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Measuring pollution based on total petroleum hydrocarbons and total organic carbon in Tigris River, Maysan Province, Southern Iraq

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ABSTRACT
In the current study, the concentrations of total petroleum hydrocarbons (TPHs) in water and total organic carbon content in sediments were measured. A number of environmental factors, including water temperature, dissolved oxygen, pH, electrical conductivity, nitrates, and phosphates, have also been measured in the Tigris River deposits and in three selected stations on its course in Maysan Province in southern Iraq during the months of November, 2014, December, 2014, and January, 2015. The highest temperature, 25 °C, was recorded at Ali Al Gharbi District Station (St_1) in November, while the lowest temperature, 15°C, was recorded at Al-Amara District Station (St_2) in January. Moreover, the highest value of dissolved oxygen, 9.1 mg L^{-1}, was recorded in St_1 in January, while the lowest value, 7.5 mg L^{-1}, was recorded at the same station in December. All pH values were within the baseline trend for the duration of the study, with the highest level, 7.8, recorded at St_2 in November, while the lowest level, 7.05, at Al Majar Al-Kabir District Station (St_3) in the same month. The highest value for electrical conductivity was 2416 µs cm^{-1} at Al-Amara District Station in November, while the lowest value was 2000 µs cm^{-1} at the same station in December. The highest concentration of nitrate ion, 6.25 mg L^{-1}, was recorded at St_2 in November, while the lowest value, 3.8 mg L^{-1}, at St_1 in December. Moreover, the highest phosphate value, 0.59 mg L^{-1}, was recorded at St_2 in January, while the lowest, 0.049 mg L^{-1}, at St_3 in November. The highest rate of total organic carbon content was 2.15%, recorded at St_2 in November, while the lowest, 1.7%, at St_1 in the same month. The highest concentration of TPHs in the water was 5.22, recorded at St_2 in January, while the lowest, 2.85, at St_1 in November. The present study concluded that St_2 was heavily contaminated with organic matter. This station had the highest concentration of total petroleum hydrocarbons, which is mainly due to the increased population density beside the station, leading to increased human activities and the introduction of various wastes. These wastes contain nutrients and organic compounds, such as compounds containing petroleum derivatives, discharging to the water and sediment. Furthermore, there was a positive correlation between the organic carbon rate (%) and the concentration of petroleum hydrocarbons.

Keywords: water pollution, total petroleum hydrocarbons, total organic carbon, environmental factors, Tigris River.

INTRODUCTION
Water pollution is defined as changes in the physical, chemical, and life characteristics of the water directly or indirectly caused by human intervention through introducing unwanted materials or energy sources into the aquatic environment, causing disturbance or imbalance in the ecosystem, which adversely affects the living environment and the human existence. The growth of the global population has led to increased water demand and pollution (Fang 2019). Moreover, low to moderate pollution still poses a threat to humans and aquatic organisms (Edori 2019). Hydrocarbon compounds are common pollutants in water and have been reported to

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cause severe health effects in humans and the ecosystem (Basumatary *et al.* 2017). They are found in all environmental components and have the capacity for bioaccumulation. Some of these compounds are biodegradation-resistant, and hydrocarbons have high toxic effects, leading to carcinogenic mutagenic mutations (Shamilishvily *et al*. 2018). Oil hydrocarbon pollution is an issue that threatens the entire environment, since it can impact soil, surface water, and groundwater, while it can have significant consequences in terms of numerous health and environmental issues (Ossai *et al.* 2020). In addition, removing these pollutants from the environment is a real-world problem (Varjani 2017). Since the primary source of energy involves petroleum derivatives (Kafilzadeh *et al.* 2011), waste from plants and houses, as well as electrical power generators, public transport, oil tankers, and oil exchanges is a significant source for this type of pollutants (Adeniji *et al*. 2017a; Ogada *et al.* 2021). Moreover, dumping heavy industrial waste contaminates the river's water by various pollutants (Allafta & Opp 2020). The total petroleum hydrocarbons (TPH) content of water is the most important criterion for evaluating the relations between water and oil and gas deposits. Recently, analyzing these compounds in water has been used in petroleum exploration. Organic matter plays a crucial role in aquatic systems, affecting biochemical processes, nutrient cycling, biological availability, and chemical reactions (Niemirycz *et al*. 2006). Chemical interactions in the organic residue, caused by suspended organisms, represent the primary source of pollutants entering the water (O'connell *et al.* 2000). Furthermore, as they are highly nutritious, they go through degradation by microorganisms (Arzayus & Canuel 2005). Once these organisms die, they settle at the bottom, and they are exposed to chemical and bacterial degradation (Krishnanandan & Sriwafastmy 2013; Zubaidha 2019; Sulaimon 2020; Krishanamurthy 2020; Mirahmadi Babaheydari 2021).

