

# Screening of physicochemical and bacteriological parameters of the soap industry wastewater: Development of an efficient treatment model

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**Abstract** : Wastewater deriving from soap and oil industries contains many toxic pollutants that affect the receiving environment. Therefore, an efficient treatment prior to its discharge is required. Various technologies have been used in the treatment of this wastewater such as coagulation-flocculation and membrane filtration but these methods are expensive and unsafe to the environment. In the present study, the efficiency of a new soap wastewater treatment model based on the evaporation and condensation process was evaluated using physicochemical and microbiological standard methods. We also assessed the phytotoxicity of soap wastewater before and after treatment. Our results revealed that total suspended solid levels ( $20\,467 \pm 371.2$  mg/l), chemical oxygen demand ( $41\,667 \pm 928$  mg/l), biochemical oxygen demand ( $22\,000 \pm 503.3$  mg/l), total organic carbon ( $17\,333 \pm 432.2$  mg/l), nitrate ( $49.35 \pm 0.04$  mg/l), phosphorus ( $445.8 \pm 0.75$  mg/l), chloride ( $227.2 \pm 0.43$  mg/l), sulfate ( $56 \pm 0.21$  mg/l) and phenols ( $757.97 \pm 8.2$  mg/l) exceeded the Tunisian standards. This indicates that this effluent is highly charged with organic and chemical pollutants. The concentrations of Cu, Zn and Fe in soap wastewater were also above the standard limits. Microbiological analysis revealed however the absence of pathogenic bacteria in raw and treated effluents. Moreover, our treatment system was able to reduce significantly the values of all pollutants. *Triticum turgidum durum* L and *Solanum lycopersicum* L wetted with treated effluent showed also high germination percentages (74.41% and 79.42%) compared with raw effluent (0%). Therefore, our new system was demonstrated as an efficient and feasible approach for the soap wastewater treatment.

**Key words:** Soap industry wastewater, Toxic pollutants, Wastewater treatment, Bacterial parameters, Physicochemical parameters, Phytotoxicity.

