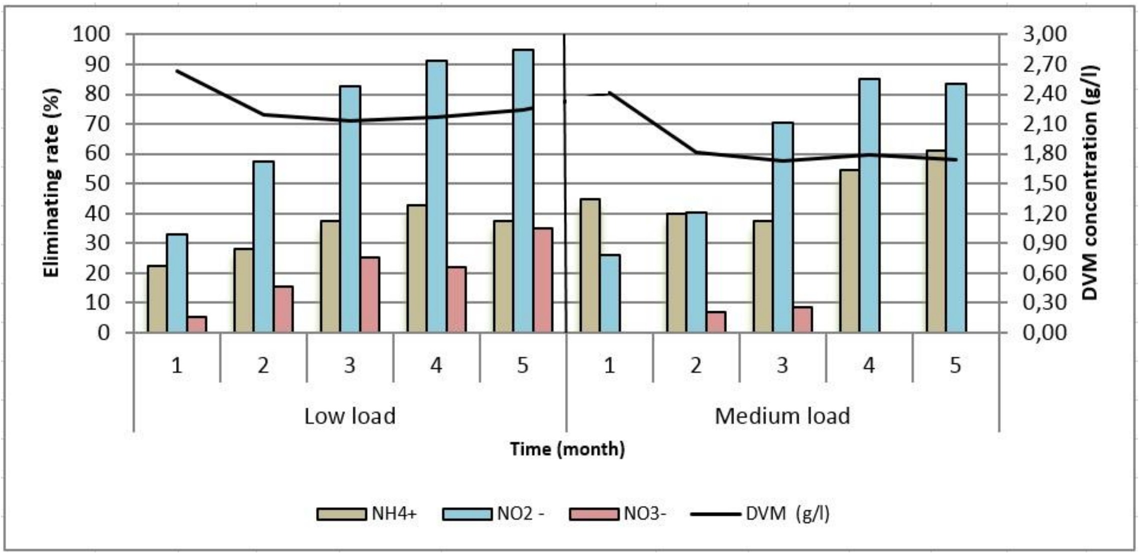


Fig. 4: Abatement rate evolution of NH_4^+, NO_2^-, NO_3^- ions at the output of each SBR cycle on the basis of time and DVM.

Change in NH_4^+, NO_2^-, NO_3^- ion concentration during a 24-hour cycle is shown in Figure 5 graphs.