Total organic carbon (TOC) in the sediment represents the amount of organic matter remaining after decomposition. Its value is influenced by factors such as primary productivity, precipitation rate, detritus resulting from the disintegration of organisms after death, and the rapid decomposition of organic matter by bacteria (Avramidis *et al.* 2015). The impurities found are typically of biological origin. However, anthropogenic contaminants from domestic and industrial waste and farming can also increase the number of organic compounds. Recent studies have confirmed that increased human activity and elevated nutrient supply will upraise the amount of TOC in the sediment (Chuan *et al.* 2016). There are several sources of organic pollution, including the release of sewage. Most urban settlements in third-world countries put sewage into nearby water bodies, particularly rivers, without treatment, thereby paving the way for severe environmental problems (Ribeiro *et al.* 2008). Some chemicals, such as various agricultural fertilizers (NPK), used to improve and increase agricultural production, as well as chemical pesticides, used to eliminate agricultural pests, are considered an important source of organic pollution (Voutsa *et al*. 2001), which is an essential issue for soil fertility in the ecosystem (Abbott & Manning 2015; Abdullatif & Wheib 2019). In addition, industrial wastewater dumped into nearby rivers or lakes without prior treatment is loaded with toxic chemicals and organic matter. The proportion of industrial wastewater has recently increased due to the expansion of industrial facilities and the increased application of chemicals, resulting in adverse effects on the water system (Kuyukina *et al.* 2020). The current study aimed to evaluate the monthly and locational changes in the concentrations of total petroleum hydrocarbons (TPHs) and total organic carbon in the Tigris River deposits. Moreover, the study measures a number of environmental variables in the river water, such as temperature, pH, electrical conductivity, dissolved oxygen, nitrates, and phosphates. In addition, to determine the ratio of the total content of the sedimentary organic carbon, the physical and chemical properties are studied.

MATERIALS AND METHODS

The study area

Three monthly sampling stations were selected for the current study, all located on the banks of Tigris River in Maysan Province, southern Iraq.

Ali Al Gharbi District Station (St_1): N: 32° 27′ 44.8 ″, E: 46° 41′ 15.41″

The station is located about 80 km northeast of Al-Amara City (the capital city of the province). The reduction in height continues throughout Tigris River in the left and right directions, so that the river continues to decline to the south, which is noted through the map of Iraq. This area is characterized by the spread of many forests and agricultural land along the banks of the river. In this region, many prominent aquatic plants, such as *Phragmites australis* and *Typha domingensis,* have been observed.

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Al-Amara District Station (St_2): N: 31° 50′ 03.9″ E: 47° 08′ 51.8″

The station is located in the center of Maysan Province. It is characterized by population density, which accounts for 47.8% of the province's total population. Furthermore, this site is characterized by fertile agricultural land on both sides of the river, and increased human activities characterize the region as a result of the increased population.

Al Majar Al-Kabir District Station (St_3): N: 31° 34′ 21.1″ E: 47° 10′ 23.8″

The station is located 29.86 km to the south of Al-Amara City (the capital of the province). The area is characterized by the proliferation of many agricultural lands, and there are grazing lands for herds of livestock on both sides of the river. The growths of many prominent aquatic plants, such as reeds,*Phragmites australis* , papyrus, *Typha domingensis* , and submersibles, such as *Ceratophyllum demersum,* are observed in all stations, located using a GPS device.

Collection of the Samples

Samples of water and sediment were collected on a monthly basis from the stations from November, 2014 to January, 2015 during the day, one time per month. The samples were collected using plastic bottles for the analyses of the physical and chemical parameters. The polyethylene bottles used in the study could hold 2 liters of water. The water samples for TPHs were collected in dark glass bottles with a capacity of 5 liters, and 50 mL of CCl_4 was added to these samples. Moreover, the samples of the sediment specimens were collected using Van Veen grab sampler from the middle of the river (IMRP 2006).

Measurement of the temperature

The water temperature was measured directly from the sampling sites using a mercury thermometer, expressed as °C (APhA 2005).

