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Research Article

Physicochemical Analysis and PAHs Determination of Waste Water Discharged from Romi River, Kaduna State, Nigeria

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Abstract

This study evaluated the physicochemical properties and polycyclic aromatic hydrocarbon (PAH) contamination of Romi River, which receives effluent from the Kaduna Petroleum Refinery Company (KPRC). Water samples collected from selected upstream, discharge, and downstream locations were analyzed using standard physicochemical procedures and Gas Chromatography–Mass Spectrometry (GC–MS). The findings revealed acidic water conditions (pH 5.63 ± 0.26), high turbidity (455.00 ± 20.82 NTU), and elevated biochemical and chemical oxygen demand (BOD_5: 19.50 ± 9.85 mg/L; COD: 71.75 ± 37.04 mg/L), suggesting considerable organic pollution. The dissolved oxygen concentration (4.68 ± 1.23 mg/L) also indicated potential oxygen depletion, while detectable oil and grease (1.20 ± 0.50 mg/L) suggested petroleum-related contamination. GC–MS analysis identified six out of thirteen targeted PAHs, with a total concentration of 368.945 µg/L. Naphthalene was the predominant compound (360.443 µg/L), exceeding the stated World Health Organization drinking-water guideline, while the remaining detected PAHs occurred at considerably lower concentrations. The dominance of low-molecular-weight PAHs suggests petroleum-related contamination. Overall, the findings indicate that Romi River is affected by physicochemical deterioration and hydrocarbon pollution despite the prolonged inactivity of the refinery. The study therefore emphasizes the need for regular water quality monitoring, effective effluent management, and improved wastewater treatment to mitigate potential ecological and public health risks.

Keywords: *Romi River, Refinery Effluent, Polycyclic Aromatic Hydrocarbons, Petroleum Contamination, Water Quality, GC–MS.*

1.0 Introduction

Industrialization is an important factor in the development of a nation's economy; however, the wastes discharged from industries are disastrous to the environment in such a way that surface water, ground water, air, and soil are contaminated. Petroleum refineries generate large volumes of untreated or partially treated wastewater effluent, which is discharged into the environment through the process of refining crude oil. River systems that are located closer to the industries become where these effluents are disposed. These effluents from industries have a great deal of influence on the pollution of the water body, which alters the physical, chemical, and biological nature of the receiving water body (Ishaya, 2016). Petroleum refining industries generate large quantities of wastewater containing organic and inorganic pollutants, including petroleum hydrocarbons, suspended solids, nutrients, heavy metals, and polycyclic aromatic hydrocarbons (PAHs). phenols (creosols and xylenols), metals and their derivatives, ammonia, suspended solids, and sulphides, etc.

These contaminants can significantly alter the physicochemical characteristics of receiving water bodies and pose serious ecological and public health risks (Adeniji et al., 2019). Refinery effluents may increase turbidity, conductivity, biochemical oxygen demand (BOD), chemical oxygen demand (COD), and concentrations of toxic substances in surface

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waters. Polycyclic aromatic hydrocarbons (PAHs) are compounds made up of carbon and hydrogen atoms only in a form of fused aromatic rings that are bonded linearly (e.g., as anthracene) or angularly (e.g., dibenzo (a, h) anthracene) or clustered (e.g., pyrene) (Albayati and Kalash, 2019; Mojiri *et al.,* 2019; Onwuka *et al.,* 2020; Hedayati and Li, 2020).

PAHs are released to the environment through natural processes such as; forest fires, volcanic eruptions, and anthropogenic activities such as; incomplete combustion of fossil fuel, petroleum spills, emissions from power plants and wastewater effluent discharge from other industries such as, coal mining, petrochemical plants, coking and metal industries (Albayati and Kalash, 2019; Mojiri *et al.*, 2019). Due to their long transport potential and resistance to biodegradation, they tend to be ubiquitous and persistent in the environment for long periods of time. They have been detected in different media, including air, soils, sediments, water, wastewater, as well as food and aquatic organisms (Al-Agroudy *et al*., 2019; Pan *et al*., 2015; Itodo *et al*., 2018; Chen *et al*., 2019; Olayinka *et al*., 2018; Honda and Suzuki, 2020; Asagbra *et al*., 2020). Assessment of physicochemical parameters provides valuable information on the overall condition of water bodies, while determination of PAH concentrations serves as an indicator of petroleum-related pollution. Therefore, integrating physicochemical analysis with PAH determination offers a comprehensive approach for evaluating the environmental impact of refinery effluent discharge on Romi River.

2.0 Materials and Methods

2.1 Materials

2.1.1 Equipment

Gas chromatograph equipped with a mass spectrophotometer detector (GC-MS) Model 7683 series (Agilent Technologies Inc., Wilmington, DE, USA), Synergy UV Milli-Q water purification system from Millipore, Centrifuge machine (Beckman Coulter J2-21, Palo Alto California USA). ICP-AES (Perkin Elmer Optima 2100 DV, Shelton USA), Hydrolab Multiparameter (probe MS-5), HI98311 EC/TDS/Temperature tester, BODTrak, DO meter (YSI Model 59 equipped with oxygen electrode type YSI 5905 DO Probe) and incubator (Model 205, Hach US). COD reactor (HI 839800 COD), VTX-3000L model Mixer UZUS10 (Harmony), rotary evaporator, vacuum filtration system, Oasis HLB solid phase extraction cartridges (Waters, Milford, USA), peristatic pump, orbital shaker, Terumo syringes (3mL), Agilent syringe filters PTFE (13mm, 0.2µm), amber glass vials.

