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Physicochemical characterization and environmental assessment of oily effluents from petroleum production

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ABSTRACT

In Algeria, oil fields generate significant quantities of wastewater on a daily basis. These waters are often laden with hydrocarbons and heavy metals, with concentrations exceeding established standards. However, addressing this issue to prevent pollution at the Marine Terminal of Bejaia is one of the major objectives of our study. During this study, the degree of pollution in the wastewater of the Marine Terminal Center in Bejaia is assessed through the analysis of various parameters; a comprehensive diagnosis of the purge waters from the investigated site was established. Characterization tests of these waters revealed a significant and irregular pollution, both organic (hydrocarbons) and mineral (heavy metals), which could be detrimental to the receiving aquatic environment (river, sea). For these reasons, it is considered that these purge waters require prior treatment before being discharged into the sea.

Keywords: petroleum production**,** oily effluent, pollution, caracterization.

INTRODUCTION

Human exploitation of oil deposits has steadily increased since the beginning of the last century. The extraction, transportation, and utilization of this energy source pose risks of pollution (both accidental and chronic) to the environment, which can influence the ecological balance and sometimes lead to the destruction of ecosystems.

Oil fields generate significant quantities of water daily, which are typically loaded with total hydrocarbons (THC) and heavy metals exceeding the established standards. According to the MALCOM and LIGHT model (1987) on the maturation of oil source rocks, the presence of substantial amounts of heavy metals in oil reservoirs originates from basin waters that are saline and highly mineralized.

The regional transport center (RTC) is responsible for transporting crude oil and condensate, connecting Haoud El Hamra (Hassi Messaoud) to the marine terminal of Bejaia (TMB), ensuring the delivery of its products crude oil and condensate for export via the port of Bejaia. Thus, this city remains one of the most illustrative examples of hydrocarbon pollution in the country.

Faced with this precarious situation and for the proper preservation of the receiving environment, we attempt in this study to assess the degree of water pollution through the analysis of various characterization parameters.

MATERIAL AND METHODS

Material

Sampling is defined as a procedure in which a portion of a substance, material, or product is taken to provide a representative sample of the whole for analysis purposes. Since sampling generally has a specific purpose, the sampling strategy must always be tailored to the objectives of the study (Gueguen and Martin, 2001).

It is within this framework that our study was conducted at the oil site located at the crossroads of Bejaia. The samples were taken from four tanks named as follows: 4C4, 4D3, 4F5, and 4A8.

The determination of these parameters is performed according to standard analytical methods (Bellache *et al.*, 2022). Water discharge samples were taken from the oil tanks in opaque bottles with a capacity of one liter. Prior to sampling, the bottles were rinsed with distilled water and labeled. Two physico-chemical parameters (temperature, pH) were measured directly in the field. To prevent significant modification of these parameters due to potential exchanges that the samples could undergo when in contact with the external environment, these measurements were carried out using a portable HANNA HI 8314 pH meter.

Subsequently, 1.5 ml of 65% nitric acid was added to the samples intended for heavy metal measurement by AAS (atomic absorption spectrophotometry). This acidification limits the adsorption of metals on the walls and organic matter, prevents precipitation, and inhibits the development of microorganisms that could sequester metals. The samples were then stored in a cooler at a temperature of 4 °C and transported to the Materials Technology and Process Engineering laboratory at A/MIRA-Bejaia University. Great care was taken to maintain cleanliness conditions during sample collection and handling, aiming to avoid potential sample contamination (Gueguen and Martin, 2001). Heavy metal analysis in water requires prior filtration of the solution to remove any suspension that may contain hydrophobic organic contaminants (Philippe, 2010). We used 0.2 μm porosity filters to distinguish the dissolved phase (< 0.2 μm) from the particulate phase (> 0.2 μm). The filtrates were then collected in test tubes and analyzed.

Methods

The various physico-chemical analyses conducted for the characterization of purging waters are presented in Table 1.

RESULTS AND DISCUSSION

This part will be dedicated to discussing the results obtained from the characterization of purge waters. In order to assess the quality of these waters, we compared our results with the standards proposed by the World Health Organization (WHO), as well as with the Algerian standards regarding the required quality of discharge waters.

Characterization of purge waters

pH

pH is one of the most important factors influencing the reactivity of metals in water. The pH of water measures the concentration of protons H^+ in the water. It reflects the stability of the equilibrium established among different forms of carbonic acid and is linked to the buffer system developed by carbonates and bicarbonates. Figure 1 illustrates the pH variation over sampling time for four oil tanks located at the crossroads

Table 1. Physico-chemical analysis of purging waters

Parameters	Equipment and reagents	Reference
Temperature and pH	Portable HANNA HI 8314 pH meter	(Samake, 2009)
Conductivity	Consort C 813 type conductivity meter	
Suspended solids	Gravimetric method	
Turbidity	WTW TURB 550 Turbidimeter	
Chemical oxygen demand (COD)	Reflux method in an open system; apparatus: BEHROTEST TRS200	
Alkalimetric Titer (TA, TAC)	Titration with 0.02 N hydrochloric acid or sulfuric acid	
Hardness (TH)	Complexometric method by titration with EDTA (0.1N) in the presence of eriochrome black T	
Chloride determination	By volumetry, following the Mohr method	
Sulfates, naphthalene and chromium (IV) content	Nano COLOR UV/VIS spectrophotometer by Macherey-Nagel	
Determination of zinc, and lead concentrations	Atomic absorption spectrophotometry (AAS)	