Measurement of pH

The pH values of the water were measured using an HI 8424 pH-meter, calibrated with standard buffer solutions at pH 4, 7, and 9 prior to the work (ASTM D1125-64, 1976).

Measurement of Electrical Conductivity

The electrical conductivity of the water samples was measured *on-site* using an EC meter (EC 215), expressed as $\mu s\ cm^{-1}$ prior to the work (Mackereth *et al*. 1978).

Measurement of the Phosphate Levels

The phosphates in the water sample were measured according to the APHA (1992). The water samples were collected using dark bottles with a capacity of 250-300 mL, kept at 4 °C, acidified using 1 mL of 50% H_2SO_4 and kept for further analyses. At first, the mixed solution was prepared by adding 21 g ammonium heptamolybdate $(NH_4)6Mo_7O_{24}.4H_2O$ and dissolving it in 250 mL distilled water (A). Then, we dissolved 0.05 g of antimony potassium tartrate ($KsbO.C_4H_4O_6$) in 100 mL distilled water (B). Thereafter, we poured both A and B solutions into a 2-L flask. Next, we slowly added one liter of 5N H_2SO_4 to the mixture, and diluted it to a 2-L volume. Finally, we dissolved 1.065 g ascorbic acid ($C_6H_8O_6$) in 200 mL distilled water, and added it to the resulting mixed solution. Afterward, we took 25 mL of the water sample and added 2.5 L of the reduced solution, followed by shaking the sample well, and leaving it to rest for an hour. Then, the sample was examined and measured using a spectrophotometer at a wavelength of 880 nm.

Measurement of the nitrate levels

The nitrates were measured according to the Ultraviolet Method described in APHA (1992) using a spectrophotometer (UV- 7200). At First, we took 25 mL of the selected sample and added 1 mL hydrochloric acid. Next, measurements were performed using a spectrophotometer at a wavelength of 220 nm and again at a wavelength of 275 nm. Then, the difference between the two readings was calculated, and the results were expressed in $\mu g\ L^{-1}$, as described in Equation 1:

$$NO_3(\mu g/L) = \text{Dilution} \times F \times (A - B) \qquad (\text{Eq. } 1)$$

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where A = nitrogen reading at the wavelength of 220 nm;
B = nitrogen reading at the wavelength of 275 nm; and
F = Factor = 4.43

Measurement of TPHs and Total Organic Carbon

The method used by UNEP (1989) was followed for measuring the concentration of Total Petroleum Hydrocarbons (TPHs) in the water. Moreover, the method proposed by Gaudette *et al.* (1974) was used to measure the content of total organic carbon (TOC), as summarized below:

At first, the sediment samples were air-dried at room temperature. After removing the suspended substances and impurities from the samples, they were dried in the oven at 60 °C. Afterward, 1 g of the dried sample was weighed and placed in a 500-mL beaker, followed by adding 10 mL 1 N potassium dichromate ($k_2Cr_2O_7$) solution using a pipette, and blending it well. Thereafter, we carefully added 20 mL concentrated H_2SO_4 using a dispenser, shook the beaker to mix the suspension, and allowed it to rest for 30 min. Afterward, we added 200 mL distilled water and 10 mL concentrated H_3PO_4. The mixture was allowed to cool, followed by adding 10 to 15 drops of diphenylamine indicator $(C_6H_5)_2NH$, and titration of the resulting mixture with 0.5 M ferrous ammonium sulfate ($(NH_4)_2SO_4$ $FeSO_4$. $6H_2O$) solution until the color changed from violet-blue to green. At the same time, we prepared two blank samples containing all the reagents, but without soil samples. The process for preparing the blank samples is exactly the same as the process used for the soil sample. Finally, the percentage of total organic carbon was calculated according to Equations 2 and 3.

$$\text{Oxidizable Organic Carbon } (\%) = \left[\left(V_{\text{blank}} - V_{\text{sample}}\right) \times 0.3 \times M\right]/\text{Wt} \qquad \text{(Eq. 2)}$$

$$\text{TOC}(\%) = 1.334 \times \text{Oxidizable Organic Carbon } (\%) \qquad \text{(Eq. 3)}$$

where M = Molarity of the $(NH_4)_2SO_4FeSO_4.6H_2O$ solution (about 0.5 M);
V_{blank}= Volume of the $(NH_4)_2SO_4FeSO_4$.6H2O solution required to titrate the blank (mL);
V_{sample}= Volume of the $(NH_4)_2SO_4FeSO_4.6H_2O$ solution required to titrate the sample (mL);
Wt = Weight of air-dried soil (g); and
$0.3 = 3 \times 10^{-3}$, where 3 is the equivalent weight of C.