2.1.2 Reagents

A sixteen (16) component standard mix of Environmental Protection Agency (EPA 525 PAH mix A) certified reference material (CRM), 500 µg/mL each component in dichloromethane that contains Naphthalene (Naph), Acenaphthylene (Acy), Acenaphthene (Ace), Fluorene (Fluo), Phenanthrene (Phen), Anthracene (Anth), Pyrene (Pyr), Fluoranthene (Flt), Chrysene (Chr), Benzo[a]anthracene (B[a]A), Benzo[k]fluoranthene (B[k]F), Benzo[b]fluoranthene (B[b]F), Benzo[a]pyrene (B[a]P), Benzo[ghi]perylene (B[ghi]P), Dibenz[ah]anthracene (D[ah]A) and Indeno [1,2,3-cd]pyrene (I[cd]P) was obtained fromSigma–Aldrich (Schnelldorf, Germany) with 99 % purity. The solvents; n-hexane, methanol, acetonitrile, dichloromethane, acetone were

2.2 Site Description

Romi River is one of the tributaries of River Kaduna, in Chikun local government area of southern part of Kaduna State. The river is located between Latitudes 10º 29' 25" N and Longitudes 7º 20' 26" E (Aliyu *et al.*, 2021; Liman and Rabiu 2018). It rises from the Rido, Kabusu, and Kujama Hill at elevation 882 meters, 685 and 847 meters above the mean sea level respectively. The river traverses Romi Village crosses the Abuja Road and eventually discharges into Kaduna River at Garko. This flows throughout the year, but its volume is reduced during the dry season (Aliyu *et al.,* 2021).

2.3 Sampling

Water samples were collected from Romi River Kaduna. It was collected from three selected locations (A, B C). The sample from each location was collected in a 1 L washed amber glass bottle with Teflon screw cap. The samples were transported on ice chest to the Instrumentation Laboratory of Kano State Polytechnic. A composite sample was obtained by combining all the samples from the three locations in order to have a representative sample. The first sampling point was the upstream (C), which was located before the discharge of waste water treatment plant of KRPC enters the river. The second sampling point was at the refinery wastewater discharge point (A), before the refinery effluents water enters the river and the third sampling point was at the point just immediately after the refinery effluent mixed with the river water (B).

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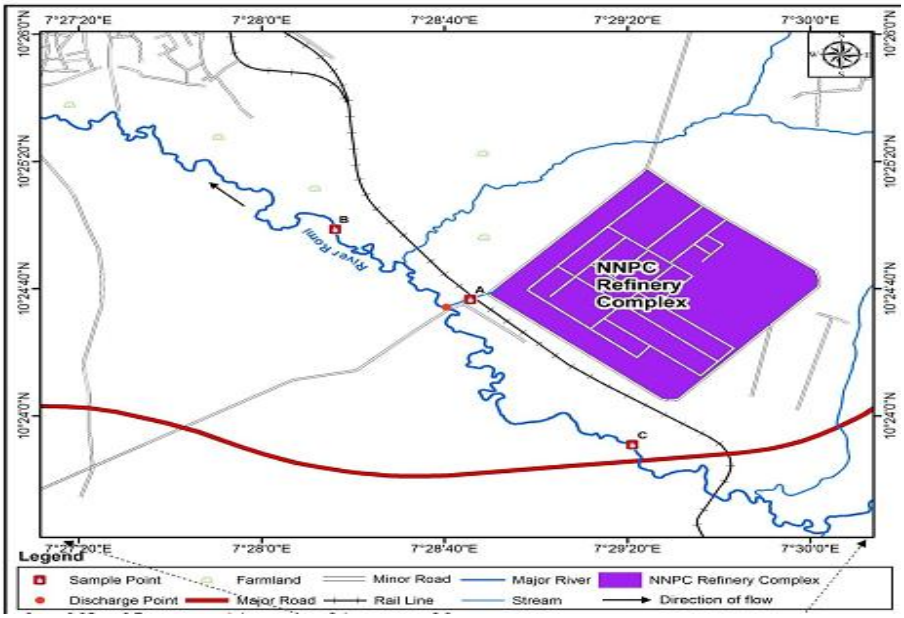


Figure 1: Map Showing the Sampling Points at River Romi (Ishaya, 2016).

2.3 Physicochemical Analysis of the Water Sample

The temperature, pH, turbidity and dissolved oxygen (DO) were determined using Hydrolab Multiparameter probe MS-5. Electrical conductivity and total dissolved solids (TDS) were determined also in-situ using HI98311 EC/TDS/Temperature tester. Biological Oxygen Demand (BOD) test measures the strength of the wastewater by measuring the amount of oxygen used by the bacteria as they stabilize the organic matter under controlled conditions of time (5 days) and temperature **(**20°C)**.** The standard method of 5 day incubation at 20°C, recognized by U.S. EPA, which is labeled as Method 5210B in the Standard Methods for the Examination of Water and Wastewater as described by Dasgupta and Yildiz, (2016) **was employed**. The Chemical Oxygen Demand (COD) measures the concentration of organic matter in the sample, by using a chemical oxidant such as potassium dichromate ($K_2Cr_2O_7$). Oxidizable organic compounds in the sample reduce the dichromate ion (orange) to the chromic ion (green). The amount of chromic ion formed is determined. The COD of the water sample was determined using HI 93754C-25 COD reactor (150°C) according to the procedure described in the instrument manual (Hanna Instruments, 2017) based on EPA approved method (410.4).

2.5 PAHs Standard Stock Solutions

The certified reference material standard solutions (EPA 525 PAH mix A) were used as the stock solution.

2.6 Calibration

Ultra-pure water was used for reconstitution of all solutions. The calibration standard was prepared by serial dilution from the CRM standard with the concentration of each component as 500 µg/mL at five concentration levels; 25, 50, 75, 100 and 125µg/mL. The working standard solutions were freshly prepared every day. Calibration graphs for SIM mode were plotted using five points in the range of the above concentrations and was used for quantification of the extracts. Calibration equation obtained in SIM mode and R^2 values are presented in Table 3.6.

2.7 PAHs Extraction Methods

The water sample was extracted for PAHs determination using two different methods; liquid-liquid extraction (LLE) and solid phase extraction (SPE). The SPE procedure described by Wang, (2012) with modification was employed which involve the following steps:

i. Conditioning the SPE cartridges with 5 cm^3 of dichloromethane (DCM), followed by deionized water then with 5cm^3 of methanol, drained but was not allowed to dry.
ii. The sample was then pumped through each cartridge at a flow rate of 1 cm^3 min^{-1}, using SPE unit vacuum pump, then the cartridges were allowed to dry for about 30 minutes, then rinsed with 1cm^3 of 5% mixture of methanol/deionized water. The cartridges were vacuumed again to remove the moisture before eluting with 6cm^3 DCM.
iii. The extracts were dehydrated using anhydrous sodium sulfate and the volume reduced to 1ml under nitrogen flow.