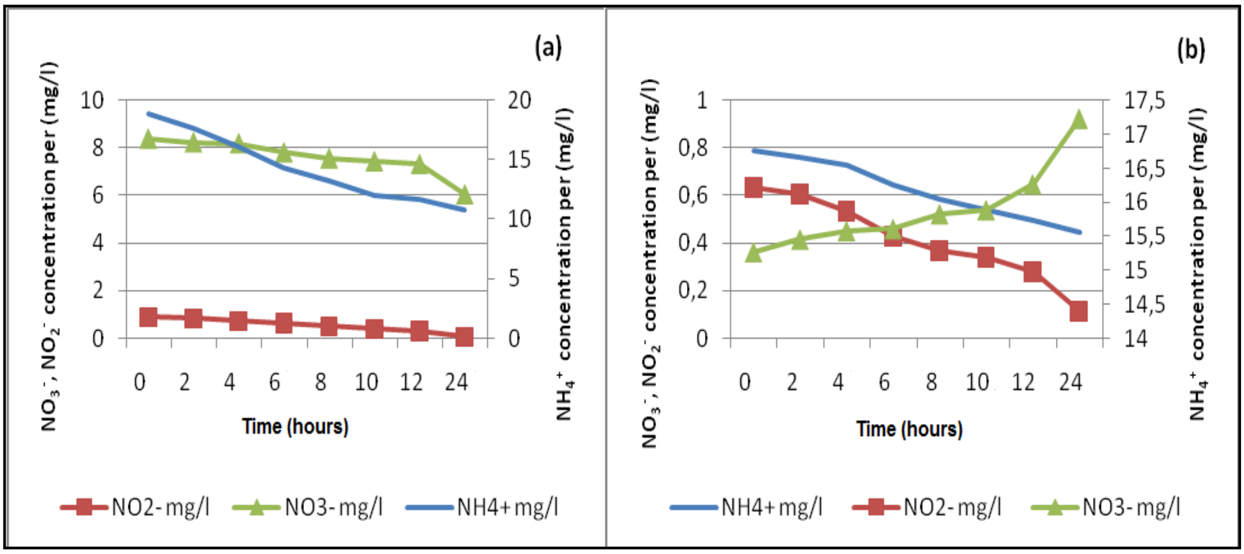


Figure 5: Variation in NH_4^+, NO_2^-, NO_3^- average concentration in treated effluent during SBR operation (a) low load, (b) medium load.

During low load treatment (0.3kg de COD m^{-3} d^{-1}), the observed values show that there was a decrease in ammonium concentration in the aeration phase and extending up to 22 hours with a stabilization of the DVM concentration around 2.20 ± 0.03 g/l (Figure 5). Towards the end of the treatment process, the ammonium concentration in the treated effluent decreased to 9.7 mg/L. With the medium organic load (0.7kg de COD m^{-3} d^{-1}), the NH_4^+ concentration at the output of SBR starts to decrease from the first day. Indeed, the biological oxidation of ammonium ions to nitrite ions has gone well during the aeration phase (22 hours) as a result of

microorganisms forming mixed liquor. The same observations have been reported by several authors [25-30]. Several others [31-32] have pointed out that ammonium ions are oxidized in nitrite ion according to nitritation reaction catalysed by *Nitrosomonas* bacteria.
The average recorded values for nitrite during treatment for low to medium load show that there was no important variation during the microorganisms acclimatization phase (Fig. 4). After this long phase, the DVM concentration stabilized at around 2.2 ± 0.03 g/l at low load and 1.81± 0.01 g/l at medium load, which coincided with nitrite ions abatement rate of 95% and 84% respectively. This is due to the oxidation of nitrite ions in the ion nitrate during the aeration phase (nitration) caused by *Nitrobacter* bacteria that require oxygen to complete the nitrification process [26-32]. The recorded values of nitrate ions during treatment at low load had a decrease of 45% after a 22 hours aerobic phase (Figure 5). This variation suggests that the conversion of nitrate ions to molecular nitrogen (N_2) (denitrification process) [33] has taken place even after a 22 hours aeration, which means the presence of specific microorganisms able to denitrify under aerobic conditions. Indeed, [30] has pointed out that the denitrification in SBR system is possible, but following an alternation of aerobic and anoxic phases during treatment cycle.
Increasing the flow rate of 225 to 525 ml/d through a medium load has caused an accumulation of ions nitrates in the reactor, an increase of 15.27 to 17.23 mg/l during a 24 hours processing cycle (Figure 5). This accumulation is due to the interruption of the nitrogen pollution process to the conversion of nitrites to nitrates, the nitration stage catalyzed by *Nitrobacter* bacteria.
Indeed, high concentration of heavy metals affects the activity of the purifying micro-organisms of mixed liquor [34]. These results obtained are coherent with those COD_t and BOD_5 abatement rate when medium organic load is applied (increase of the flow rate to 525 ml/d). This leads us to conclude that the denitrifying bacteria activity responsible for lowering the aerobic ions nitrate is also inhibited at medium load. On the other hand, the increase of the effluent toxic property at medium load is likely correlated to the increase in the metallic load [35]. Figure 6 shows the PO_4^{3-} ions abatement rate recorded during each SBR cycle at low load and medium load.