Statistical analyses

By using the Two-Way ANOVA and Duncan Test using SPSS software (SPSS 19), the values were calculated. P values less than 0.05 were considered to be statistically significant.

RESULTS AND DISCUSSION

The highest temperature was 25 °C, recorded at St_1 in November, while the lowest was 15 °C at St_2 in January (Fig. 1). The results of the statistical analyses showed significant differences between the months of the study. However, there were no significant differences recorded ($p > 0.05$) between the selected stations. The highest dissolved oxygen value was 9.1 mg L^{-1}, recorded at St_1 in January, while the lowest was 7.5 mg L^{-1} at the same station in December (Fig. 2). The results of the statistical analyses showed no significant differences between the months of the study, except for Station 1 (LSD = 0.76), and there were no significant differences ($p > 0.05$) between the selected stations.

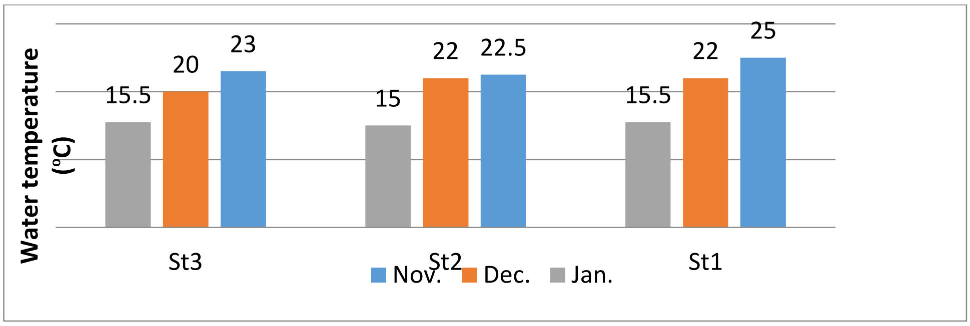

Fig. 1. Monthly and locational changes in water temperatures (°C) for the three studied station.

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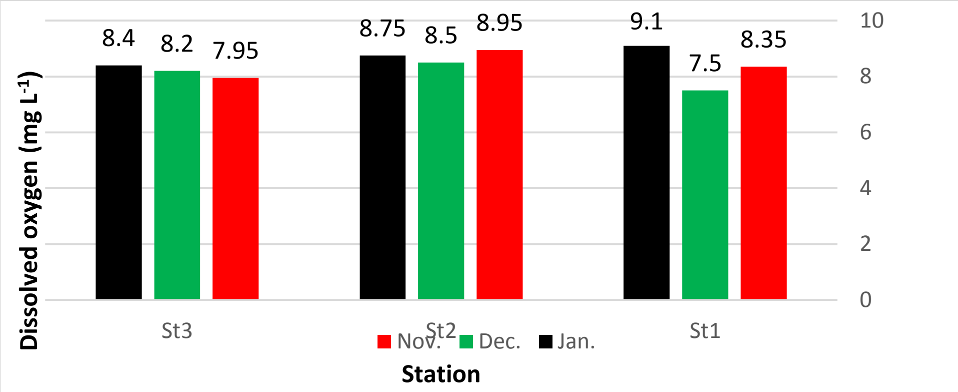

Fig. 2. Monthly and locational changes in dissolved oxygen values (mg L^{-1}) for the three studied stations.

Water temperature plays a vital role in the aquatic environment, affecting many physical, chemical, and biological properties in the aquatic environment by influencing the dissolving of gases and salts (Kennish 1986; Magaña 2020). Dissolved oxygen (DO) is vital for aquatic life. However, decomposing organic matter, dissolved gases, industrial waste, and agricultural runoff result in lower DO (Addo *et al*. 2013). The results show a negative correlation between DO and water temperature. In other words, the higher the temperature, the lower the value of DO, since dissolving of gases is inversely proportional to the temperature (Moodley *et al.* 2005).

According to the DO concentrations in the current study, Tigris River is considered healthy and suitable for the survival of aquatic organisms according to the limits allowed by the USEPA (1980; Table 1). All pH values are within the baseline trend throughout the study, with the highest, 7.8, recorded at St_2 in November and the lowest, 7.05, at St_3 in the same month (Fig. 3). The results of the statistical analyses revealed no significant differences between the months of the study (LSD = NS), and no significant differences were recorded between the selected stations ($p > 0.05$).