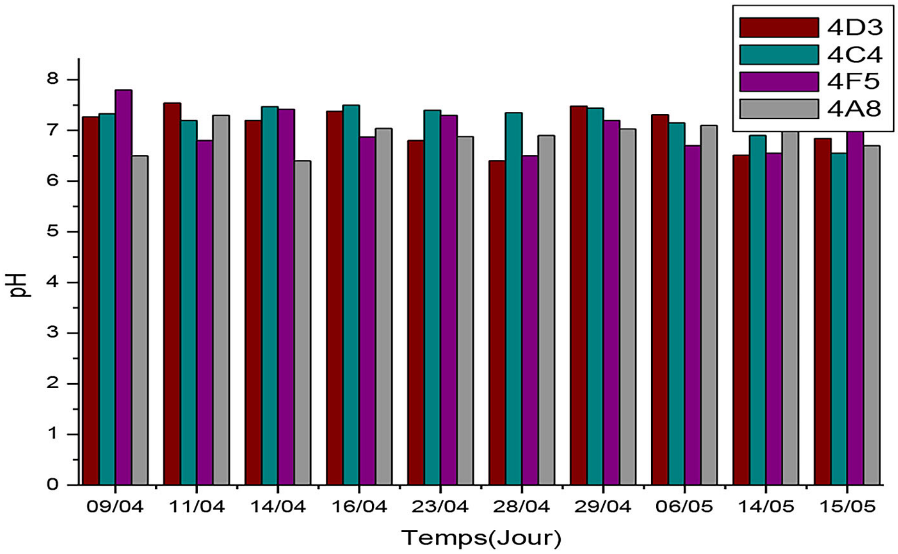


Figure 1. pH variation of purge waters over time for tanks 4D3, 4C4, 4F5, and 4A8

of Bejaia. The values observed in Figure 1 reveal that the pH is neutral in all purge water samples along our sampling and analysis campaign. The values range from 6.4 to 7.54, and these recorded values fall within the maximum limit range for the pH of industrial discharge waters, where pH is typically between 6.5 and 8.5.

Conductivity

The measurement of conductivity provides a good assessment of the degree of mineralization of water, where each ion contributes based on its concentration and specific conductivity. Figure 2 depicts the variation in water conductivity over the sampling time for four oil tanks. The conductivity values for the samples vary from one tank to another, ranging between (30.63 and 67.23) mS/cm. The highest value is observed in tank 4D3 on 09/04. All these values exceed the Algerian standard set at 2.8 mS/cm (Vierling, 2008). The relatively high conductivity values indicate the highly mineralized nature of the purge waters (conductivity ≥ 1000 µS/cm).

Turbidity

Turbidity is caused by the presence of suspended matter or substances in solution, such as mineral substances, organic matter, or other

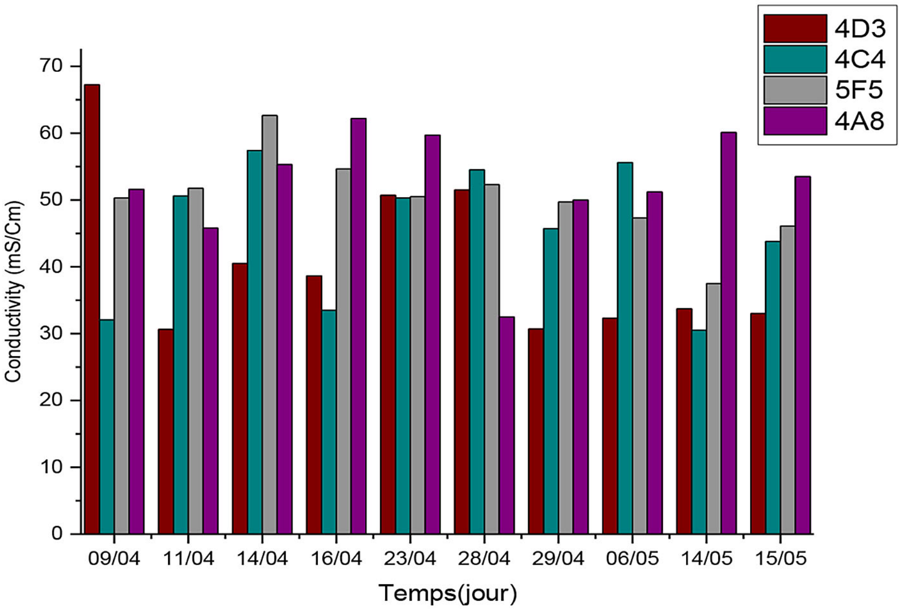


Figure 2. Variation in conductivity of purge waters over time for tanks 4D3, 4C4, 4F5, and 4A8