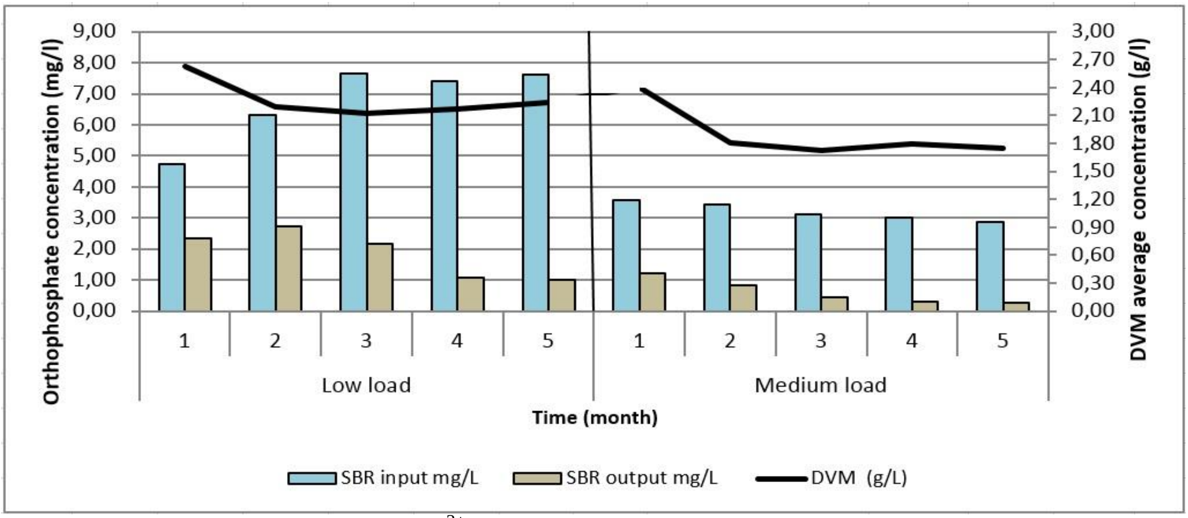


Figure 6: Variation in concentration of PO_4^{3+} ions recorded at the input and output of each SBR cycle with low and medium organic load.

As shown in Figure 6, the variation of concentration observed during treatment with low and medium load has shown that there significant abatement in the orthophosphate concentration at the SBR output. Orthophosphate concentration of the treated effluent was 1.01mg/L at low load with an average abatement rate of about 91%. When medium load is applied, and from the 2th month, we have observed that orthophosphate concentration in the treated effluent has reached a concentration of 0.76mg/l, with an average abatement rate that exceeds 83%. This decrease could be the outcome of orthophosphate assimilation by microorganisms of the mixed liquor [25]. Indeed, the treated effluent concentration is below Moroccan discharge standards [29]. That makes it possible to release the treated effluent into the environment.
Figure 7 shows heavy metals abatement rate during brassware effluent treatment through SBR at low and medium load.