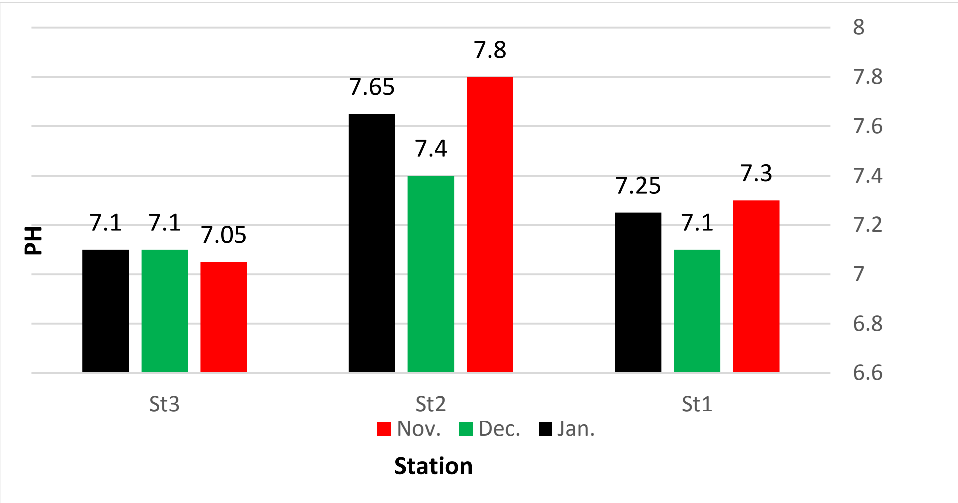

Fig. 3. Monthly and locational changes in pH values in different stations.

The results of the current study showed that pH values were within the weak base, consistent with the characteristics of Iraqi waters, ranging from 7.05 to 7.8. The pH is based on ions (H^+ and OH^-) in water and the quality of the dissolved gases, such as carbon dioxide, released into the aquatic environment as a by-product of organic degradation (McMahon *et al*. 2019; Al-Janabi *et al*. 2012). The results of the statistical analyses showed a positive correlation between pH values and water temperature, as more gases are dissolved by decreased temperature. The values from all the stations were within limits allowed by the USEPA (1980; Table 1). The highest electrical conductivity value was 2416 µs cm^{-1} at St_2 in November, 2014, while the lowest, 2000 µs cm^{-1} at the same station in December, 2014 (Fig. 4). The results of the statistical analyses showed no significant differences between the months of the study, except for St_3 (LSD = 1.6), although there were no significant differences ($P > 0.05$) between the selected stations. Moreover, the highest value of the electrical conductivity

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was at the station located in the Al-Amara district (Fig. 4), which was higher than the limits allowed by the USEPA (1980; Table 1). This may be due to a large volume of salts flowing into the river as a result of landslide (Shashi & Dwivedi 2009) and the increased population density in the capital city of the province (i.e., Al-Amara) in recent years.

Table 1. Some local and international specifications for drinking water.

Test	Unit	(USEPA 1980)	Current Study
pH	---	5.5-8.5	7.05-7.8
EC	µs cm^{-1}	1000	2000-2416
PO_4	mg L^{-1}	0.5	0.049-0.59
NO_3	mg L^{-1}	50	3.8-6.25
Temp.	C°	25	15-25
DO	mg L^{-1}	< 6	7.5-9.1
TOC	%	---	1.7-2.15
TPHs	µg L^{-1}	----	2.59-5.22