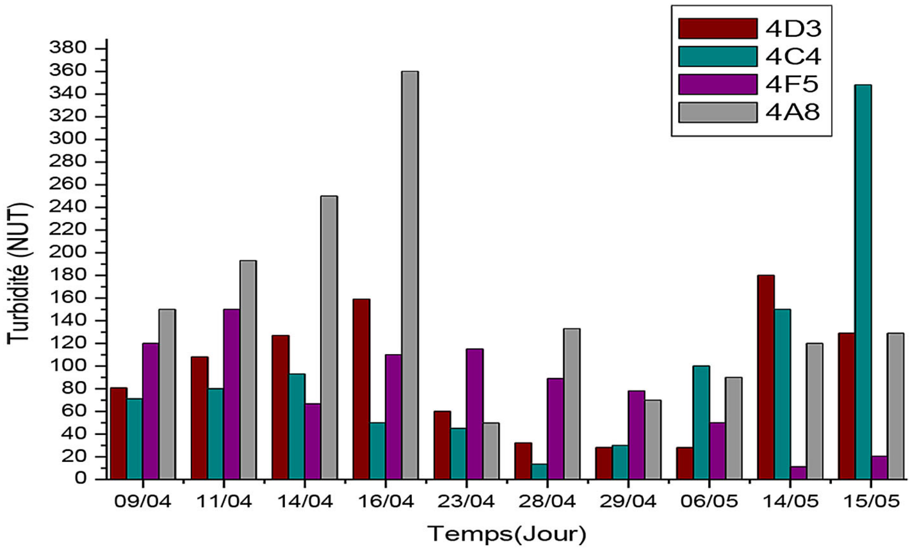


Figure 3. Turbidity variation of purge water samples over time for tanks 4D3, 4C4, 4F5, and 4A8

microscopic materials, which obstruct the passage of light in water (Hade, 2007). Figure 3 illustrates the variation in turbidity over the sampling time for the four studied oil tanks 4D3, 4C4, 4A8, and 4F5.

The turbidity values recorded in the purge waters range between (10 and 360) NTU. Two peaks of high intensity were observed during our monitoring, which are respectively 348 NTU and 360 NTU, at tanks 4A8 and 4C4 respectively. The majority of the turbidity values measured are outside the WHO standards range which varies between (5 to 30) NTU. This allows us to conclude that the purge waters are turbid.

Suspended solids (SS)

To assess the quality of water, it is always necessary to quantitatively evaluate its load of dissolved and particulate matter. Suspended solids represent the entirety of mineral and organic particles contained in water. Suspended particles can lead to unpleasant tastes and odors in water, depending on the nature of the traversed terrain, season, rainfall, water flow regime, nature of discharges. (Rodier, 2016). Figure 4 illustrates the variation in suspended solids (SS) over the sampling time for the four oil tanks. The results of suspended solids (SS) calculations show that the

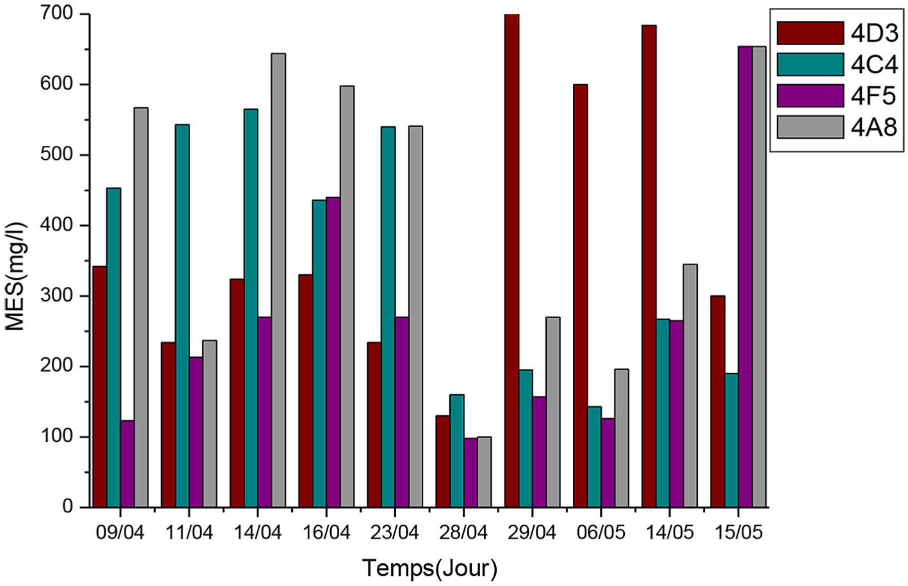


Figure 4. Variation in suspended solids (SS) of purge waters over time for tanks 4D3, 4C4, 4F5, and 4A8

concentration varies between 98 and 700 mg/l, exceeding the permissible limit of 30 mg/l required by Algerian discharge standards and WHO guidelines. This may be due to the presence of heavy oils with high molecular weights. These high levels of suspended solids can be considered a form of pollution. Furthermore, they can cause nuisances, including sludge deposits and clogging of receiving water bodies. Settled sludge is detrimental to the maintenance of natural biological structures (Rodier *et al.,* 2009), while clogging hinders the biodegradation of micropollutants (Timoléon and Fulbert, 2013).

Total alkalimetric titre (TAT)

The relative values of the alkalimetric titre (AT) and the total alkalinity titre (TAT) allow determining the quantities of alkali or alkaline earth hydroxides, carbonates, or bicarbonates present in the water through the following reactions:

$$OH^- + H_3O^+ \rightarrow 2H_2O \qquad (1)$$

$$CO_3^{2-} + H_3O^+ \rightarrow H_2O + HCO_3^- \qquad (2)$$

$$HCO_3^- + H_3O^+ \rightarrow H_2O + H_2CO_3 \qquad (3)$$

The methyl orange endpoint occurs as soon as the pH drops below 4.4, meaning that it indicates the presence of excess strong acid in the medium. Therefore, the measurement involves the sum of OH^-, CO_3^{2-}, and HCO_3^- ions; this determination corresponds to the Total Alkalinity (TA). Table 2 summarizes the concentration values of different anions based on AT and TA. Figure 5 illustrates the variation of total alkalinity (TA) over the sampling time for the four oil tanks.