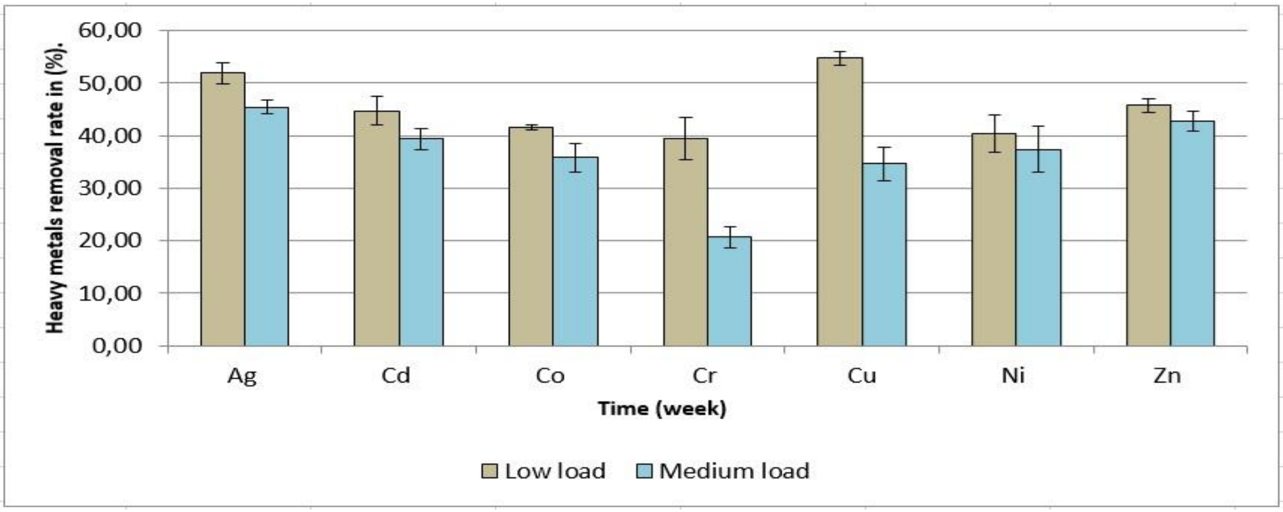


Figure 7: Heavy metals abatement rates during brassware effluent treatment through SBR at low and medium load. Values ± standard deviations.

Results analysis in Figure 7 show that the heavy metals abatement observed at low load for Ag, Cd, Co, Cr, Cu, Ni and Zn are about 32.8 ± 0.83, 34.74 ± 2.77, 31.29 ± 0.27, 34.36 ± 2.02, 41.42 ± 4.05, 30.98 ± 2.43 and 38.79 ± 3.11% respectively. The most important abatement rates were recorded for copper and chromium, with percentage removal that exceeds 45%. These results have been obtained towards the end of the treatment process. During the treatment at average load with the increase of flow rate of 225 to 525 ml/d, heavy metals abatements recorded are still below the recorded results at low load with averages of Ag : 27.77 ± 2.37 %, Cd : 29.33 ± 1.93%, Ni : 26.06 ± 3.90 % and Zn : 32.83 ± 1.91 %. We also note that Nikel and copper concentration, main pollutants brassware effluent, underwent a decrease representative for the two applied organic loads.
Removal of these metal-based compounds after biological treatment of brassware effluent in sequencing batch bioreactor could be due to activated sludge micro-organisms action. They act though different mechanisms that can be classified based on their metabolic needs as bioaccumulation and adsorption or biosorption (passive fixation process which is usually fast) [16,36-37].
Within our treatment system, organic matter and heavy metals are reduced by purifying biomass in the sludge [35,38-39]. Sirianuntapiboon and Chaiyasing (2000) have proved that dry volatile matter increase could enhance the system efficiency and improve the quality of the effluent at the output of the treatment process, thing we were able to notice in this study. Metals abatement rate variation may be due to the ionic form of each metal, the ability of each bacterium to accumulate it and to physico-chemical conditions which differ from one germ to another) [35]. It was an average efficiency, because of the difficulties of maintaining the microbial population at peak activity due to heavy metals toxicity and the problem of nutrient availability) [40].

Conclusion

In this work, we have applied the SBR process at low and medium organic load, to treat a brassware effluent in the city of Fez, which is characterized by the presence of heavy metal and organic loads.
Removal rates of organic pollution elements detected after treatment with SBR at low load are high, compared with the medium load. Ideed, by applying low load, removal rate for COD, BOD_5, ammonium, nitrite and orthophosphate is around 79, 95, 38, 96 and 89%, respectively. However, these removal rate after treating at medium load are 74, 81, 60, 86 and 98% respectively for COD, BOD_5, ammonium, nitrite, and orthophosphate. Indeed, abatements observed for heavy metals Ag, Cu and Ni were at around 32.87 ± 0.83, 41.42 ± 4.05 and 30.98 ± 2.43 % respectively for low load and 27.77 ± 2.37, 34.63 ± 3.29 and 26.06 ± 3.90 % successively for Ag, Cu and Ni shifting to medium load.
Finally, this study found that the sludge contains adapted strains, heavy metals resistant which have led to better abatement of organic and metallic load (e.g. brassware effluent), especially at low load. The sludge also contains denitrifying strains resistant to high heavy metals concentrations that have produced a reduction of NO_3 to N_2. This is a very important result in the use of these strains to treat brassware effluents on an industrial scale without adding anoxic reactor. SBR is therefore an efficient process in the abatement of organic and metallic load with low installation and operating cost.

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