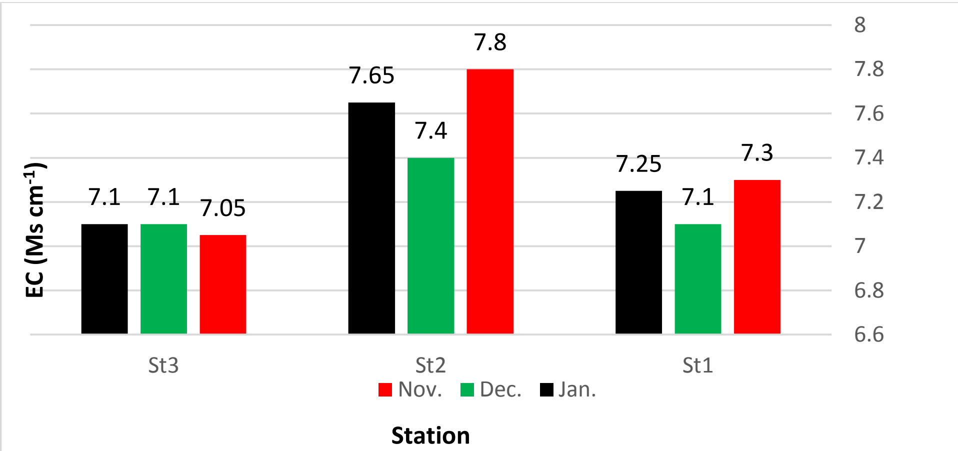

Fig. 4. Monthly and locational changes in electrical conductivity values (µs cm^{-1}) in the studied stations.

Regarding the monthly and locational changes in nitrate values for the three stations, the highest concentration of nitrate ions (6.25 mg L^{-1}) was recorded at St_2 in November, while the lowest (3.8 mg L^{-1}) at St_1 in December (Fig. 5). The results of the statistical analyses showed no significant differences between the months of the study, except for St_1 (LSD = 0.79). Moreover, there were no significant differences ($P > 0.05$) between the selected stations.

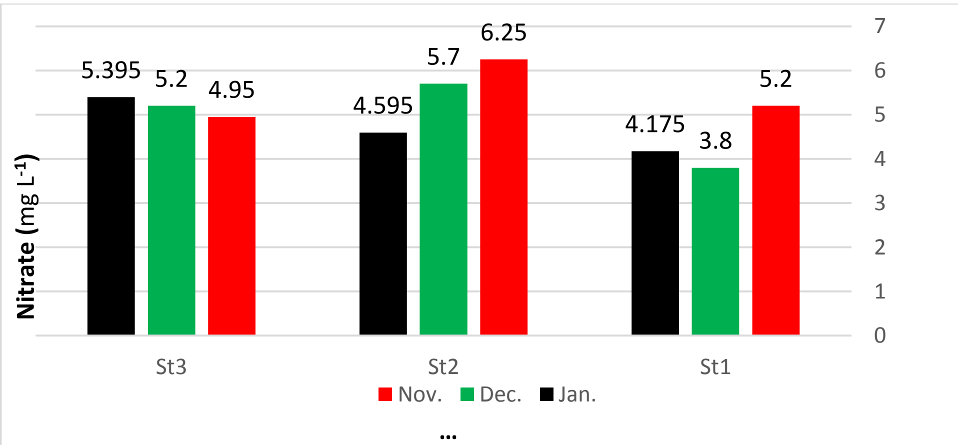

Fig. 5. Monthly and locational changes in nitrate values (mg L^{-1}) for the three studied stations.

Fig. 6 depicts the monthly and locational changes in phosphate values at different stations. As shown in this Fig., the highest phosphate value was 0.59 mg L^{-1}, recorded at St_2 in January, while the lowest, 0.049 mg L^{-1} at St_3 in November. The results of the statistical analyses showed no significant differences between the months of the study, and no significant differences were recorded between the selected stations ($p > 0.05$).

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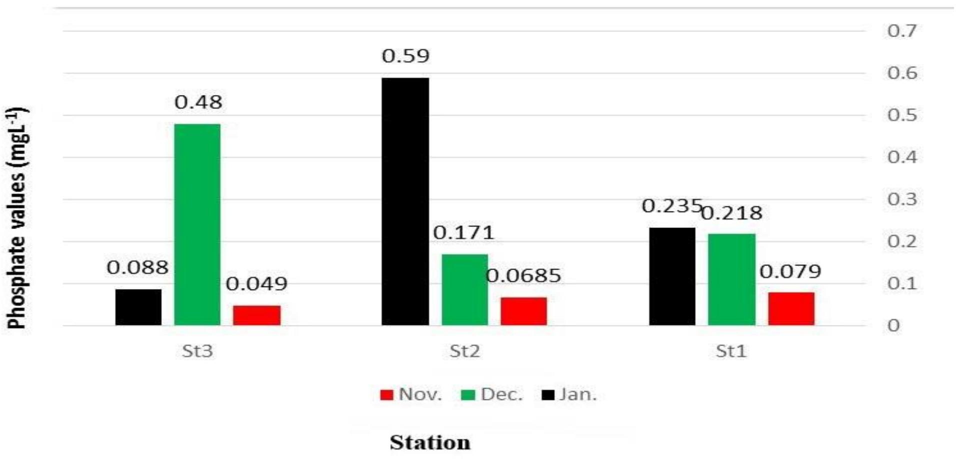

Fig. 6. Monthly and locational changes in phosphate values (mg L^{-1}) for the three studied stations.