Based on the results illustrated in Figure 5 and recorded in Table 2, it is observed that the values of total alkalinity (TA) vary between 8 and 10 meq/l during the sampling period. These values significantly exceed the WHO standard set at 2 meq/l (100 mg/l of $CaCO_3$). The alkalimetric titre is zero, a value lower than the WHO standard of 50 mg/l of $CaCO_3$, as the pH ranges from 6.4 to 7.54. Therefore, it can be concluded that the purge waters primarily consist of bicarbonates.

Table 2. Concentration values based on TA and TAC

Alkalinity	Hydroxide	Carbonate	Bicarbonate
TA = 0	0	0	TAC
TA <TAC/2	0	2TA	TAC - 2TA
TA = TAC/2	0	2TA	0
TA >TAC/2	2TA - TAC	2 (TA - TA)	0
TA = TAC	TA	0	0

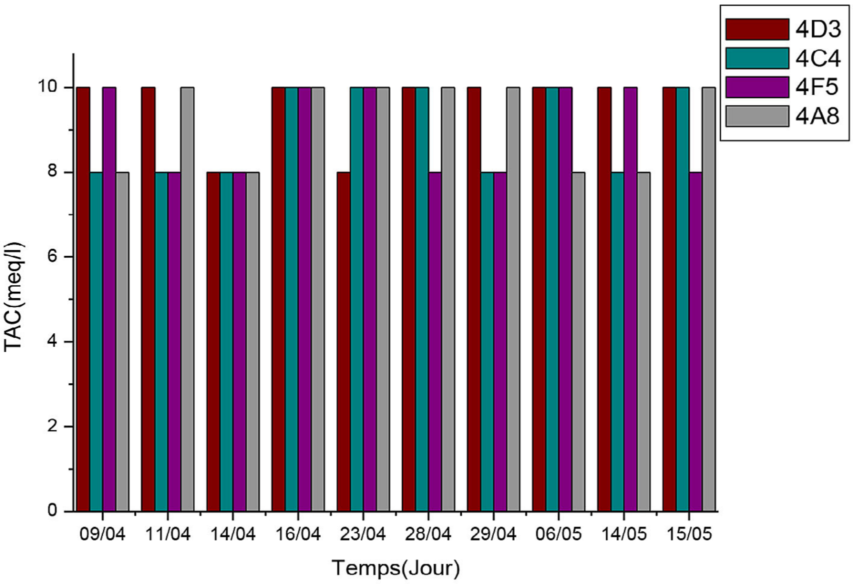


Figure 5. Total alkalinity (TA) variation of purge waters over time for tanks 4D3, 4C4, 4F5, and 4A8

Hydrotimetric titre (HT)

The hardness of water essentially corresponds to the sum of concentrations of alkaline earth cations such as calcium ions and magnesium ions. Figure 6 illustrates the variation of HT over time during the two months of sampling for the four oil tanks. According to the results illustrated in Figure 6, it is observed that the total hardness of purge waters fluctuates between (45 and 97.13) meq/l. These values significantly exceed the WHO standard of 3 meq/l (150 mg/l of $CaCO_3$). Therefore, these waters can be considered as very hard water. This could lead to the production of calcium bicarbonate precipitate ($Ca(HCO_3)_2$) or scale.

Chemical oxygen demand (COD)

The COD is an indication of the amounts of chemically oxidizable organic substances present in water. Figure 8 illustrates the variation in COD over the sampling time for the four studied oil tanks 4D3, 4C4, 4A8, 4F5. The values of COD in the purge waters, as illustrated in Figure 7, vary from one tank to another, ranging between 192 mg O_2/l and 4793.5 mg O_2/l. These COD values exceed the Algerian discharge standards of 120 mg O_2/l, as well as the standards of the Official Journal of the European Community (125 mg O_2/l) and the WHO standards (90 mg O_2/l).

Chloride content

Chlorides are important inorganic anions present in varying concentrations in water, usually in the form of sodium salts (NaCl) and potassium salts (KCl). They are often considered an indicator of water pollution. Figure 8 illustrates the variation in chloride content over the sampling time for the four oil tanks.

Your summary accurately reflects the information. It states that, according to the histogram in Figure 8, the chloride concentration values are notably high, falling within the range of (1235.8 and 4354.87) mg/l. Additionally, it highlights that these concentrations surpass the WHO standard, which is established at 200 mg/l.

Sulfate ion content (SO_4^{2-})

The concentrations of sulfates in water are variable and depend on the proportion of mineral sulfates present in the subsurface. Most sulfates are soluble in water and can, however, be reduced to sulfides, volatilized into hydrogen sulfide (H_2S), precipitated into insoluble salt, or assimilated by living organisms. Figure 9 shows the evolution of sulfate ion concentrations over the sampling time for the four studied oil tanks 4D3.

