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Original Paper

Identifying effluents from a food processing industry

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characteristics.

Abstract

The agri-food industry in Morocco generates significant volumes of liquid waste, contributing to environmental challenges that directly impact public health. To address this issue, it is crucial to characterize this wastewater comprehensively, enabling the development of suitable treatment strategies to alleviate pollution and potentially facilitate recycling. This study focuses on the identification of effluents from an olive and caper preservation industry, employing physicochemical and bacteriological analyses on raw, decanted, and filtered effluent samples. The findings reveal that the effluent from the olive and caper preservation industry is characterized by high acidity and an exceptionally elevated mineral load. Notably, the application of decantation and filtration methods demonstrates a limited influence, primarily affecting the reduction of suspended solids. Understanding these physicochemical and bacteriological characteristics is pivotal for devising targeted treatment protocols, ensuring effective pollution reduction, and exploring avenues for potential recycling of this agri-food industry wastewater. This research serves as a foundation for informed decision-making in the development of sustainable and efficient wastewater management practices, balancing environmental preservation with industrial needs.

1. Introduction

The agri-food industry in Morocco confronts a pressing environmental challenge with the substantial generation of liquid waste, presenting significant implications for public health. The water available on earth currently represents less than 1% of the total volume of fresh water. The quality of the part that can potentially be used by humans (0.1%) has been steadily deteriorating as a result of our liquid, solid and gaseous toxic discharges. As a result, clean water is becoming increasingly scarce in many parts of the world, such as North Africa, India, the Persian Gulf countries, ... [1-2] In Morocco, the potential of natural water resources is estimated at 22 billion m³/year, or the equivalent of 700 m³/capita/year, commonly accepted as the critical threshold indicating the onset of shortages and latent water crises [3]. This limitation of water resources is compounded by the deterioration in water quality as a result of increased pollution [4-9]. The main sources of pollution are domestic [10,12], industrial [13,14], agricultural [15-17], mining [18,19], accidental [20,21], leachate from landfill sites [22-24], quarries [25,26], ...

The agri-food industries generate liquid waste, made up of organic and mineral compounds, which could easily be treated using traditional pollution control methods. Despite considerable efforts in effluent treatment, it is estimated that around 60% is currently sent to a wastewater treatment plant. The rest is discharged into the natural environment [27-30]. One of the most alarming phenomena is the growing accumulation of organic and inorganic substances in water that are difficult to biodegrade [31,32]. The situation is made worse by the lack of inadequacy of a suitable water treatment system capable of reducing the concentration of these substances [33]. As a result, these poorly treated effluents inevitably lead to a deterioration in the quality of water sources, and hence a lack of significant quantities of drinking water in many countries [34].

This study looks at the characterization of effluent generated by an olive and caper preserving industry, which is currently stored in a pit to be decanted and then discharged into the sewage manhole.

2. Experimental details

2.1. Origin of effluent

The effluent under consideration emanates from a company engaged in the preservation of olives, capers, caprons, fresh red and green chillies, and onions through the process of brining. To effectively anticipate the parameters of the effluent requiring analysis, an understanding of the distinct stages involved in the processing of these food items proves instrumental. The preservation process for olives and capers encompasses four key stages preceding packaging. This sequence involves debittering in a 1.5 to 2% soda bath, washing with potable water, fermentation, and brining in a 6 to 8% brine bath, and finally, sorting and grading. Throughout these operations, there is a generation of modest quantities of solid waste and liquid discharges originating from three specific units: leaching, washing, and brining. These byproducts are systematically stored in a purpose-built pit to manage the environmental impact of the preservation process.