The concentration of nitrate is used as an indication of micronutrients in water bodies, which can support plant growth. The high concentration of nitrate favors the growth of phytoplankton. Phosphate is a vital nutrient for all living organisms. However, excessive phosphorus in the form of phosphate in the aquatic environment may cause algae to bloom and make plants grow excessively, thereby resulting in eutrophication (Gebreyohannes *et al.* 2015; Adesuyi *et al.* 2015; Allison *et al.* 2020). The highest value of nitrates and phosphates was found in the Al-Amara Station (Figs. 5 and 6), which may be due to the increased application of agricultural fertilizers, detergents, and washing powders containing these compounds, which discharge directly into the river (Walakira & Okot-Okumu 2011). On the other hand, the values of these substances were low at St_3 and St_2, which may be due to the low population density and human activities near these two stations. Noteworthy, the highest phosphate value was higher than the limits allowed by the USEPA (1980; Table 1).

Regarding the total organic carbon (TOC) ratios for the three stations, the highest value was 2.15%, recorded at St_2 in November, while the lowest was 1.7% at St_1 in the same month (Fig. 7). No significant differences were observed between the months of the study, except for St_1 (LSD= 0.14). In addition, there were no significant differences ($P > 0.05$) between the selected stations.

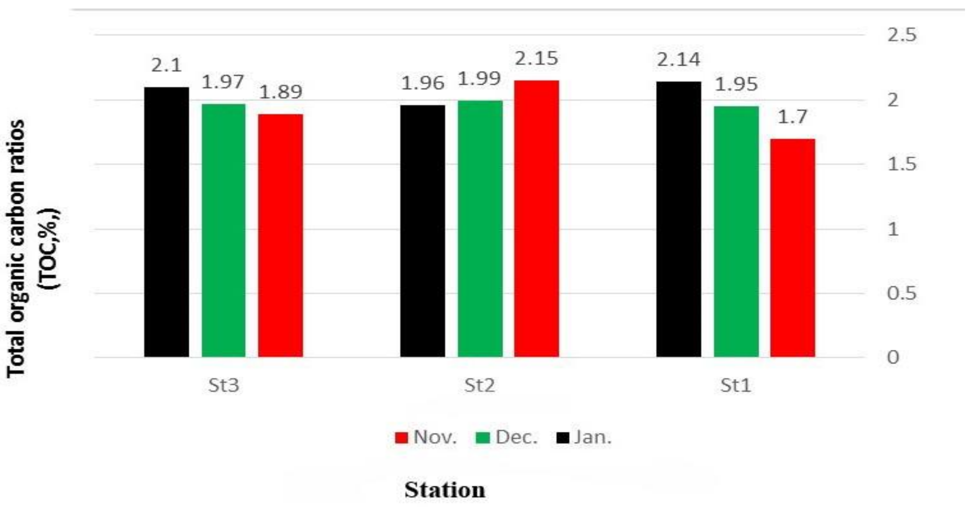

Fig. 7. Monthly and locational changes in the total organic carbon ratios (TOC, %) for the studied stations.

The total organic carbon estimation is a measure of the amount of organic matter in sediments. Its high concentration is evidence of organic pollution, which plays an essential role in the accumulation and release of various pollutants in the aquatic environment, such as hydrocarbon compounds (Al-Obaidy *et al.* 2010; Xu *et al.* 2017) found in the highest value in St_2 (Fig. 8), which may be due to the frequent introduction of sewage water rich in organic materials as a result of the increased population density beside this station. Furthermore, there is a

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correlation between temperature and the value of total organic carbon (Abdullatiff & Wheib 2019), since the degradation of organic matter increases by elevating the temperature. This plays an important role in the activity of organisms degrading organic matter in sediments (Niemirycz *et al.* 2006). However, this finding contradicts the results of Zhang *et al.* (2018). The highest concertation of TPHs in the water was 5.22, recorded at the Al-Amara District Station in January, 2014, while the lowest value was 2.85 at Ali Al Gharbi District Station in November, 2014 (Fig. 8). The results of the statistical analysis showed a number of significant differences between the months and the stations in the study.
By the increased global use of petroleum hydrocarbons as a major source of energy and raw materials in various fields, a wide range of pollutants have been released into the environment, affecting soil, surface water, and groundwater. These compounds are common pollutants in water and have been reported to result in severe health effects in humans and in the ecosystem (Ossai *et al*. 2020).