## 1. Introduction

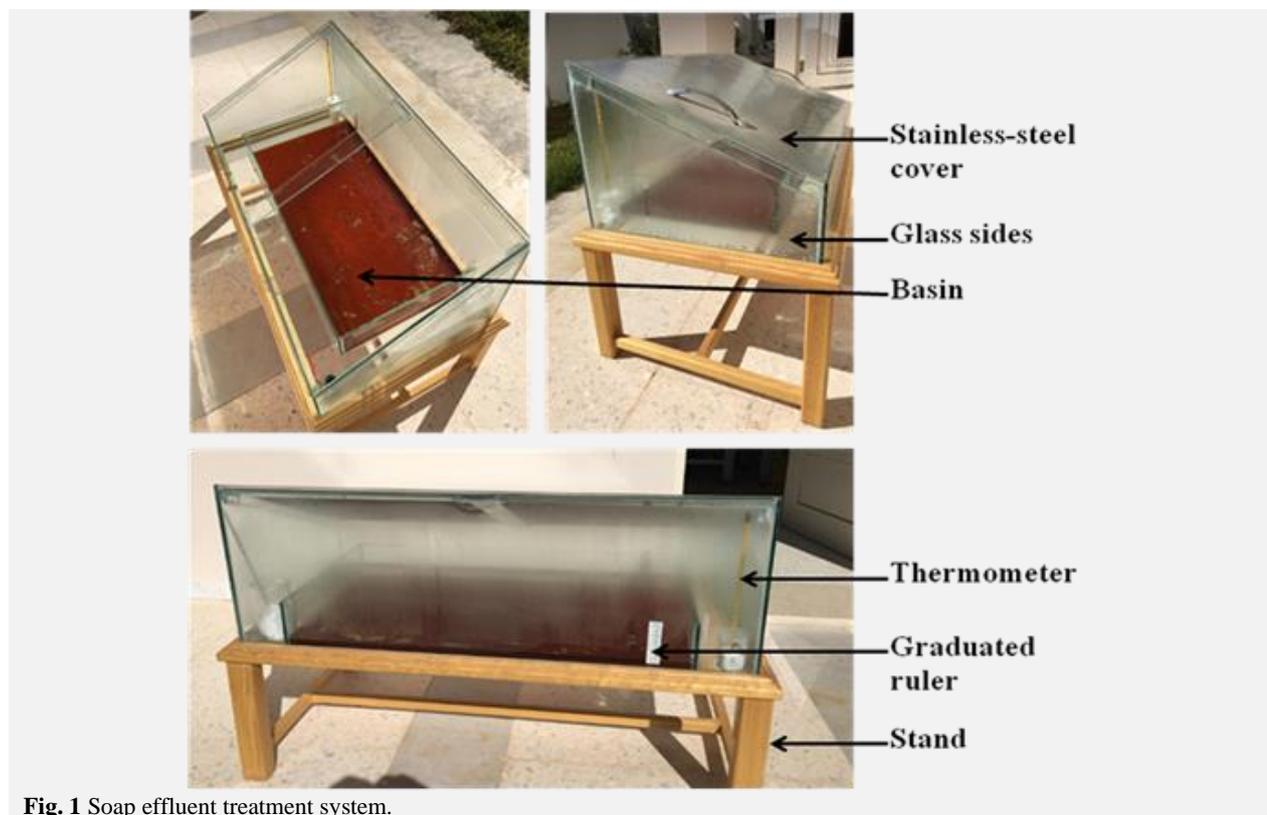
In the past decades, water pollution caused by the discharge of industrial and domestic wastewater has become an environmental problem of great concern for public health and aquatic ecosystems. Previous studies have demonstrated the heavily contamination of seawater receiving many effluents such as textile, hospital and urban effluents in Tunisia (Afsa et al. 2020; Methneni et al. 2021; Hassen et al. 2022). Wastewaters deriving from soap and detergents industries may also contain substantial amounts of chemicals including surfactants, dyes, fragrances, co-solvents and organic matters characterized by low biodegradability and high toxicity (Martins et al. 2011; Mohammed et al. 2021). For this reason, the treatment of these wastewaters become a crucial step to ensure environment protection. A variety of approaches have been developed for their treatment including physical and physicochemical methods (i.e membrane filtration, coagulation-flocculation and adsorption), biological methods and chemical treatment (i.e electrochemistry, chemical oxidation, ion-exchange, chemical precipitation, catalytic degradation) (Deng et al. 2020). These technologies are complex, expensive and tend to consume a high amount of chemicals (Azimi et al. 2016). Therefore, it is particularly important to develop suitable and new treatment processes treating wastewater with high performance and lower cost. The aim of this study was to develop an efficient soap wastewater treatment based on the simple evaporation-condensation process. We also explored physicochemical and microbiological parameters of the wastewater before and after treatment. Moreover, the phytotoxicity of the effluent was evaluated.

## 2. Material and methods

### 2.1. Description of treatment system

Our system is based on the evaporation and condensation process (Hassen et al. 2021). The wastewater storage basin is constructed with a diameter of 100 cm x 50 cm and a height of 50 cm as illustrated in Figure

1. The cover is made of stainless-steel in order to increase the temperature inside the system. The inclination of this cover is about 40° to facilitate the recuperation of water. The sides of the system are made of tempered transparent glass to allow the passage of solar energy. Two thermometers are used inside the system to monitor wet and dry temperatures. The level of wastewater is controlled using a graduated ruler.



## 2.2. Wastewater samples

Wastewater was sampled from the main collector of the ZOUILA Company, Mahdia-Tunisia. This Company operates in three segments: soap making, oil refining and extraction of oils from pomace. The samples used for the present study consisted of soap wastewater. The collected wastewater samples were transported to the laboratory to determine the different physicochemical parameters and stored at -20° C until further analysis.