2.2. Collecting samples

The effluent was systematically collected through the grab sampling method, ensuring a representative sample for analysis. Following collection, each sample was meticulously rinsed with

the same effluent and then stored in 30-litre plastic drums. To prevent any potential contamination or alteration of the sample, the drums were filled to capacity, eliminating the presence of air bubbles, and subsequently hermetically sealed. The sealed samples were then transported to the laboratory under controlled conditions and promptly placed in a refrigerator set at 4°C for preservation until the initiation of analytical procedures. Importantly, the collected effluents underwent no additional chemical preservation processes due to their inherently high salt content. The comprehensive analysis involved the measurement of various physico-chemical parameters, including pH, temperature (T), conductivity (EC), suspended solids (SS), chloride (Cl⁻), chemical oxygen demand (COD), biological oxygen demand (BOD₅), and total phosphorus (Pt). Furthermore, bacteriological parameters, specifically faecal coliforms (FC) and faecal streptococci (FS), were assessed. All analyses strictly adhered to the AFNOR standards as stipulated by Rodier, and to ensure the robustness and reliability of the results, each measurement was conducted thrice. This rigorous approach to sample collection, handling, and analysis ensures the accuracy and integrity of the obtained data.



Fig. 1. Collecting samples

3. Methods for characterizing the effluents studied

3.1. Physicochemical parameters

3.1.1. pH

The pH is one of the fundamental characteristics of water. The pH gives an indication of the acidity of an aqueous solution. It is determined from the quantity of hydronium ions (H⁺) or hydroxyl ions (OH⁻) contained in the substance. When the quantities of these two ions are equal, the water (or substance) is considered neutral, and the pH value is around 7. The pH of a substance varies between 0 and 14. Above 7, the substance is considered basic and the quantity of OH⁻ ions is greater than that of H⁺ ions. Below 7, the substance is acidic and there are more H⁺ ions.

The hydrogen potential (pH) was measured using a Hanna pH 209 pH meter after calibration with buffer solutions of pH = 4 and 8.

3.1.2. Conductivity (EC)

The electrical conductivity of water reflects its ability to allow an electric current to pass through it. Charges are transported by the ions contained in the water. The higher the mineral content of the

water, the greater its conductivity. It was measured using a Hanna EC214 conductivity meter after calibration with a 0.1 M KCl solution.

3.1.3. Suspended solids (SS)

SS The determination of suspended solids (SS) involved a centrifugation process. A known volume (V_e) of the effluent, whether decanted or not, was subjected to high-speed centrifugation at 4500 rpm for a duration of 20 minutes, resulting in the separation of two distinct phases. The solid phase obtained was carefully recovered, subsequently dried in an oven set at $105 \pm 2^\circ\text{C}$, then cooled within a desiccator and re-weighed to obtain the final mass (M_2). The calculation of the suspended solids content was performed using the Eq. 1, providing a quantitative measure of the suspended solids present in the effluent. This meticulous centrifugation and analytical procedure ensured a precise determination of suspended solids, contributing to the comprehensive physico-chemical characterization of the effluent sample.

$$[SS] = \frac{(M_2 - M_1)}{V_e} \times 1000 \quad (\text{mg} / \text{L}) \quad (1)$$

3.1.4. Chemical Oxygen Demand (COD)

COD serves as a crucial indicator for assessing the concentration of organic matter, whether dissolved or suspended, in water. This measurement is based on the quantification of oxygen needed for the complete chemical oxidation of the organic components. By determining COD, it becomes possible to evaluate the pollutant load of an effluent in terms of organic matter, both prior to and after undergoing physical, chemical, or biological treatment. This assessment aids in monitoring the efficiency of a treatment plant and the activity of microorganisms involved in the process. The COD analysis adhered to the AFNOR T90-101 standard. The COD (2) calculation is performed using the Eq. 2.

$$DCO = \frac{8000(V_0 - V_1) \times C}{V} \quad (2)$$

Where V_0 is Volume of iron ammonium sulphate (II) required for the determination of oxidised organic matter (mL), V_1 represent the volume of iron(II) ammonium sulphate required for the blank test (mL), C is the concentration of the iron (II) ammonium sulphate solution (in mol/L) and V sayand for Volume of test sample (in mL).

3.1.5. Biological Oxygen Demand (BOD_5)

BOD_5 represents the amount of oxygen required by living microorganisms to oxidize and stabilize the organic matter found in an effluent. This parameter serves as a key indicator of the water's biodegradable organic content. At a temperature of 20°C , the degradation of organic matter initiates promptly. Conventionally, BOD_5 is quantified in terms of milligrams per liter (mg/L) of oxygen consumed during a 5-day incubation period at 20°C . The respirometric, or nanometric, method is employed for BOD_5 determination, utilizing the WARBURG respirometer principle. This technique involves directly measuring the respiration of microorganisms through a device equipped with a pressure sensor. The analysis is conducted in strict accordance with the AFNOR T90-103 standard to ensure precision and reliability in assessing the biodegradability of organic matter in the water sample.