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iv. For further purification, the samples were filtered using Terumo syringe (3 cm^3) fitted with Agilent syringe filters PTFE (13mm, 0.2µm) prior to GC-MS analysis.

The extraction of PAHs from the water sample using LLE method was carried out according to modified procedure of Joshua *et al.,* (2016) whereby 10 cm^3 of the sample in a separatory funnel was extracted three times using 3, 2 and 1cm^3 of dichloromethane with vigorous shaking. The extracts obtained were treated as in the SPE extraction above.

2.8 Quantification and Method Validation

The spiked recovery method was adopted for validation studies for the water samples. Three replicates were spiked with 10 µL of 500 µg/mL stock standard solution consisting of 16 PAHs to 10 mL of pre-extracted waters. Ultrapure water was first pre-extracted in triplicate with 6 mL dichloromethane as a blank sample. The spiked samples were then extracted and analyzed. Recoveries for all matrices were obtained by comparing the concentration of the spiked to the non-extracted standard solutions concentration (the concentration of blank was subtracted). The peak area and retention time for each compound were used for identification and quantification of the analyte of interest (PAHs).

2.9 Chromatography Analysis

Gas Chromatography Mass Spectrometry (GC–MS) analyses were performed on an Agilent Technologies (USA) system consisting of a Trace GC 7890B gas chromatograph, an Auto Liquid Sampler (G4513A), and Mass Spectrometer (5977A MSD USA). The Trace Finder software from Agilent Technologies was used for data processing. A DB5-MS (30 m x 0.25 mm, 0.25 µm) gas chromatography column was used to separate PAHs (Agilent, USA). Helium was used as a carrier gas at 1 mL/min. The following temperature program was used: isothermal at 70 ℃ for 1 min, then temperature was increased by 10℃ /min to 270℃, then by 2 ℃ /min to 280 ℃, then held for 3 min, then increased by 2 ℃ /min to 310 ℃, and finally held for 1 min. The GC was interfaced by a heated transfer liner (310 ℃) to the mass spectrometer in electron ionization mode with electron energy of 70 eV. Nitrogen was used as the collision gas at the rate 1 mL/min. The criteria for the identification of PAHs were both the same retention times as the standard within ± 2%, and correct relative abundance of two characteristic ions within ±15 %. (Okoronko *et al*., 2014).

3.0 Results and Discussion

This chapter presents the results of the physicochemical analysis for the four sampling points and polycyclic aromatic hydrocarbon (PAH) determination of composite water samples collected from Romi River receiving Kaduna Refinery effluent. The results are discussed in relation to World Health Organization (WHO), National Environmental Standard Regulation and Administration (NESREA), Federal Environmental Protection Agency (FEPA), and United States Environmental Protection Agency (USEPA) standards where applicable. The findings provide insight into the influence of refinery effluent discharge on the water quality of the river and the potential environmental implications. Table 3.1 presents the physicochemical characteristic of the water samples.

Table 3.1: Physicochemical Parameters of the Water Samples from Romi River

Parameters	Unit	Composite	Discharge point	Downstream	Upstream
PH	-	5.9	5.9	5.4	5.8
Temperature	ºC	25.0	25.5	24.0	25.5
Turbidity	NTU	460	480	450	430
TDS	mg/L	1000	1600	800	400
TSS	mg/L	26	32	26	27
Conductivity	µS/cm	054	054	052	054
Oil &Grease	mg/L	1.20	1.84	1.13	0.61
Dissolved Oxygen	mg/L	4.8	3.2	6.43	6.70
Biological Oxygen Demand	mg/L	18	32	4.37	3.43
Chemical Oxygen Demand	mg/L	65	120	66.50	45.17

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Discussion of Results

pH

The pH values ranged from 5.4 to 5.9 across the sampling locations. The lowest pH values were recorded at the discharge point and downstream locations (5.4), while the highest value was observed in the composite sample (5.9). These values indicate slightly acidic conditions in the river. The acidic nature of the water may be attributed to refinery effluent constituents and the decomposition of organic matter. Acidic water conditions can influence metal solubility and adversely affect aquatic organisms (Chapman, 1996).

Temperature

Water temperature varied between 24.0 and 25.5°C. The relatively small variation among sampling points suggests that the effluent discharge had minimal influence on the thermal characteristics of the river. The temperatures recorded are typical of tropical surface waters and are within ranges that can support aquatic life. Temperature is an important factor affecting dissolved oxygen levels, metabolic activities of aquatic organism and chemical reactions in aquatic ecosystem (Wetzel, 2001).

Turbidity

Turbidity values were extremely high, ranging from 430 to 480 NTU. The highest turbidity was recorded at the discharge point (480 NTU), while the lowest value occurred upstream (430 NTU). Elevated turbidity may result from suspended particles, industrial discharges, and surface runoff entering the river system. High turbidity reduces light penetration into water, thereby affecting photosynthetic activity and aquatic productivity (APHA, 2017).

Total Dissolved Solids (TDS)

The concentration of total dissolved solids ranged from 400 to 1600 mg/L. The highest concentration was recorded at the discharge point (1600 mg/L), indicating a significant input of dissolved substances from refinery wastewater. The upstream location had the lowest concentration (400 mg/L), suggesting relatively lower anthropogenic influence. Elevated TDS levels may alter water taste, increase salinity, and affect aquatic ecosystems (WHO, 2022).

Total Suspended Solids (TSS)

TSS values ranged from 26 to 32 mg/L. The highest concentration was observed at the discharge point (32 mg/L), reflecting the contribution of suspended particulate matter from refinery effluent. Increased suspended solids can reduce water clarity and interfere with aquatic habitats and feeding activities of aquatic organisms (Chapman, 1996).