According to Figure 9, it is evident that the sulfate concentrations recorded in the purge

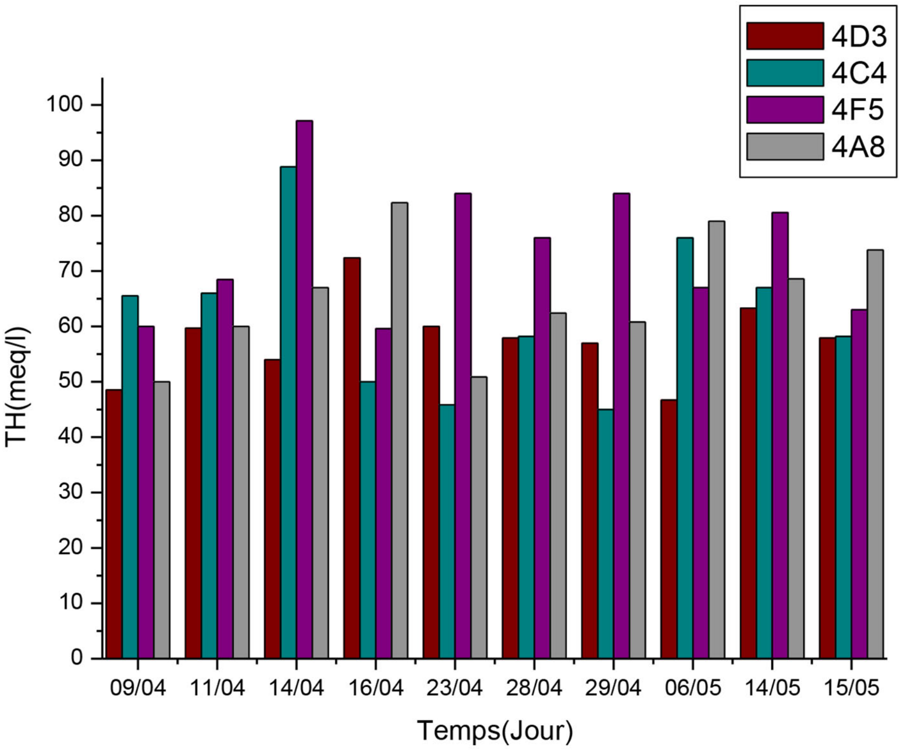


Figure 6. Variation in hydrotimetric titre (HT) of purge waters over time for tanks 4D3, 4C4, 4F5, and 4A8

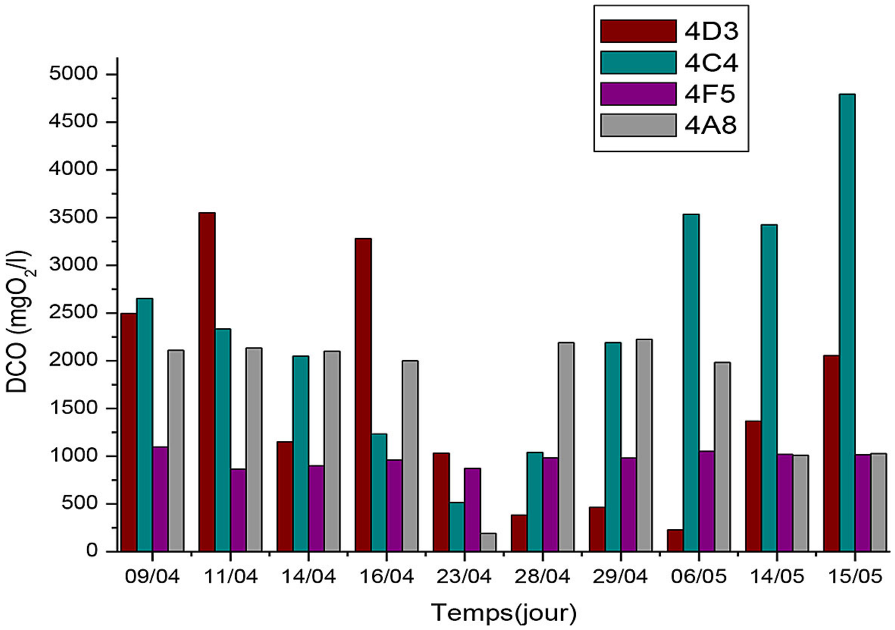


Figure 7. Variation in COD of purge waters over time for tanks 4D3, 4C4, 4F5, and 4A8

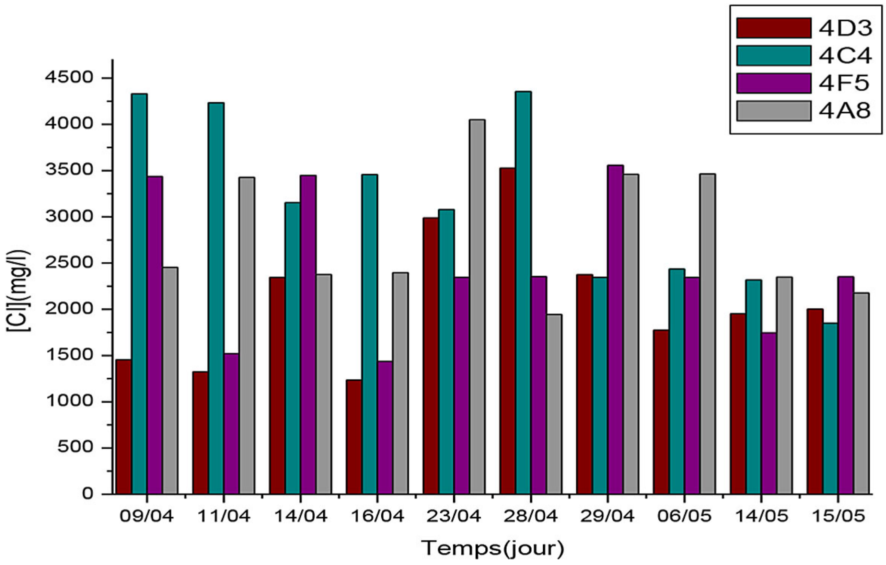


Figure 8. Variation in chloride concentration of purge waters over time for tanks 4D3, 4C4, 4F5, and 4A8