3.1.6. Total phosphorus (P_t)

Total phosphorus (P_t) is the collective measure of phosphorus present in water, encompassing various forms such as polyphosphates, organophosphates, and orthophosphates. The determination of total phosphorus is crucial due to phosphorus being a key contributor to the eutrophication of aquatic environments. The analysis involves mineralization in an acid medium, conducted in the presence of sodium persulphate at 150°C for a duration of two hours. This process effectively transforms the organic and combined forms of phosphorus into soluble orthophosphates, allowing for accurate quantification of the total phosphorus content in the water sample. This method serves as an essential tool in understanding and managing the impact of phosphorus on water ecosystems and supports environmental monitoring efforts.

3.1.7. Chlorides (Cl⁻)

The concentration of chlorides (Cl⁻) was determined following the AFNOR standard T90-014. The analysis employed Mohr's titrimetric method, utilizing silver nitrate and potassium chromate. The concentration of chloride ions is calculated using the expression:

$$[\text{Cl}^-] = 35.5 \times V_{\text{AgNO}_3} \times N_{\text{AgNO}_3} \text{ (mg/L)}$$

3.1.8. Nitrates (NO₃⁻)

Nitrate ions (NO₃⁻) are the main form of inorganic nitrogen found in natural waters. They are the final stage in the oxidation of nitrogen. The main source of nitrates is leaching from agricultural land. Nitrates are the main staple food, promoting the growth of micro-organisms. They also come from industrial and municipal effluents because nitrates can be used as detergents or food preservatives and as a raw chemical in various industrial processes.

Nitrates are reduced to nitrites by amalgamated Cd. The nitrites thus produced form a diazo compound with 4-aminobenzenesulphonamide, which when coupled with NED (N-(1-Naphthyl) Ethylene Diamine) gives a pink complex suitable for colorimetric determination at a wavelength λ around 415 nm.

$$[\text{NO}_3^-] = [\text{NO}_3^-] \text{ de la droite d'étalonnage} \times 4,43$$

3.1.9. Ammonium (NH₄⁺)

Ammoniacal nitrogen in natural waters can come from the leaching of agricultural land and clay minerals, as well as from municipal and industrial sewage. Its concentration is generally low in groundwater, as it absorbs onto soil particles and clay. In some drinking water treatment plants, ammonia is added to react with the chlorine to form a residue. The presence of ammonium generally indicates pollution of organic origin. In an alkaline environment and in the presence of nitroprussianate, which acts as a catalyst, NH₄⁺ ions treated with a chlorine solution and phenol produce indophenol blue, which can be measured colorimetrically at a wavelength of 630 nm.

3.1.10. Heavy metals and other elements

They were measured using an inductively coupled plasma (ICP) mass spectrometer after mineralization of the sample to be measured. The residues remaining after treatment of the effluents were put into a heated solution, in an Erlenmeyer flask on a sand bath hot plate at 250°C, with 3.5 mL of nitric acid and 7.5 mL of hydrochloric acid. The Erlenmeyer flask was covered with a watch

glass. After boiling, the solution became clear and 2 mL of 5% HCl was added. The resulting solution was made up to 5mL with distilled water and stored in a polyethylene tube for ICP determination.

3.2. Bacteriological analyses

The bacteriological analysis of the samples is concentrated on assessing pollution indicator microorganisms, specifically faecal coliforms (FC) and faecal streptococci (FS). This targeted approach aligns with Moroccan standards, which mandate the testing of water for these two types of bacteria. Faecal coliforms and faecal streptococci serve as vital indicators of potential fecal contamination in water sources, and their detection is crucial for evaluating water quality and ensuring compliance with established health and safety standards.