## 2.3. Physicochemical characterization of Samples

All samples were analyzed for various physicochemical parameters including chemical oxygen demand (COD), biochemical oxygen demand (BOD), total suspended solids (TSS) and total organic carbon (TOC) using a portable UV analyzer (Pastel UV, Secomam, Alès, France). Conductivity and pH were measured using conductometer WTW 315i and pHmeter WTW. The chemical parameters such as phosphorus, sulfate, chloride and phenols were determined in laboratory following the Standard Methods for the Examination of Water and Wastewater (Aniyikaiye et al. 2019). Concentrations of metals (Iron, Cadmium, Zinc, Chromium, Nickel, Plomb, Cobalt and copper) were determined inductively with coupled plasma mass spectrometer (model JY-2000; HORIBA Jobin Yvon, Switzerland). All parameters were measured before and after wastewater treatment. For each determination, average values of three replicates were taken.

## 2.4. Microbiological analysis

Wastewater and treated samples were aseptically pipetted into a sterile Erlenmeyer flask and diluted tenfold followed by subsequent decimal dilution (up to 10<sup>-6</sup>). The enumeration of the Mesophilic, Coliform bacteria, *Staphylococci*, *Streptococci*, *Enterococci* and *Salmonella* was performed with Plate Count Agar, TTC Tergitol Agar, Chapman, Litsky, Slanetz and Macconkey mediums, respectively. Next, the inoculated dishes were incubated at 37°C for 48h. Results were expressed by colony forming units (CFU) per 100 mL of sample.

## 2.5. Phytotoxicity assay

Germination test rates was performed as described by Buchmann (2015). Tests were conducted using plastic Petri dishes and two layers of filter paper. Ten undamaged seeds of tomato (*Solanum lycopersicum L*) and

durum wheat (*Triticum turgidum durum L*) were laid on the filter paper in each dish, which contained 4 mL of raw or treated wastewater. Each condition was tested in triplicate. Petri dishes were incubated at 25°C for 72h. A control test with distilled water was performed in triplicate for every tested condition. Then, relative seed germination (RSG), relative root growth (RRG) and germination index (GI) were determined according to the following formula (Buchmann et al. 2015):

$$\text{RSG (\%)} = (\text{number of seeds germinated of the sample} / \text{number of seeds germinated of the control}) \times 100$$

$$\text{RRG (\%)} = (\text{Mean root length of the sample} / \text{Mean root length of the control}) \times 100$$

$$\text{GI (\%)} = (\text{RSG} \times \text{RRG}) / 100$$

## 2.6. Statistical analysis

Statistical analyses were performed using SPSS, version 17.0 (SPSS Inc., Chicago, IL, USA). Student's t test was applied to compare parameters before and after treatment. Data were expressed as the mean ± standard error. A p value < 0.05 was considered statistically significant.

## 3. Results and discussion

### 3.1. Daily control of treatment system

In this study, a novel system for soap wastewater treatment using an evaporation–condensation process was developed. It is low cost and easy to operate. The transparent sides of the system enable maximum absorption of solar radiation. The heated water evaporates from the basin and condensates on the inclined cover. The salts and pollutants contained in the effluent are left in the basin and the condensed water is delivered into a valve at the bottom of the system.

The different parameters collected during soap wastewater treatment (dry and humid temperature, relative humidity, raw effluent level and treated effluent quantity) are listed in Table 1.

**Table 1.** Daily control of treatment system.

Date	Dry temperature (°C)	Humid temperature (°C)	Relative humidity (%)	Effluent level (cm)	Quantity of effluent (ml)	Treated water (ml)
1	35	44	49	3.5	10000	-
2	20	25	59	3.5	-	-
3	34	43	47	3.4	-	-
4	21	26	60	3.3	9550	450
5	29	37	49	3.3	-	-
6	30	37	55	3.2	-	-
7	35	42	59	3.2	9050	500
8	23	30	48	3	-	-
9	36	45	50	3	-	-
10	32	40	51	2.7	8315	735
11	25	32	50	2.7	-	-
12	25	33	44	2.7	7815	500
13	31	40	45	2.7	-	-
14	36	45	50	2.5	7165	650
15	20	28	37	2.5	-	-
16	40	49	52	2.5	-	-
17	20	25	59	2.1	6665	500
18	40	47	61	2.1	-	-
19	33	42	47	1.8	-	-
20	36	46	45	1.8	5995	670
21	18	25	41	1.8	-	-
22	34	43	47	1.7	-	-
23	20	25	59	1.7	5345	650
24	29	37	49	1.3	-	-
25	30	37	55	1.3	4845	500
26	35	42	59	1.2	-	-
27	23	30	48	1.2	4095	750
28	36	45	50	1.1	-	-
29	32	40	51	1	-	-
30	25	32	50	0.7	3245	850

