

OCCURRENCE AND DISTRIBUTION OF DICLOFENAC IN SURFACE WATER AND SEDIMENT OF TERENGGANU RIVER

ADRINA SAIDIN¹, TUAN FAUZAN TUAN OMAR^{1,2*}, AHMAD FAWWAZ AHMAD TARMIZI¹ AND AZRILAWANI AHMAD^{1,2}

¹Faculty of Science and Marine Environment, Universiti Malaysia Terengganu, 21030 Kuala Nerus, Terengganu, Malaysia.

²Ocean Pollution and Ecotoxicology Research Group, Faculty of Science and Marine Environment, Universiti Malaysia Terengganu, 21030 Kuala Nerus, Terengganu, Malaysia.

*Corresponding author: tuan.fauzan@umt.edu.my

<http://doi.org/10.46754/jssm.2025.10.004>

Submitted: 27 August 2024

Revised: 16 March 2025

Accepted: 24 March 2025

Published: 15 October 2025

Abstract: This study was carried out to determine the occurrence and distribution of Diclofenac (DCF) in surface water and sediment of the Terengganu River and to assess the relationship between physicochemical parameters and concentration of diclofenac. An analytical method employing a Solid-Phase Extraction (SPE) with the combination of HPLC-DAD was optimised for the analysis of diclofenac in the surface water and sediment, which showed extraction recovery at 70% and 98%, respectively. The calibration curve was constructed for HPLC optimisation at seven concentrations for diclofenac, demonstrating excellent linearity with an R^2 value of 0.9996. The result shows the concentration of diclofenac in surface water ranged between $< 0.02 \mu\text{g/L}$ and $6.49 \mu\text{g/L}$. There was no significant correlation between the level of diclofenac and physicochemical parameters. Meanwhile, diclofenac was found in sediment ranging from $< 3.50 \text{ ng/g}$ to 60.96 ng/g . Nevertheless, this preliminary finding represents a significant contribution to pollution research in the tropical coastal ecosystems of Malaysia, particularly in the east coast region and provides a valuable baseline for future studies.

Keywords: NSAIDs, diclofenac, surface water, sediment, Terengganu River.

Introduction

The Terengganu River, originating from Lake Kenyir, flows through Kuala Terengganu and the South China Sea. The city of Kuala Terengganu and the town of Kuala Berang are major urban areas, causing environmental pressures from agricultural and industrial practices (Boelee *et al.*, 2019).

Diclofenac (DCF) is a non-steroidal anti-inflammatory drug (NSAID) that is usually used to relieve pain such as swelling that relates to inflammation and also joint stiffness caused by arthritis (WebMD, 2023). Diclofenac, a pain reliever in pharmaceuticals is sourced from human urine, faeces, vomit, and sewage waste, which undergoes wastewater treatment to remove impurities (Nathanson & Ambulkar, 2024). Diclofenac can be found in various aqueous environmental compartments such as drinking water, sewage, and groundwater. Non-steroidal anti-inflammatory drugs (NSAIDs),

released from residences and establishments through discharge from Sewage Treatment Plants (STP) in Malaysia have reportedly been found in the water stream with the range of 1.40×10^{-4} to $2.76 \times 10^{-2} \text{ mg/L}$ (Hanafiah *et al.*, 2022).

Diclofenac can interact with various compounds, including metals, organic contaminants, and diclofenac metabolites, forming new possible emerging contaminants (Lonappan *et al.*, 2016). Several papers confirm the toxicity of diclofenac in algae and bacteria, mainly on acute toxicity (Von Der Ohe *et al.*, 2011). A research study on zebrafish by Bio and Nunes (2020) stated that zebrafish or *Danio rerio* embryos and larvae were exposed to diclofenac concentrations, suggesting potential oxidative stress in their natural habitats, based on environmental observations. Next, a study by Świacka *et al.* (2022) showed the effects of 4-OH DCF on *Mytilidae* mussels, revealing

toxicity through gill deformations, digestive gland necrosis, gonadal atresia, and atrophy.