3.2.1. Faecal coliforms (FC)

Total coliforms and faecal coliforms are counted using the Most Probable Number (MPN) analysis, which begins with a presumptive test. Three tubes filled with double concentration tryptose lauryl sulphate broth (2C) to which 10 mL of sample was added, and six tubes containing single concentration tryptose lauryl sulphate broth (C). For the first 3, 1mL of sample was added and 0.1mL for the other three. Incubation was carried out at $(37\pm 1^{\circ}\text{C})$ for 48 h.

If a positive tube count was found (cloudiness + gas), a confirmation test would be carried out by subculturing them on the shiny green culture medium, to which must be added a smear of the presumptive test samples (10 mL, 1 mL and 0.1 mL) using a flamed and cooled loop. Incubation takes place at $(44 \pm 0.5^{\circ}\text{C})$ for 24 h. Next, count the number of positive tubes (cloudiness + gas).

3.2.2. Faecal Streptococci (FS)

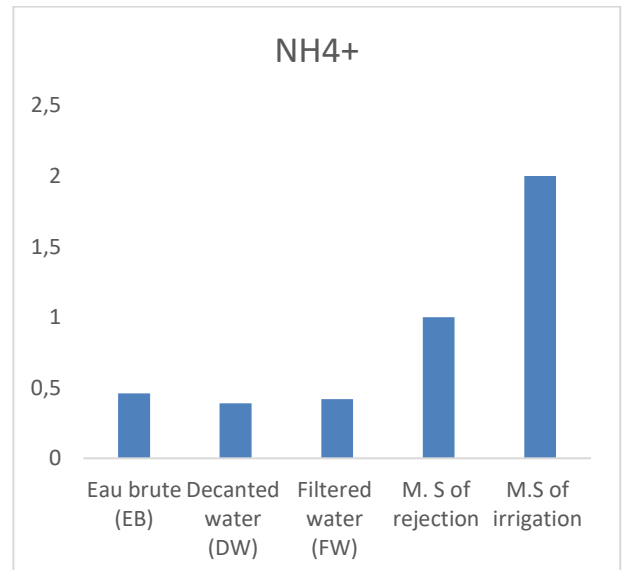
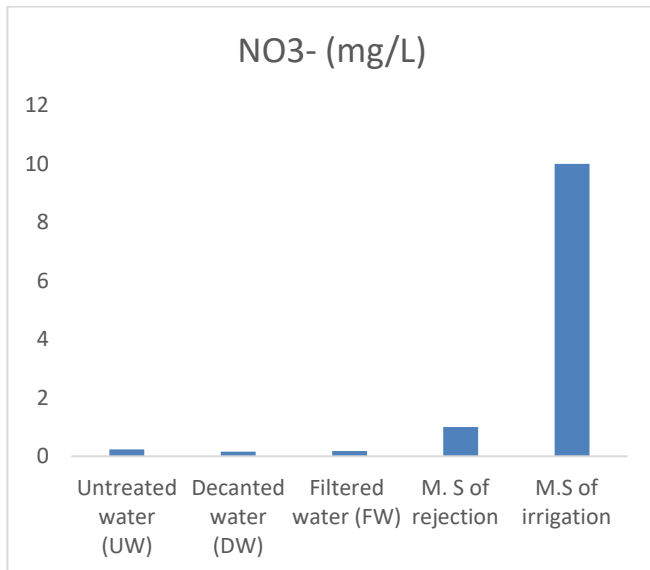
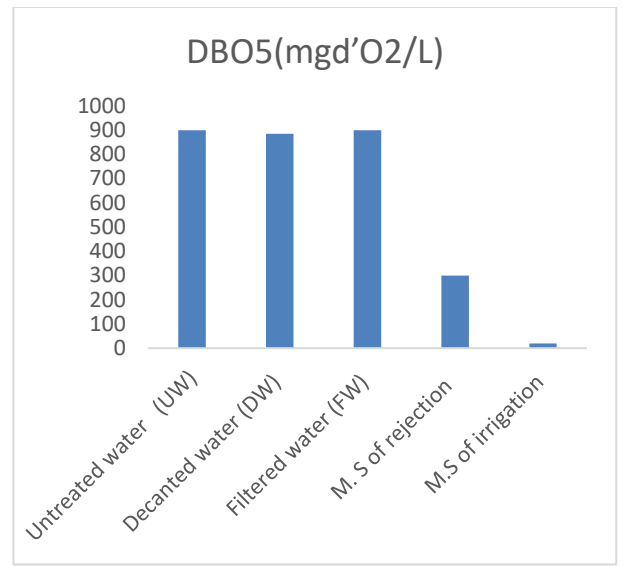
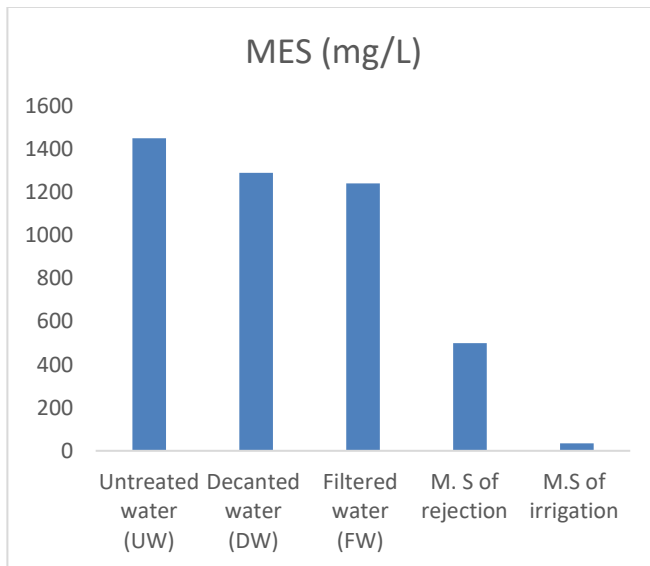
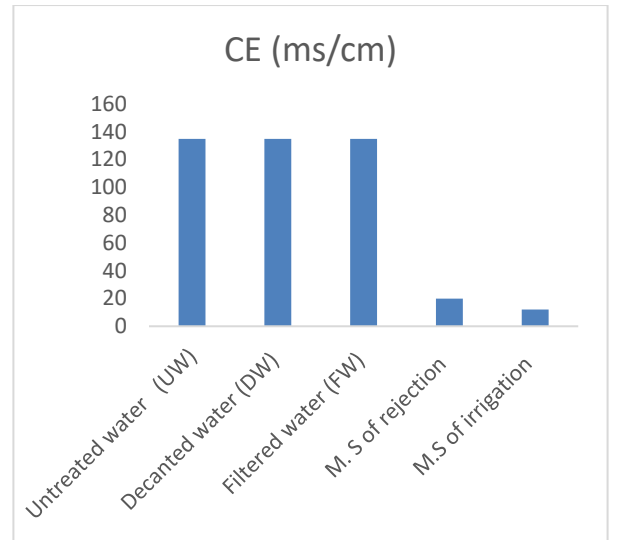
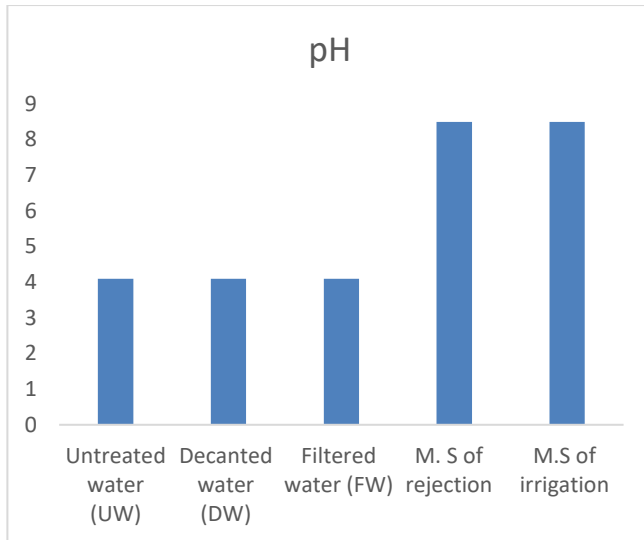
Faecal streptococci are counted using the Most Probable Number (MPN) test, which begins with a presumptive test. Three tubes were filled with double-strength sodium acid broth (2C) to which 10 mL of sample was added, and six tubes were filled with single-strength sodium acid broth (C). For the first 3, 1 mL of sample was added and 0.1 mL for the other three. Incubation took place at $(37 \pm 1^{\circ}\text{C})$ for 48 h.