Electrical Conductivity

Conductivity values ranged from 52 to 54 µS/cm, showing little variation among sampling locations. This suggests that the ionic composition of the river water was relatively uniform despite the influence of the refinery discharge. The low conductivity values indicate a relatively low concentration of dissolved ionic substances. Electrical Conductivity is commonly used as an indicator of the total ionic content of water and may reflect the influence of industrial discharges and geological characteristics of the watershed (APHA, 2017).

Oil and Grease

Oil and grease concentrations ranged from 0.61 to 1.84 mg/L. The highest concentration occurred at the discharge point (1.84 mg/L), while the lowest concentration was recorded upstream (0.61 mg/L). This pattern suggests the influence of petroleum-related activities on water quality. The presence of oil and grease in aquatic systems may impair oxygen transfer from the atmosphere and negatively affect aquatic organisms (United Nations Environment Programme UNEP, 2011).

Dissolved Oxygen (DO)

Dissolved oxygen concentrations ranged from 3.2 to 6.2 mg/L. The lowest value was recorded at the discharge point, whereas the highest value occurred upstream. The reduced DO concentration at the discharge point may be attributed to the decomposition of organic pollutants and oxidation of petroleum-derived contaminants, both of which consume dissolved oxygen. Low dissolved oxygen levels can cause stress to aquatic organisms and reduce biodiversity. Dissolved oxygen is an important indicator of aquatic ecosystem condition because it supports the respiration of aquatic organism and is affected by temperature, aeration, and the decomposition of organic matter (Chapman, 1996; Wetzel, 2001).

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Biological Oxygen Demand (BOD)

BOD values ranged from 18 to 32 mg/L. The highest BOD concentration was observed at the discharge point, indicating a high level of biodegradable organic matter. The upstream location recorded the lowest value, reflecting better water quality conditions. BOD increased from 3.43mg/L upstream to 4.37 mg/L downstream. Biological oxygen demand measures the oxygen consumed by microorganisms during the breakdown of biodegradable organic matter under specified test conditions. Elevated BOD values suggest increased microbial activity and greater oxygen demand within the aquatic environment, (Metcalf and Eddy, 2014).

Chemical Oxygen Demand (COD)

COD values ranged from 30 to 120 mg/L. The highest value was recorded at the discharge point, indicating the presence of substantial amounts of oxidizable organic and inorganic pollutants (APHA et al, 2023; Metcalf and Eddy, 2014). The lower COD value upstream suggests reduced contamination. The elevated COD concentrations near the discharge point further confirm the impact of refinery effluent on the river system.

Overall Assessment

The results demonstrate that the discharge point was the most impacted location, characterized by elevated turbidity, TDS, oil and grease, BOD, and COD concentrations, as well as reduced dissolved oxygen levels. The upstream station generally exhibited better water quality, while downstream values indicated partial dilution and recovery of the river after receiving the effluent. These findings suggest that refinery effluent discharge contributes significantly to the deterioration of water quality in the Romi River and may pose ecological risks to aquatic organisms and downstream water users. Similar observations have been reported for rivers receiving petroleum refinery wastewater, where organic and inorganic pollutants contribute to water quality deterioration and ecological stress (Oluwakemi *et al,* 2023; Liman and Rabi'u, 2018; Adekunle et al. 2013; Owamah et al. 2013).

Table 3.2: Comparison of Measured Values with WHO and NESREA Standards

Parameter	Range Obtained	WHO/NESREA Standard	Compliance Status
pH	5.4-5.9	6.5-8.5	Not compliant
Temperature ˚C	24.0-25.5	Ambient ± 3 ˚C	Compliant
Turbidity (NTU)	430-480	5 NTU (drinking water)	Not compliant
TDS (mg/L)	400-1600	1000	Partially compliant
TSS (mg/L)	26-32	30 (effluent limit)	Slight excedance at discharge point
Conductivity (µS/cm)	52-54	No specific WHO limit)	Within acceptable range
Oil and grease (mg/L)	0.61-1.84	10	Compliant
DO (mg/L)	3.2-6.2	≥ 5 for aquatic life	Partially compliant
BOD (mg/L)	8-32	30	Exceeded at discharge point
COD (mg/L)	30-120	90	Exceeded at discharge point

Discussion of Compliance with Regulatory Standards

Table 3.2 presents the comparison of the physicochemical parameters with WHO and NESREA standards.The pH values recorded in the study area were below the recommended WHO and NESREA range of 6.5–8.5, indicating acidic conditions within the river. Acidic waters can increase the mobility of toxic metals and may adversely affect aquatic organisms.

The turbidity values obtained from all sampling locations greatly exceeded the WHO guideline value of 5 NTU for potable water. Such high turbidity levels suggest a substantial load of suspended particles and indicate poor water clarity. High turbidity can interfere with water treatment processes and reduce light penetration required for aquatic photosynthesis.

Total dissolved solids exceeded the recommended limit of 1000 mg/L at the discharge point, suggesting significant contamination from refinery effluent. Elevated TDS may affect water palatability and alter the ecological balance of aquatic systems.

Although most TSS values were within acceptable limits, the discharge point slightly exceeded the recommended effluent standard, indicating the introduction of suspended matter into the river from industrial activities.

Oil and grease concentrations remained below the NESREA permissible limit of 10 mg/L; however, the elevated concentration observed at the discharge point compared with upstream locations suggests the influence of petroleum-related operations.

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Dissolved oxygen concentrations were below the minimum recommended value of 5 mg/L at some locations, particularly at the discharge point. Reduced dissolved oxygen levels can impair aquatic life and indicate increased organic pollution.

The BOD value recorded at the discharge point exceeded the recommended limit of 30 mg/L, indicating a high concentration of biodegradable organic pollutants. Similarly, the COD concentration at the discharge point exceeded the recommended standard of 90 mg/L, reflecting a substantial load of oxidizable contaminants.

Overall, comparison with WHO and NESREA standards indicates that refinery effluent discharge has adversely affected several aspects of water quality in the Romi River. Parameters such as pH, turbidity, dissolved oxygen, BOD, and COD reveal evidence of environmental stress and potential ecological risk within the study area. The discharge points consistently recorded the poorest water quality, confirming the influence of refinery wastewater on the receiving river system.