waters range between (4.789 and 57.112) mg/l. These values are below the permissible limit set by the WHO, which is 250 mg/l.

Naphthalene content

Figure 10 shows the evolution of naphthalene content over the sampling time for the four studied oil tanks.

According to these results, it is observed that naphthalene concentration values vary from one tank to another, ranging between (0.170 mg/l and 2.95 mg/l). All these values exceed the Algerian standard set at 1.17 µg/l for industrial wastewater. Therefore, it can be concluded that the purge waters are heavily laden with naphthalene.

Heavy metal content in purge waters

This study highlighted the presence of heavy metals in the purge waters, namely chromium, lead, and zinc. It is noteworthy that two other elements, nickel and cadmium, were also analyzed. Their concentrations were found to be

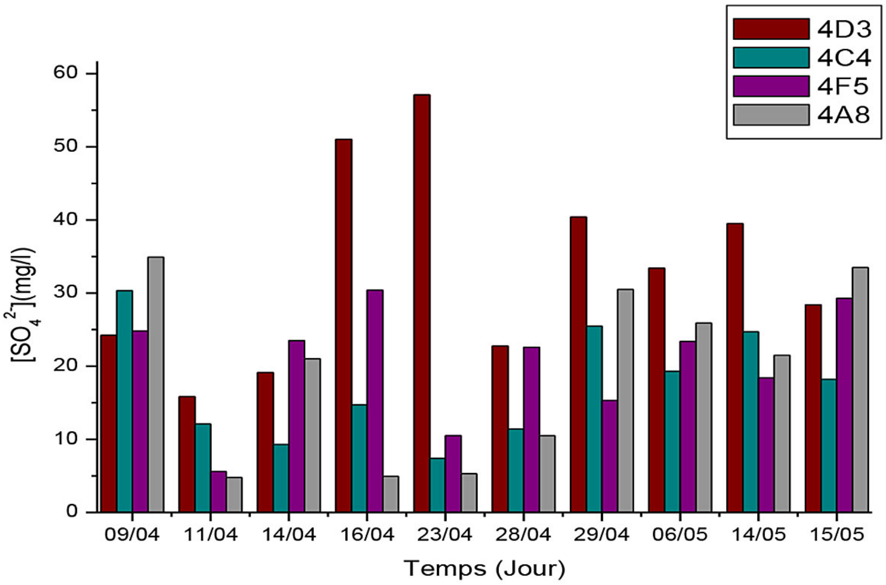


Figure 9. Variation in sulfate ion content of purge waters over time for tanks 4D3, 4C4, 4F5, and 4A8

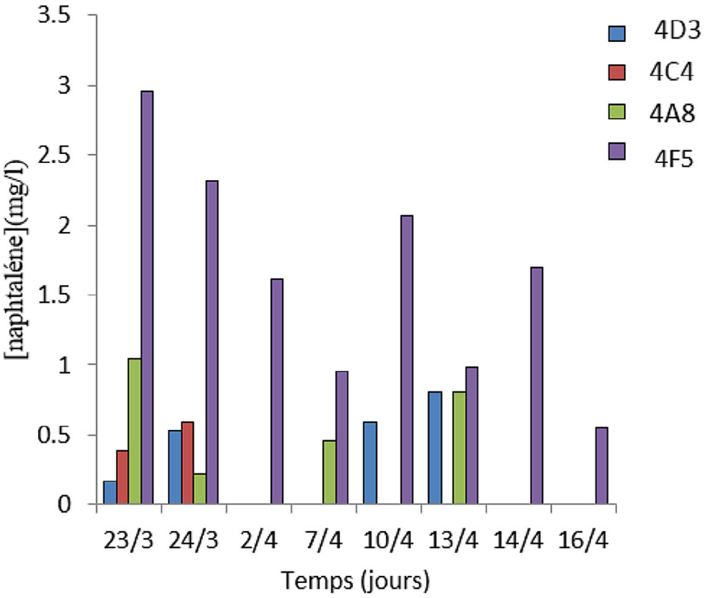


Figure 10. Variation in Naphthalene content of purge waters over time for tanks 4D3, 4C4, 4F5, and 4A8

below the detection limit for all purge water samples. However, the results for the concentrations of the other ions (chromium, lead Pb, and zinc are presented below.

Chromium

Figure 11 depicts the evolution of hexavalent chromium content over the sampling period for the four studied oil tanks. The hexavalent chromium concentrations recorded in the four tanks show values fluctuating between (0.05 and 0.09) mg/l. They exhibit significant variability from one tank to another, and none of the values substantially exceed the regulatory limit (0.1 mg/l) required by Algerian discharge standards.