Relative humidity varies in the range 37% to 61% during the treatment of soap wastewater. The evaporation was tightly dependent on the relative humidity and temperature. After 30 days of soap wastewater treatment with our system, 6755 ml of treated wastewater was collected (Table 1). The efficiency was calculated according the following formula:

$$\text{Efficiency: } (V_1/V_0) * 100 ; V_1 = \text{treated wastewater}; V_0 = \text{raw wastewater}$$

$$\text{Efficiency: } (6755/10000) * 100 = 67.55\%.$$

### 3.2. Physicochemical parameters

The physicochemical parameters of the soap effluent before and after treatment are summarized in table 2

**Table 2.** Physicochemical parameters of soap wastewater before and after treatment.

Parameters	Before treatment	After treatment	P value	Tunisian standards, public hydraulic discharge NT.106.002 (1989)	Tunisian standards, public canalization NT.106.002 (1989)
pH	13.23±0.18	8.1±0.12	<0.001	[6.5-8.5]	[6.5-8.5]
Conductivity (µs/cm)	1223.33±6.67	687±3.51	<0.001	5000	5000
TSS (mg/l)	20 467±371.2	1.25±0.03	<0.001	30	400
COD (mg/l)	41 667 ± 928	0.61±0.1	<0.001	90	1000
BOD (mg/l)	22 000 ±503.3	0.02±0.01	<0.001	30	400
TOC (mg/l)	17 333±432.2	1.09±0.04	<0.001	-	-
Nitrate (mg/l)	49.35±0.04	5.9±0.03	<0.001	50	90
Phenol (mg/l)	757.97±8.2	0	<0.001	-	-
Chloride (mg/l)	227.2±0.43	28.4±0.11	<0.001	600	700
Sulfate (mg/l)	56±0.21	0	<0.001	600	400
Phosphorus (mg/l)	445.8± 0.75	1.68±0.006	<0.001	0.05	10
Fats (mg/l)	4750±86.6	2300±28.87	<0.001	10	30
Fe (mg/l)	2.96±0.02	0.43±0.006	<0.001	-	5
Cu (mg/l)	5.14±0.06	0.45±0.0006	<0.001	-	2
Zn (mg/l)	4.35±0.02	0.19±0.001	<0.001	-	5
Cr (mg/l)	0.09±0.0006	0.06±0.0006	<0.001	-	1
Cd(mg/l)	0	0	-	-	0.02
Pb (mg/l)	0	0	-	-	1
Co (mg/l)	0	0	-	-	1
Ni (mg/l)	0	0	-	-	1

TSS: total suspended solid, COD: chemical oxygen demand, BOD: biochemical oxygen demand, TOC: total organic carbon, Fe: Iron, Cu: copper, Zn: zinc, Cr: chrome, Cd: cadmium, Pb: plumb, Co: Cobalt, Ni: Nickel.

The pH values recorded for soap effluent varied between 13.45 and 13.65 and were above Tunisian limits. The alkaline pH can be explained by the use of caustic soda and potash in the saponification of the fatty acids. In fact, the rejections resulting from hypo-dye and from washing water of the soap are rich in hydroxide ions (Ehouman et al. 2017). These values of pH decreased after treatment to 8.3. The electrical conductivity of the wastewater samples was also analyzed, it ranged from 1216.66 µs/cm to 1230 µs/cm which reflects a high level of water salinity.