Mean ± Standard Deviation of Physicochemical Parameters

To provide an overall assessment of water quality across the sampling locations, the mean and standard deviation of each physicochemical parameter were calculated and are presented in Table 3.3.

Table 3.3: Mean ± Standard Deviation of Physicochemical Parameters

Parameter	Mean ± SD
PH	5.63 ± 0.26
Temperature ˚C	25.00 ± 0.71
Turbidity (NTU)	455.00 ± 20.82
TDS (mg/L)	950.00 ± 500.00
TSS (mg/L)	27.75 ± 2.87
Conductivity (µS/cm)	53.50 ± 1.00
Oil and grease (mg/L)	1.20 ± 0.50
DO (mg/L)	4.68 ± 1.23
BOD (mg/L)	19.50 ± 9.85
COD (mg/L)	71.75 ± 37.04

Discussion of Mean ± Standard Deviation

The mean pH value of 5.63 ± 0.26 indicates that the river water was generally acidic across all sampling locations, with relatively little variation among sites. The mean temperature of 25.00 ± 0.71°C suggests a stable thermal condition throughout the study area.

Turbidity exhibited a high mean value of 455.00 ± 20.82 NTU, demonstrating substantial suspended particulate matter within the river. Similarly, TDS showed a mean concentration of 950.00 ± 500.00 mg/L and the highest standard deviation among the measured physicochemical parameters, indicating considerable variation between sampling locations, particularly due to the elevated concentration observed at the discharge point.

The mean TSS value was 27.75 ± 2.87 mg/L, reflecting moderate levels of suspended solids with relatively small spatial variation. Conductivity recorded a mean value of 53.50 ± 1.00 µS/cm, indicating uniform ionic content across the river.

Oil and grease concentrations averaged 1.20 ± 0.50 mg/L, suggesting detectable petroleum contamination, with higher concentrations near the refinery discharge point. Dissolved oxygen recorded a mean value of 4.68 ± 1.23 mg/L, which is slightly below the level generally considered optimal for aquatic life, indicating possible oxygen depletion resulting from pollution inputs.

The mean BOD_5 and COD values were 19.50 ± 9.85 mg/L and 71.75 ± 37.04 mg/L, respectively. The relatively large standard deviations for these parameters indicate substantial differences among sampling stations and further support the influence of refinery effluent on the river's organic and chemical pollution load. The elevated BOD_5 and COD values

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observed near the discharge point suggest increased oxygen demand and the presence of oxidizable contaminants in the aquatic environment.

Overall, the mean ± standard deviation results reveal that turbidity, TDS, BOD_5, and COD were the most affected parameters, highlighting the impact of refinery wastewater discharge on the physicochemical quality of the Romi River.

Analysis of Variance (ANOVA)

A one-way Analysis of Variance (ANOVA) was conducted to determine whether significant differences existed among the physicochemical parameters measured at the upstream, downstream, discharge point, and composite sampling locations. The analysis was performed at a 95% confidence level ($\alpha = 0.05$).

The results of the ANOVA showed that some parameters exhibited significant spatial variations among the sampling stations ($p < 0.05$), indicating the influence of refinery effluent discharge on water quality. Parameters such as turbidity, total dissolved solids (TDS), biological oxygen demand (BOD_5), chemical oxygen demand (COD), and oil and grease showed greater variation between sampling locations, particularly at the discharge point.

Conversely, parameters such as temperature and conductivity exhibited relatively small differences among sampling stations and may not differ significantly ($p > 0.05$).

Table 3.4: Concentrations of PAHs in composite sample of Romi River Water

PAH	Rings	Concentration in Extract (µg/L)
Naphthalene (Nap)	2	360.443
Acenaphthylene (Acy)	3	5.298
Acenaphthene (Ace)	3	0.680
Fluorene (Flu)	3	0.685
Anthracene (Ant)	3	1.796
Phenanthrene (Phe)	3	ND
Benzo[ghi]perylene (BghiP)	6	0.043
Fluoranthene (Flo)	4	ND
Pyrene (Pyr)	4	ND
Benz[a]anthracene (BaA)	4	ND
Chrysene (Chr)	4	ND
Benzo[b]fluoranthene (BbF)	5	ND
Benzo[e]pyrene (BeP)	5	ND
ΣPAHs		368.945

Polycyclic Aromatic Hydrocarbon (PAH) Concentrations

Table 3.4 and Figure 3.1 presents the distributions of PAHs in Romi River, GC-MS analysis identified six PAHs out of thirteen target compounds analyzed in the water sample. The concentrations were calculated based on a 1 L sample volume concentrated to 2 mL extract. Chromatographic peaks were identified based on their retention times and mass spectral characteristics relative to calibration standards. Compounds detected below the method detection limit were reported as not detected (ND). The GC-MS report showed Naphthalene as the dominant PAH, while several high molecular weight PAHs were not detected (Figure 3.2). The concentration of Naphthalene exceeded the WHO guideline values, indicating substantial petroleum hydrocarbon contamination. Such elevated concentrations may pose ecological risks and may render the water unsuitable for domestic use without treatment.