If any positive tubes were found (cloudy or white deposit), a confirmation test was carried out by subculturing them on Litsky selective culture medium. An aliquot of the presumptive test samples (10 mL, 1 mL and 0.1 mL) would then be added using a cooled flamed loop. Incubation took place at $(37 \pm 1^{\circ}\text{C})$ for 48 h. Positive tubes are cloudy, possibly with a purple pellet at the bottom of the tube.

4. Results and discussion

4.1. Physicochemical characterization

The results of the physicochemical characterization of the effluent from the caper and olive preservation industry in its raw state (EB), decanted for 1 hour (ED), or filtered on vacuum filter paper (EF) and compared with the limit values of Moroccan standards for effluent discharge into surface water or for reuse in irrigation are presented in [Fig.2](#).



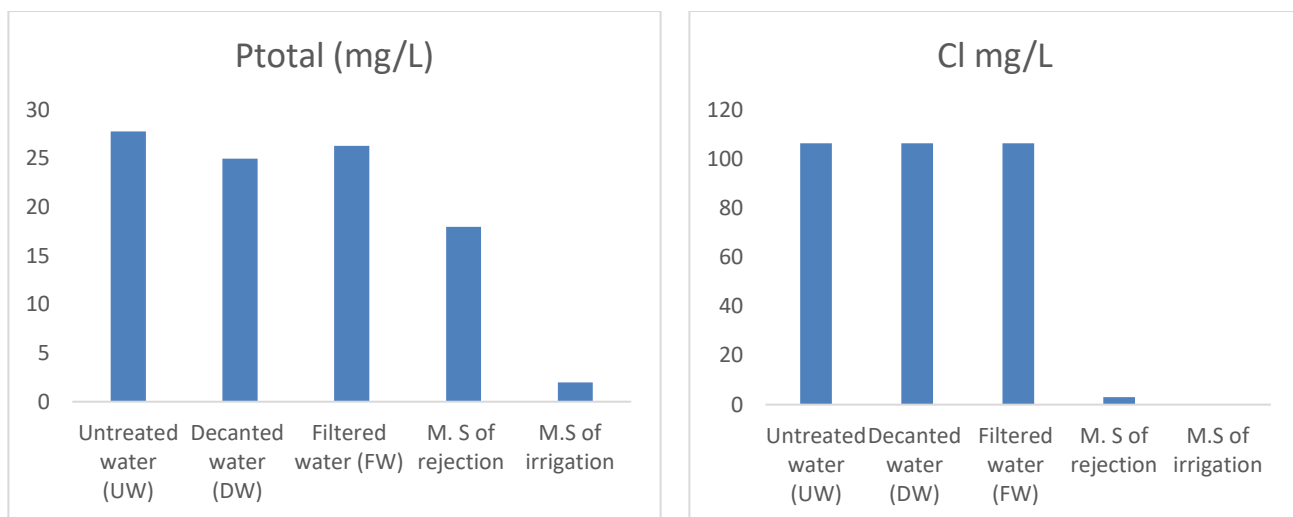


Fig. 2. Physicochemical characterization of effluent from the olive and caper preservation industry

The results show that the effluent studied is acidic ($\text{pH} = 4.1$) loaded with easily biodegradable organic matter ($\text{COD/BOD}_5 = 1.84 \leq 3$) and has a high mineral load ($\text{EC} = 135 \text{ ms/Cm}$), particularly in chloride ion Cl^- and sodium. This load is most likely due to the SS released during the washing of the capers and olives and the salts used for their brining. The phosphorus load of these waters seems to be due to the detergents used for washing the soil and utensils and/or to the release of this element during the washing of the foodstuffs to be preserved, mainly from the fertilizers used during their cultivation. In fact, filtration or decantation only have an influence on reducing the SS of the effluent by almost 32%.