Lead

Figure 12 depicts the evolution of lead content over the sampling time for the four studied oil tanks. The analysis results of the purge waters indicate that their lead concentrations vary between (0.01 and 0.1) mg/l. The highest recorded values remain well below the regulatory limit (0.5 mg/l) required by Algerian discharge standards.

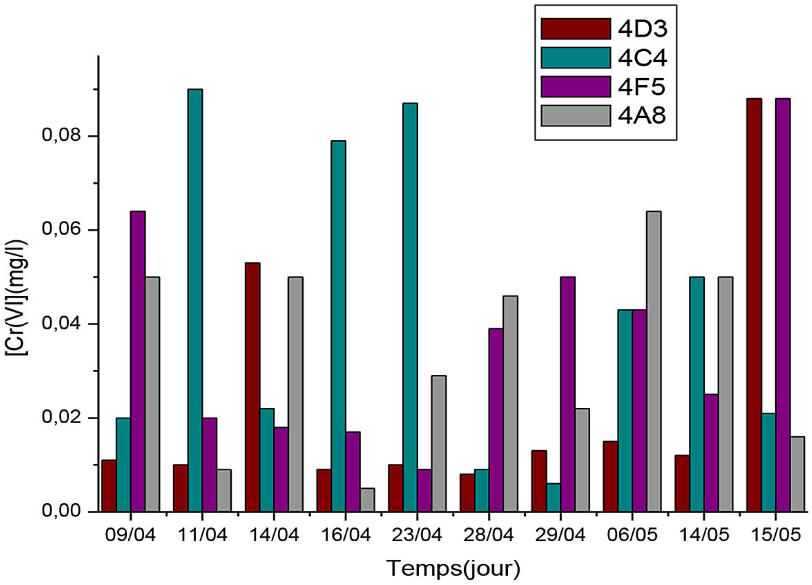


Figure 11. Variation in hexavalent chromium content over time for the specified

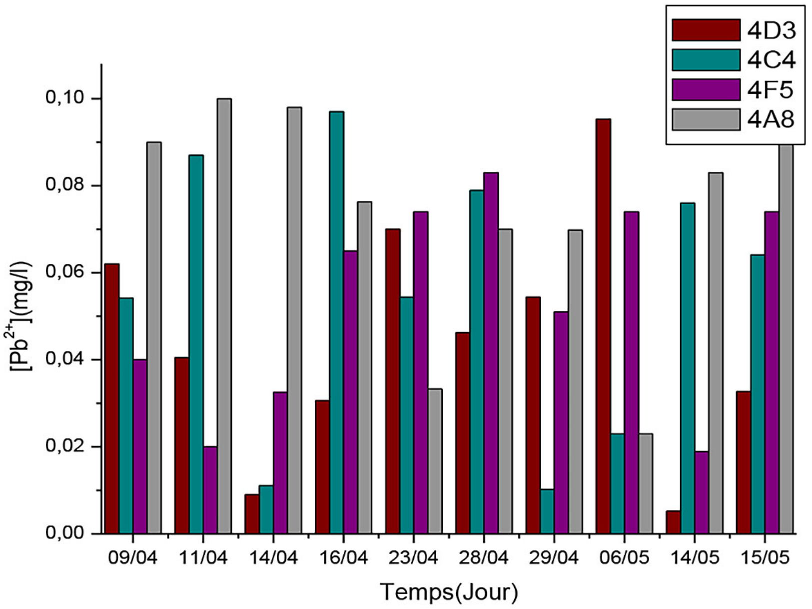


Figure 12. Variation in lead content of purge waters over time for tanks 4D3, 4C4, 4F5, and 4A8

Therefore, it is concluded that the purge water is not contaminated with lead.

Zinc

Figure 13 depicts the evolution of zinc content over the monitoring period for the four studied oil tanks. The zinc concentrations exhibit variable values during the study period. The recorded values on Figure 13 for tanks (4C4, 4F5, and 4A8) range between (0.39 and 2.87 mg/l) and are below the Algerian standard of 3 mg/l. However, elevated concentrations are observed in tank 4D3 with values ranging between (1.874 and 6.7) mg/l, significantly exceeding the Algerian standards. Therefore, the purge waters from tank 4D3 are contaminated with zinc.

Analysis of the composition of purge waters in organic matter

The infrared analysis allows the characterization of major functional groups constituting a compound or a set of compounds. This analytical technique can provide valuable insights into the presence of hydrocarbons in purge waters. Figure