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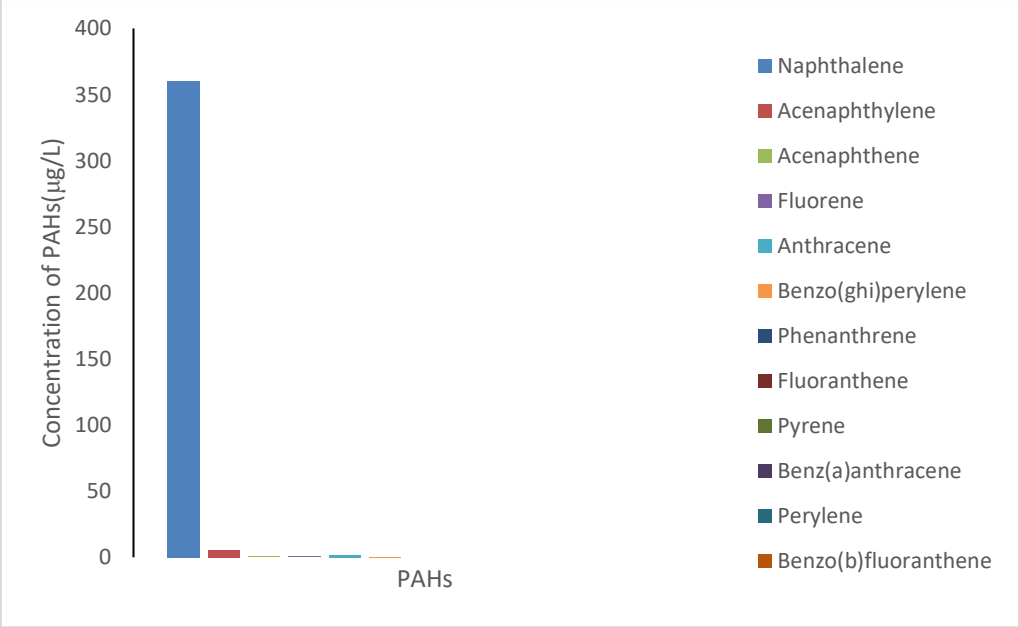


Figure 3.1: Distribution Pattern of PAHs in the Composite Sample

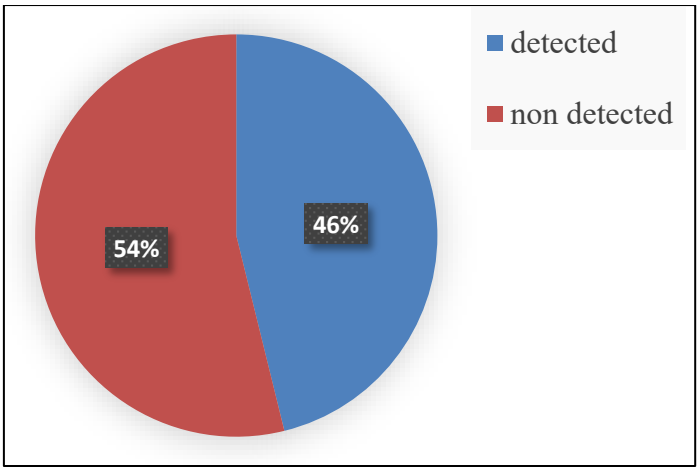


Figure 3.2: Pie chart showing the percentage of detected and Non-detected PAHs

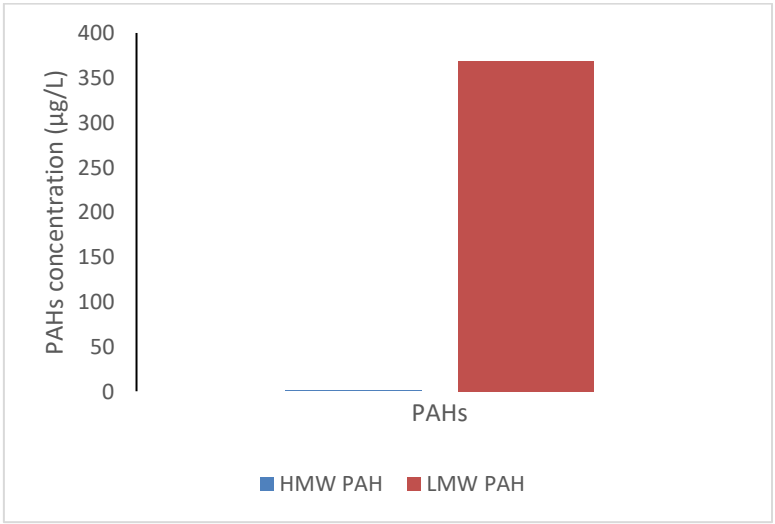


Figure 3.3: Distribution pattern of PAHs according to Molecular Weight

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Figure 3.3 presents the distribution pattern of PAHs according to molecular weight. PAHs are categorized into low molecular weight (LMW) and high molecular weight (HMW) compounds. Low Molecular Weight PAHs Detected were Naphthalene, Acenaphthylene, Acenaphthene, Fluorene, Anthracene (LMW PAHs = 368.902 µg/L, while the only High Molecular Weight PAHs detected was Benzo[ghi]perylene. (HMW PAH =0.043 µg/L. The overwhelming dominance of LMW PAHs indicates recent petroleum contamination and supports the influence of refinery effluent discharge on the river. The results showed that the contamination profile was dominated by low molecular weight PAHs. Five of the six detected compounds belonged to the LMW group, while only one HMW PAH (Benzo[ghi]perylene) was detected at a very low concentration.

Source Identification of PAHs

Source identification can be inferred from the molecular weight distribution of the detected PAHs. Petrogenic Sources: Petrogenic PAHs originate from Crude oil, Petroleum products, Refinery wastewater, Fuel spills and Lubricants. Pyrogenic Sources: Pyrogenic PAHs originate from Fossil fuel, combustion, Bush burning, Industrial combustion and Vehicle exhaust emissions. The predominance of Naphthalene and other low molecular weight PAHs suggests that the detected PAHs are predominantly petrogenic in origin. This observation is consistent with contamination arising from petroleum refinery activities and wastewater discharge into the river. The low abundance of high molecular weight PAHs further supports the conclusion that combustion-related (pyrogenic) sources contribute minimally to the observed contamination. The dominance of Naphthalene and other low molecular weight PAHs, coupled with the absence of most high molecular weight compounds, suggests that the contamination is predominantly petrogenic in origin. This observation is consistent with contamination resulting from refinery wastewater discharge crude oil spills and petroleum-related activities. (Oluwakemi *et al,* 2023).