4.2. Heavy metals and other elements

Fig. 3 shows the results obtained for the heavy metals found in the effluent studied in its raw or pre-treated state. The trace metals detected in the effluent studied are trace elements (Cu, Fe and Zn) and all meet the standard for discharge of this effluent into surface water, or for use in the irrigation of green spaces.

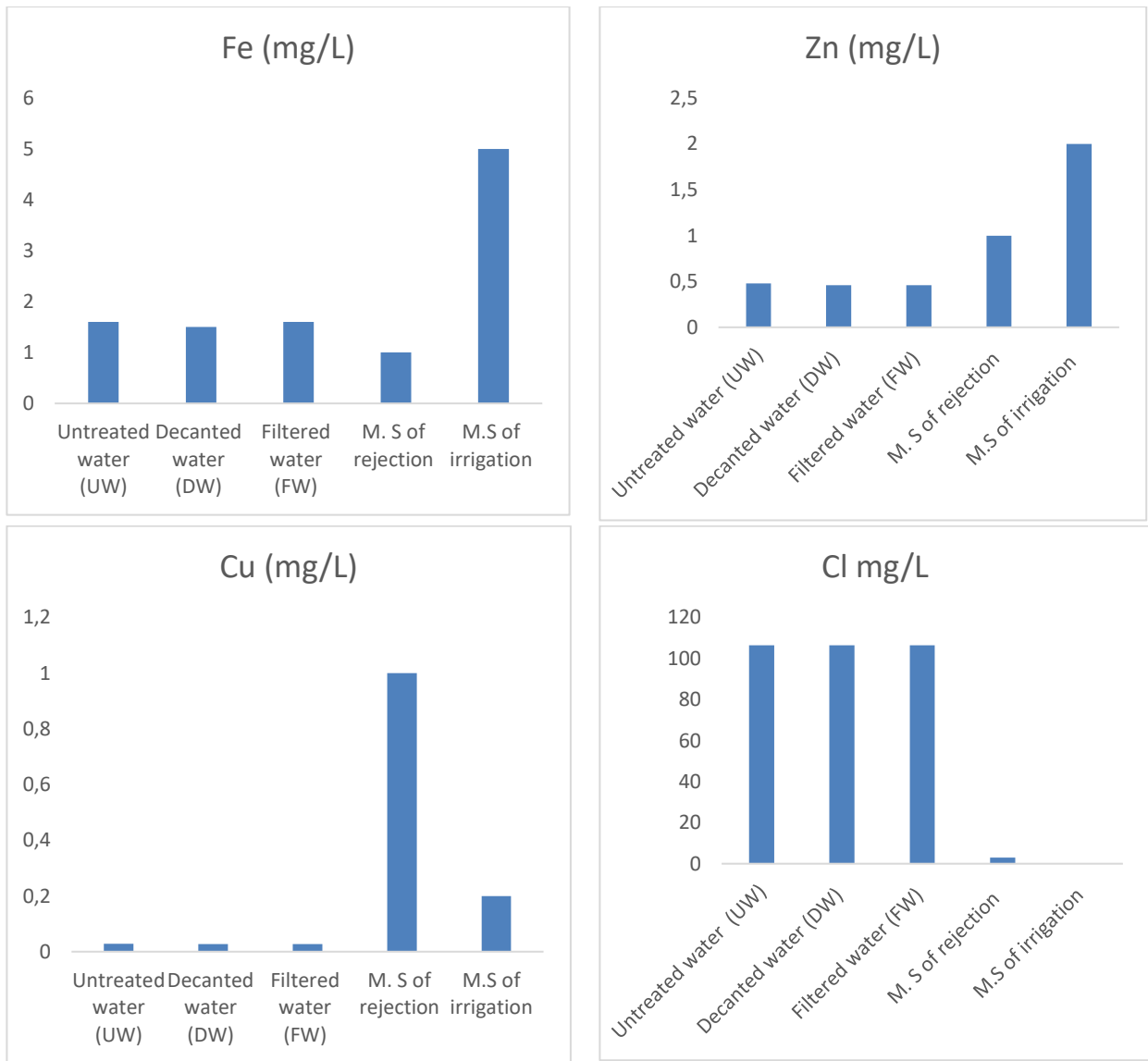


Fig. 3. Metal load in raw or pre-treated effluent

4.3. Bacteriological analysis

The results of bacteriological analyses of the effluent studied in its raw or pre-treated state in terms of faecal coliforms and faecal streptococci are presented in [fig. 4](#). Analysis of the values obtained shows that this effluent, in its raw, decanted or filtered state, can neither be discharged into surface water nor considered as an alternative source of irrigation for green spaces, given its load in FC and FSC, even if it is low, which seems to be due to its stay in the pit for more than a month. For this reason, it is recommended that this effluent from the caper and olive preservation industry be treated after one hour's decantation and as soon as it is generated, using a suitable physicochemical or bacteriological method to enable it to be reused.