The mean values of TSS, COD, BOD and TOC in soap wastewater were 20 467 ±371.2 mg/l, 41 667 ± 928 mg/l, 22 000 ±503.3 mg/l and 17 333±432.2 mg/l, respectively. These values exceeded the acceptable Tunisian limits. Similar values were previously detected (Abdel-Gawad and Abdel-Shafy 2002; Ehouman et al. 2017). The high concentration of COD and BOD in soap effluent could be linked to the presence of high levels of non-saponified fatty matters as well as the organic compounds such as surfactants, colorants, glycerin, phosphorus and phenolic compounds (Ehouman et al. 2017). TSS, COD, BOD and TOC recorded after wastewater treatment with our system decreased significantly and didn't exceed the acceptable limits (Table 2).

As shown in Table 2, soap wastewater contained a large amount of nitrate (49.35±0.04 mg/l). It is probably due to the use of nitrogenous compounds such as nitrated dyes, EDTA, nitrogen-containing surfactants in the formulation of soap and detergents which release ammonium and nitrate and nitrite ions (Ehouman et al. 2017). The values of phosphorus (445.8± 0.75 mg/l) in soap wastewater also exceeded the acceptable Tunisian limits. This is may be explained by the use of adjuvants or bleaching agents rich in phosphorus elements in the formulation of soap and detergents (Ehouman et al. 2017).

Our results showed also high concentrations in phenols (757.97±8.2 mg/l) and fats (4750±86.6 mg/l) in raw effluent due to fatty acids used in the saponification process.

As shown in Table 2, the mean values of chloride and sulfate in soap effluent were 227.2±0.43 mg/l and 56±0.21 mg/l, respectively (Table 2). Results obtained after treatment gave 5.9 ±0.03 mg/l for nitrate, 1.68 ± 0.006 mg/l for phosphorus, 28.4 ±0.11 mg/l for chloride, 0 mg/l for phenols.

The highest mean values of heavy metals detected in soap wastewater were Cu, Zn and Fe with concentrations of 5.14±0.06 mg/l and 4.35±0.02 mg/l and 2.96±0.02 mg/l respectively (Table 2). Cu concentration level was higher than the acceptable limit recommended by the Tunisian standard for public canalization (NT 106.002 (1989)). The presence of these metals could result from mechanical wear at the mills. In addition, the high concentration of iron may be due to colored pigments such as iron oxide used in soap and detergent formulation. The high concentration of Zn could be explained by the use of washing water of the soap rich in this metal. Cd, pb, Co and Ni heavy metals were not detected in soap wastewater before and after treatment (Table 2).