Quality Assurance and Quality Control (QA/QC)

Table 3.6: Quality Assurance and Quality Control Parameters for GC-MS Analysis

PAH		Calibration Range (µg/L)	R^2
Naphthalene	Nap	20–5000	0.9950
Acenaphthylene	Acy	20–5000	0.9996
Acenaphthene	Ace	20–5000	0.9995
Fluorene	Flu	20–5000	0.9999
Anthracene	Ant	20–5000	0.9820
Phenanthrene	Phe	20–5000	0.9938
Fluoranthene	Flo	20–5000	0.9991
Pyrene	Pyr	20–5000	0.9991
Benz[a]anthracene	BaA	20–5000	0.9979
Perylene		20–5000	0.9989
Benzo[b]fluoranthene	BbF	20–5000	0.9801
Benzo[e]pyrene	BaP	20–5000	0.9890
Benzo[ghi]perylene	BghiP	20–5000	0.9904

Quality assurance and quality control procedures were implemented throughout sampling, sample preparation, and instrumental analysis to ensure the reliability of the results obtained.

All sampling containers were thoroughly washed with detergent, rinsed with distilled water, and pre-cleaned before sample collection. Water samples were collected in clean amber glass bottles to minimize contamination and photo degradation of PAHs. Samples were preserved and transported to the laboratory in an ice chest and analyzed within the recommended holding period.

The GC-MS instrument was calibrated using certified PAH standard solutions at five concentration levels (25, 50, 75, 100, and 125 µg/mL). The calibration curves showed good linearity with coefficients of determination (R^2) ranging from approximately 0.95 to 0.9999, indicating excellent correlation between analyte concentration and detector response, shown in table 3.6 and Figure 3.4. The calibration data demonstrated acceptable analytical performance for quantitative determination of PAHs.

Fluorene showed the highest linearity (R^2 = 0.9999), while Benzo[b]fluoranthene exhibited the lowest value (R^2 = 0.9801). Nevertheless, all calibration curves met the generally accepted criterion of $R^2 \geq 0.98$ for quantitative environmental analysis. These results demonstrate that the analytical method was suitable for the determination of PAHs in water samples.

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The high linearity obtained confirms the reliability of the concentration data reported for the analyzed PAHs and indicates minimal instrumental drift during analysis. Consequently, the generated data are considered accurate and suitable for environmental assessment of PAH contamination in Romi River receiving refinery effluent.

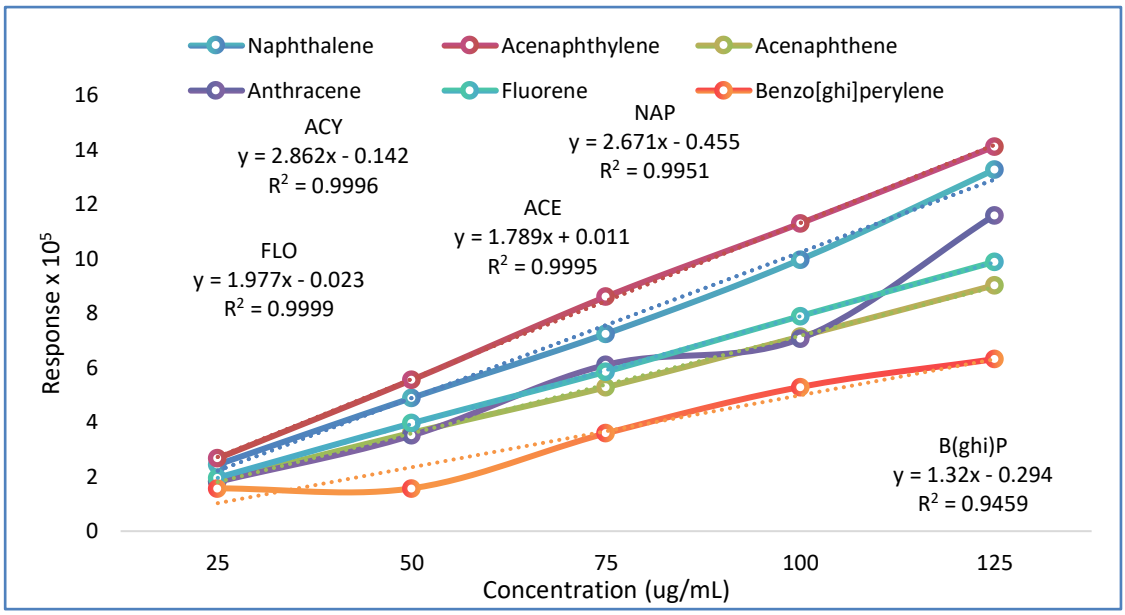


Figure 3.4: Typical Five-point Calibration Curves for the standard PAHs.

Similar Studies

Generally, the physicochemical analysis for the four sampling points from Romi River receiving Kaduna Refinery effluent, are varied and were comparable with the results obtained from similar studies across Nigeria. Liman and Rabi'u, (2018) conducted research on physicochemical analysis of water from Romi River, revealed similar results but with highest conductivity, BOD and COD when compared to this study which in turn recorded highest TDS and Turbidity. dynamics. Similar results for physicochemical and PAHs parameters in Ijegun-Egba River, Lagos State, Nigeria were reported by Oluwakemi *et al,* 2023, Conductivity levels (4,432.5 µS/cm) which was above WHO recommended limit (2,500µS/cm). pH and temperature were within the limits in all sites studied. Higher conductivity were also recorded in other studies (Adekunle et al. 2013; Owamah et al. 2013). All these changes could be due to temporal, spatial and environmental.

Several studies have reported that petroleum refinery effluents are characterized by elevated concentrations of low molecular weight PAHs, especially Naphthalene and Acenaphthylene. Yao *et al.* 2023, reported PAHs distribution of two lakes having relatively low level of high-molecular-weight (HMW; 4–6 rings) PAHs and a high level of low-molecular-weight (LMW; 2–3 rings) PAHs (> 70%). LMW PAHs frequently make up a larger portion of the 16PAHs in water due to their water solubility and higher vapor pressure (Wang *et al,* 2023). Similarly, Silva *et a*l, 2007 reported the distribution of PAHs in Brazil in which the high-molecular mass PAHs such as benzo[b] fluoranthene, benzo[k]fluoranthene, benzo[a]pyerne, indeno[1,2,3-cd] pyrene, benzo[g,h,i] perylene and dibenzo[a,h] anthracene, were not detected in the water samples, the fact attributed to their lower water solubility. The concentration of Nap in dissolved water was so high that it occupied 80.9 ± 24.1%, 52.1 ± 26.4%, and 30.8 ± 16.2% of total 16 PAHs content in South Drainage Canal, Yongding New River, and North Drainage Canal, respectively (Weixiao *et al,* 2011). This is partially due to its higher solubility in water compared with other PAHs. The ratio of Nap to total 16 PAHs in both dissolved water and sediment of South Drainage Canal were highest in the three wastewater receiving streams.