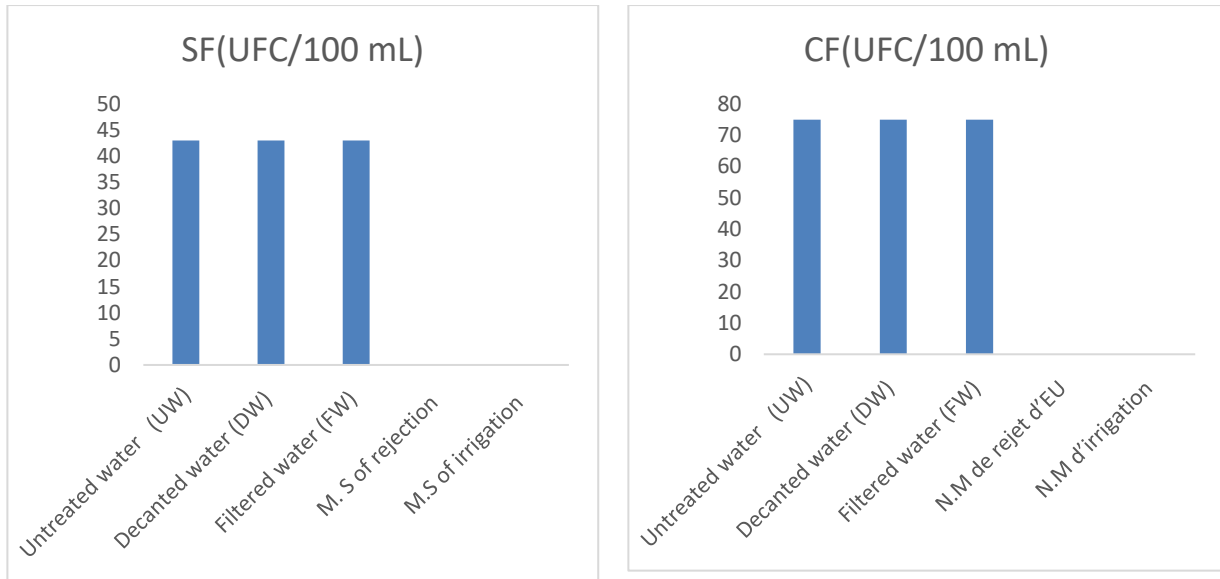


Fig. 4. Bacteriological load of the effluent studied

5. Conclusion

This study focused on the diagnosis of an effluent from the caper and olive preservation industry by means of physicochemical and bacteriological characterization. The aim was to identify the main sources of pollution and suggest a method of treatment or recovery. The results show that the caper and olive preservation industry generates around 15 m³/d of effluent, mainly from the brine and caper and olive washing units. This effluent is acidic and has a bacterial, biodegradable organic, mineral and suspended matter load that exceeds effluent discharge standards for surface water. It is therefore highly recommended to treat it as soon as it is generated. This will, on the one hand, provide an alternative source of unconventional water for irrigating green spaces and, on the other hand, reduce its secondary effects, which can be seen on a number of levels, for example, the damage to the aesthetic appearance of towns and cities, thus affecting the tourist sector, or the serious damage caused to flora and fauna, leading to the destabilization of ecosystems, etc.

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