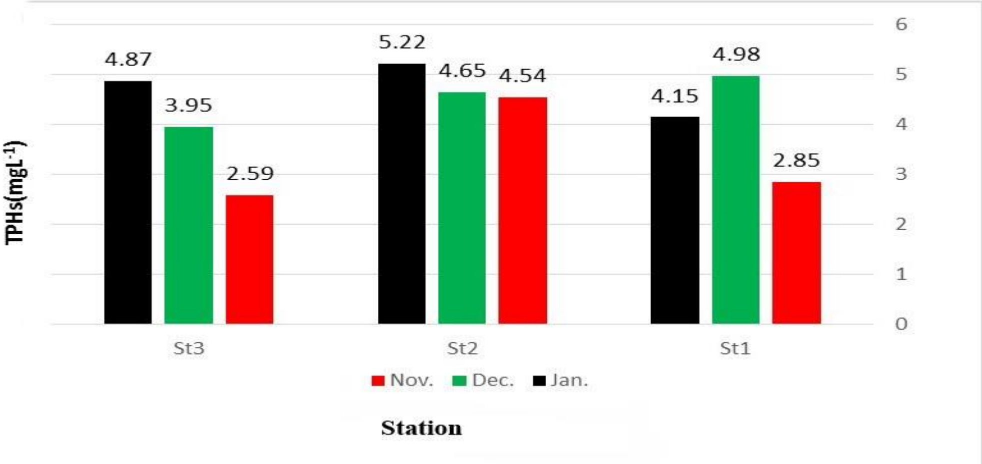

Fig. 8. Monthly and locational changes in TPHs concertation water for the studied stations.

The results showed that the highest concertation rates of total hydrocarbons in water were recorded in the second station. This may be due to a large volume of waste discharging into the river (Adeniji *et al.* 2017b) because of the increased population density and traffic beside this station, and the increased dumping of household waste. The consumption of fuel by engines and other human activities increase the concertation of these pollutants in the environment (Adeniji *et al.* 2019). Moreover, the concentration of these compounds was higher in January compared to the other months (November and December). This may be due to the decreased temperature in this month (Douabul *et al.* 2012). The effects of microbiological degradation of these compounds can also reduce in low temperatures (Marić *et al*. 2019).
The bacteria use the TPHs as carbon and energy sources. In contrast, the fungi produce enzymes that can catalyze oxidation reactions of the TPHs. In addition, the increased chances of rain in this period result in increased concentration of these types of pollutants in the air, which later reaches the surface water (Salata *et al.* 2019). These results are in line with the results of Jazza *et al. (*2016) and Ekanem *et al*. (2019), who reported that the highest concentrations of petroleum hydrocarbon compounds in the water found in winter (i.e., a rainy season). The sediments with a higher TOC content increase the share of pollutants in the potential mobile fraction (Baran *et al*. 2019). Accordingly, the current study has shown a positive correlation between the percentage of organic carbon and the concertation of petroleum hydrocarbon and also dissolved oxygen, all of them exhibit a negative correlation with temperature.
The most important recommendations of the current study include considering the environmental aspects when planning an industrial project; adopting a strict policy on dumping untreated waste from various industrial, agricultural, and service sectors into the river; preventing the establishment of factories that fail to deal with chemicals and toxic substances on the banks of rivers; and working toward changing the drainage of sewage to areas far from the river to mitigate the dangers of water pollution. Environmental assessment needs continuous work for many years to give a clear picture as the limited time of the study restricted the ability of the authors to determine the precise state of the environmental situation.

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CONCLUSIONS

The results indicated that Tigris River at Al-Amara City is contaminated with total petroleum hydrocarbons (TPHs) and organic matter in some selected stations due to the increased population density and traffic. Moreover, the results revealed that the values of some environmental factors exceeded some international limits set forth by the USEPA (1980), such as phosphates and electrical conductivity. Despite the deterioration of the river water quality in the capital city of the province (i.e., Al-Amara District Station) due to the increased flow of pollutants into the river, the ventilation of the river was good and suitable for aquatic life.

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