Davies *et al*, 2011, reported detection of PAHs at all sampling points in the Oburun Lake but were present at very low concentrations especially the lower molecular PAHs (LMPAHs). These were Naphthalene (0.1-0.5 µg/l), Acenaphthene (0.015-0.779 µg/l), Acenaphthylene (0.011-0.962 µg/l), Fluoranthene (0.017-1.04 µg/l), Anthracene (0.015-0.15 µg/l), Phenanthrene (0.022-2.63 µg/l), Fluorene (0.014-1.36 µg/l), Pyrene (0.015-1.67 µg/l) and Benzo(g,h,i) perylene (0.016-1.32) values were below the set standard of USEPA (2007) and WHO (2011). The lower molecular PAHs have opposite characteristics: they have acute toxicity to aquatic organisms and are usually lost due to volatilization and microbial

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degradation thus the recorded low values (below maximum contaminant levels). They could be from natural sources (petrogenic) (Yan et al., 2004, 2005; Darren et al., 2005). In the findings of Umudi *et al*, 2025, six out of the sixteen priority PAHs were identified. The PAH concentrations in water from various location ranged as follows: BaP (0.083 to 0.113), Nap (ND-0.543), Ant (ND-0.083), BbF (0.080- 0.093), BkF (0.083-0.093) & Ph (ND-0.083) mg/L.

PAHs parameters in Ijegun-Egba River, Lagos State, Nigeria were reported by Oluwakemi *et al,* 2023, reported Naphthalene (NAPH) as the most abundant PAH, 2221.58 µg/L. The highest values of individual PAH compounds detected belong to the low molecular weight (LMW) categories (naphthalene, acenaphthylene, acenaphthene, fluorene, anthracene, phenanthrene, fluoranthene, pyrene, chrysene) as in this study which are often classified to be of petrogenic sources. Table 3.5 present the comparison of results of this study with others using GC-MS.

Table 3.5: Comparison of results of this study (concentrations of PAHs) using GC-MS with similar studies.

Location	Number of PAHs	Range((µg/L)	References
Kaduna, Nigeria This Study)	6	0.043 – 360.443	
Osun Nigeria	22	6480.00	Ediagbonya *et al., 2025*
Osun Nigeria	7	BDL- 120.00	Abolanle *et al*, 2017
Lagos, Nigeria	13	100 – 73720.00	Ogunfowokan *et al* 2003
Osogbo, Nigeria	16	100.00 – 15810	Ogunfowokan *et al* 2003
Jos, Nigeria	16	006 – 9500	Uzochukwu 2017
Taiwain, China	16	70 – 140	Chih-Feng *et al,* 2019
Sergipe, Brazil	16	96.00 – 1390	Dorea *et al,* 2006

Relationship between Physicochemical Parameters and PAHs

The elevated PAH concentrations corresponded with changes observed in water quality parameters downstream of the effluent discharge point.

The increase in BOD from 3.43 mg/L to 4.37 mg/L and COD from 45.17 mg/L to 66.50 mg/L indicates increased organic pollution loading downstream. Petroleum hydrocarbons, including PAHs, contribute substantially to oxygen-demanding substances in aquatic systems. Consequently, microbial degradation of these compounds can reduce dissolved oxygen concentrations.

The slight reduction in DO from 6.70 mg/L upstream to 6.43 mg/L downstream supports this observation and suggests that refinery-derived pollutants are influencing the river's oxygen balance.

These findings indicate a direct relationship between refinery effluent discharge, elevated organic pollution levels, and the occurrence of PAHs in the receiving water body.

Environmental Implications

The occurrence of elevated PAH concentrations in Romi River presents several environmental concerns:

- Toxicity to aquatic organisms.
- Bioaccumulation in fish and other aquatic species.
- Potential transfer through the food chain.
- Long-term ecological degradation.
- Possible public health risks where river water is used for domestic activities.

Naphthalene, the dominant compound detected, is known to exert toxic effects on aquatic organisms when present at elevated concentrations. Continuous discharge of refinery effluent may therefore compromise ecosystem integrity and biodiversity within the river system.

Conclusion

The results revealed evidence of hydrocarbon contamination in Romi River receiving Kaduna Refinery effluent, despite the fact that the refinery was ideal for almost a decade. The significant increase in COD and BOD downstream indicates the introduction of organic pollutants from industrial activities. Although dissolved oxygen remained above the minimum requirement for aquatic life, its reduction downstream suggests increasing pollution stress.

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The PAH profile was dominated by Naphthalene, which accounted for approximately 97.7% of the total detected PAHs. The concentration of Naphthalene exceeded the WHO drinking-water guideline value, indicating substantial contamination. Similar observations have been reported in rivers impacted by petroleum refinery discharges, where low molecular weight PAHs frequently dominate due to their abundance in crude oil and refined petroleum products.

The predominance of low molecular weight PAHs and the scarcity of high molecular weight compounds indicate a petrogenic source of contamination. This finding supports the hypothesis that refinery wastewater is a major contributor to the hydrocarbon burden observed in Romi River.

The combined physicochemical and PAH data demonstrate that refinery effluent discharge has adversely affected the water quality of the river, emphasizing the need for improved wastewater treatment and regular environmental monitoring.

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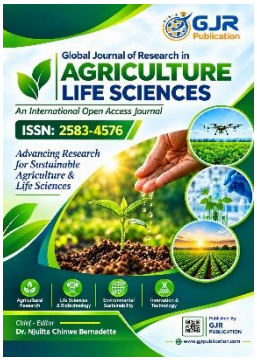
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