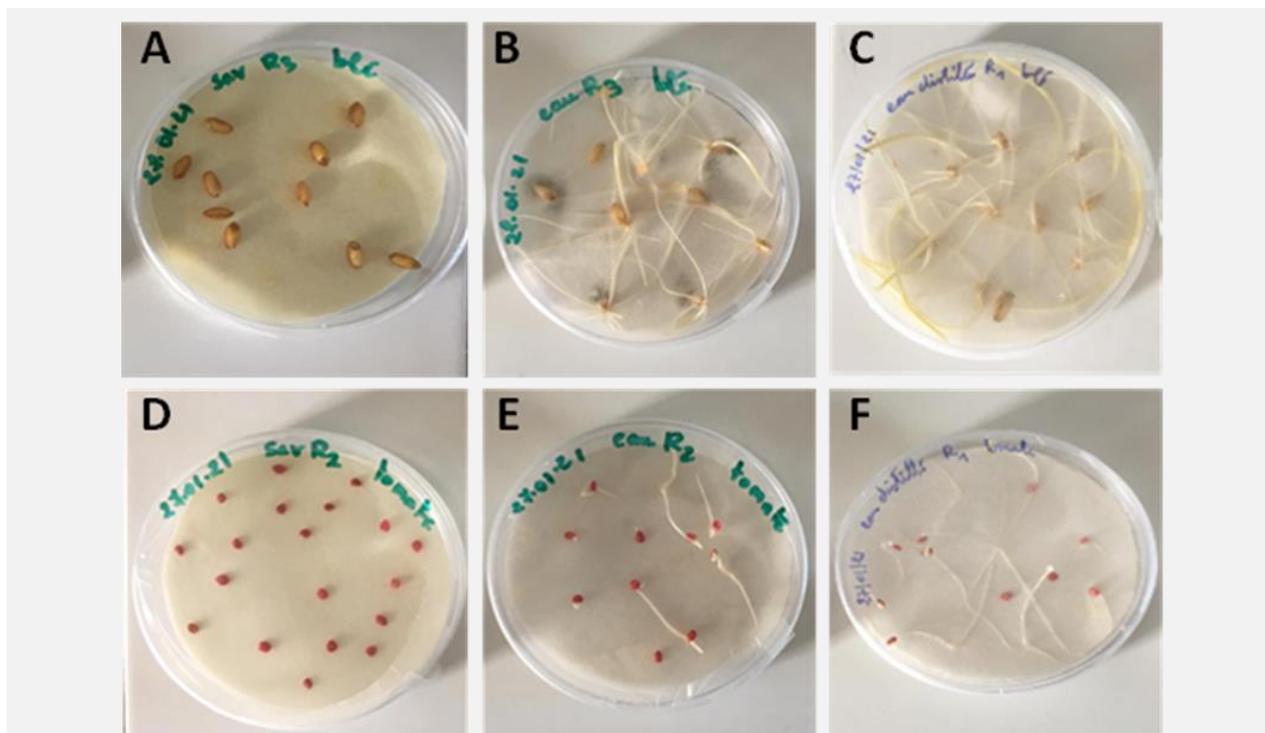
Our treatment system was able to remove 100% of sulfate, 100% of phenols, 99.9% of COD, BOD, TOC and TSS, 99.62% of phosphorus, 88.04% of nitrates, 87.5% of chlorides, 51.57 % fats, 43.84 % of conductivity, 95.63 % of Zn, 91.24 of Cu, 85.44% of Fe and 33.3% of Cr.

### 3.3. Microbiological analysis

The enumeration of bacteria community revealed the absence of pathogenic bacteria in raw and treated effluents. The average concentrations of mesophilic bacteria in raw and treated wastewater samples were  $7.35 \cdot 10^4$  CFU/ml and  $1.72 \cdot 10^3$  CFU/ml, respectively. The absence of pathogenic germs could be related to the lack of favorable conditions for survival in soap effluent and the presence of antimicrobial substances such as polyphenols, fatty acids and caustic soda (El Addouli et al. 2009).

### 3.4. Phytotoxicity assay

Phytotoxicity assay revealed no germination for durum wheat and tomato watered with raw effluent (Figure 2 A, D). These data indicate the highly phytotoxic pollutant in these effluents samples which can inhibit the growth of durum wheat and tomato. In contrast, the results of RSG, RRG for durum wheat and tomato watered with treated wastewater were 92.96 %, 80.05 % and 80.08%, 99.18%, respectively. In addition, the highest value of GI was obtained for tomato seeds wetted with treated effluent (79.42 %) followed by durum wheat seeds (74.41%). We can conclude that the treated wastewater is suitable for agricultural use.



**Fig. 2** Phytotoxicity assay

Germination of *Triticum turgidum durum* L watered with raw wastewater (A), treated wastewater (B) and distilled water (C). Germination of *Solanum lycopersicum* L watered with distilled water raw wastewater (D), treated wastewater (E) and distilled water (F).

## 4. Conclusion

Our results showed that the studied parameters of soap wastewater (TSS, COD, BOD, TOC, nitrate, phosphorus, chloride, sulfate and phenols) exceed the Tunisian standards indicating that this effluent is highly charged with organic and chemical pollutants. Compared with raw wastewater amounts, all studied parameters decreased by treatment process. Treatment of soap wastewater with our system also resulted in 74.41% and 79.42% germination of durum wheat and tomato. Therefore, our new system was demonstrated as an efficient approach for the soap wastewater treatment.

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### Authors' Contributions

SB, WH and HBM conceived and designed the experiments. SB and WH carried out the functional experiments, analyzed the data. AJ participated in the functional experiments. SB wrote the manuscript. All authors read and approved the final manuscript.

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