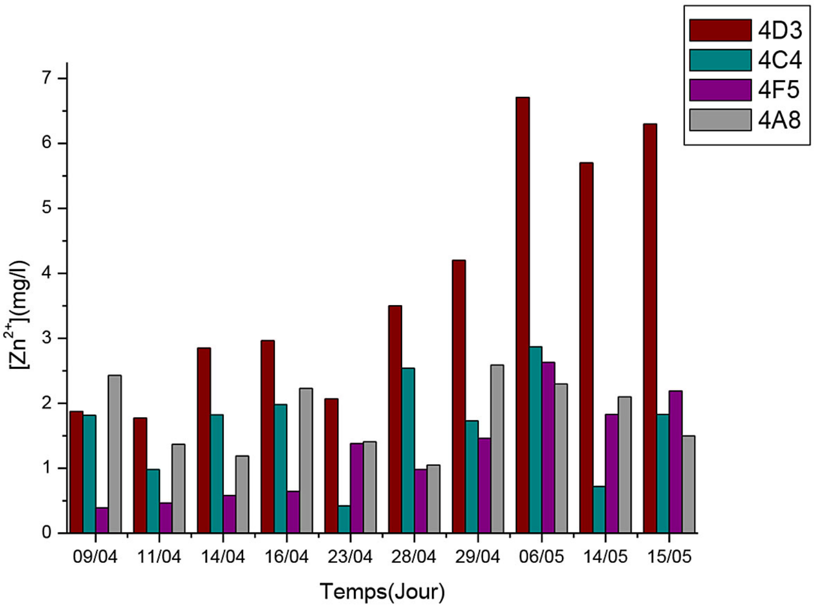
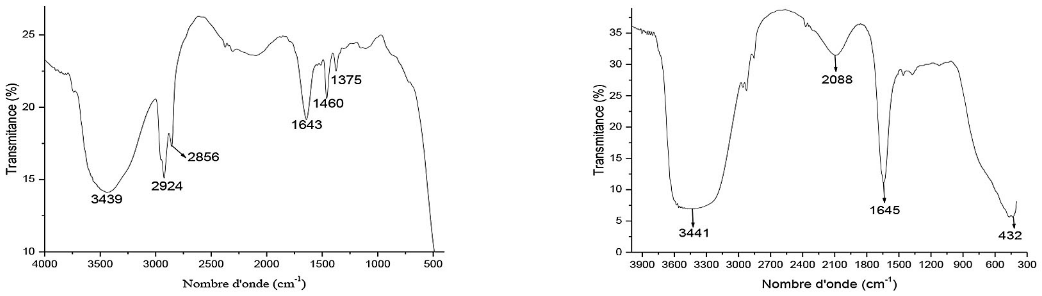


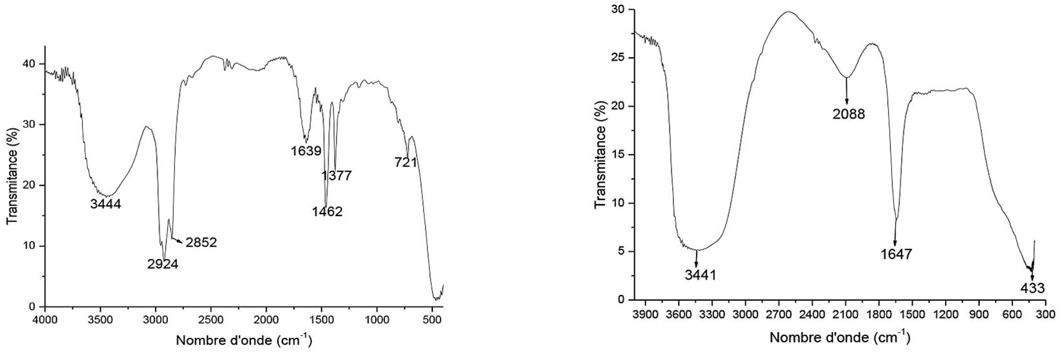
Figure 13. Variation in Zinc content of purge waters over time for tanks 4D3, 4C4, 4F5, and 4A8

14 displays the various IR spectra obtained by analyzing purge waters from tanks (4D3, 4C4, 4A8, and 4F5). This Figure reveals that the spectra show few differences among the various samples, and several bands can be attributed to different functional groups. The interpretation of these spectra is complex due to the mixture of organic and mineral matter. The broad band with a maximum around (3439, 3444, and 3441) cm^{-1} corresponds to hydroxyl groups of alcohols or acids.



a. 4D3

b. 4C4



c. 4A8

d.4F5

Figure 14. Infrared spectra of purge water samples for different storage tanks

Table 3. IR absorption bands of purge water samples for the studied tanks

Wavenumber (cm^{-1})	Vibration types	References
1643 and 1647	C=N	(Zhao-Yang *et al.*, 2010)
2088	Bond elongation C-N	(Lawrance *et al.*, 1991)
1375 and 1377	CH_2-CH_3Aliphatic	(Durand, 2003)
1645	C=C	(Zhao-Yang *et al.*, 2010)
432	M-O (M metallic element)	(Mehmet and Selahattin, 2006)

The two absorption bands around (2856, 2852, and 2924) cm^{-1} are attributable to the vibrations (C-H) of alkyl groups CH_2 and CH_3.

The band located between 1400 cm^{-1} and 1500 cm^{-1} with a maximum around (1460 and 1462 cm^{-1}) is of low intensity. This band may correspond to vibrations of aromatic C=C bonds; however, these are often not very intense. They can also correspond to vibrations of C-H out-of-plane in aromatic cycles (650–900 cm^{-1}) or alkenes (650–1000 cm^{-1}), or even the rotation of CH_2 in alkanes at 721 cm^{-1} (Durand, 2003). A band near 1639 cm^{-1}, attributed to water, frequently appears in a spectrum obtained through this technique. Other functional groups are illustrated in Table 3.

CONCLUSION

During this study, we conducted an overall diagnosis of the purge waters from the site under investigation. Characterization tests of these waters revealed a high and irregular pollution, both organic and mineral, which could be detrimental to the receiving aquatic environment (river, sea). For all these reasons, it is deemed necessary that these purge waters undergo pre-treatment before being discharged into the sea.

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