Additionally, both DCF and 4-OH DCF significantly reduced gill protein content (Świacka *et al.*, 2022). Since sediments provide food for a variety of organisms in the river, they are a crucial source of storage for diclofenac. Many aquatic organisms such as fish and bivalves prefer to live on the riverbed. The places where EDC remains and builds up as a result of being carried by water currents or the decomposition of dead organisms are called sediments. To monitor diclofenac’s harmful impacts on the environment and organisms, research on its presence in the environment is necessary. Therefore, a study was done to determine the occurrence and distribution of diclofenac in surface water and sediment of the Terengganu River and to assess the correlation between physicochemical parameters and concentration.

Materials and Methods

Sampling Site

Sampling was conducted at the Terengganu River with 15 sampling stations denoted by S1 to S15 as shown in Figure 1. The coordinates and description of the sampling stations are shown in Table 1. The Terengganu River basin

is situated on Peninsular Malaysia’s east coast at 5.34°N and 103.15°E (Lee *et al.*, 2016). These rivers flow through a variety of socioeconomic activity zones, including farms, aquaculture, commercial industries, urban areas, rural areas, reserves, and forested areas (Sabri *et al.*, 2022).

Chemicals, Standard, Samples Collection, and Pretreatment

The acetonitrile (ACN) and methanol (MeOH) were obtained from Fisher Scientific (Loughborough, UK). The ultrapure water was generated from the Milli-Q water purification system (Millipore, USA). A high-purity individual standard (99%) for the targeted compounds (diclofenac) was obtained from Dr. Ehrenstorfer GmbH (Augsburg, Germany). A stock solution was precisely diluted to create standard solutions in methanol. All standard solutions were freshly prepared and kept at 4°C in glass-stoppered volumetric flasks.

Surface water samples of 1 m depth were taken using the Niskin water sampler. Samples were then put into 500 mL amber glass bottles, which were pre-cleaned with acetone and kept in a cool box at a temperature of 6°C. YSI multiparameter was used to measure the physicochemical characteristics of water samples, including salinity, dissolved oxygen

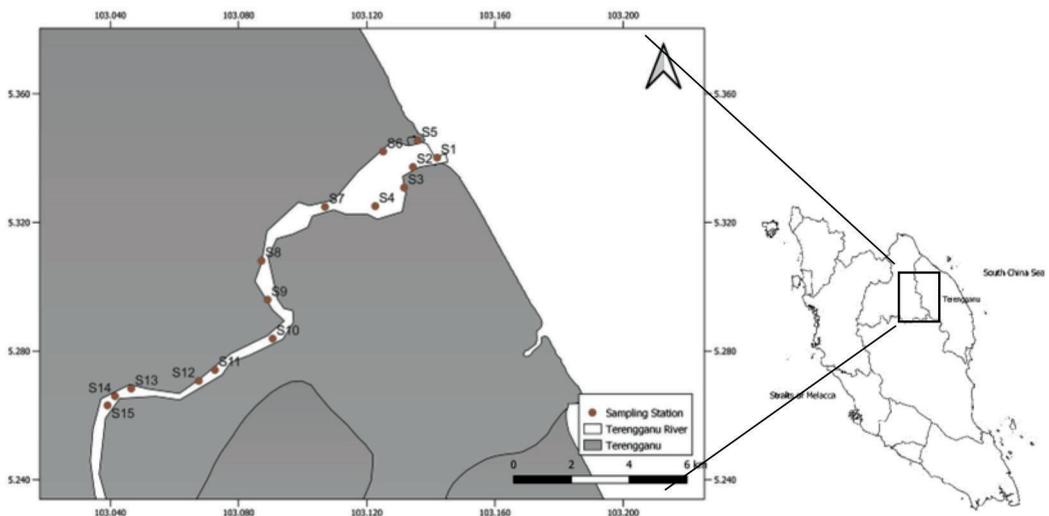


Figure 1: Sampling stations of the Terengganu River

Table 1: Coordinates and description of sampling locations for surface water samples and sediment samples at the Terengganu River

	Abbreviation	Coordinate	Description
S1		5°20'24.51"N 103° 8'31.04"E	Before Drawbridge
S2		5°20'14.04"N 103° 8'03.90"E	Near Pasar Payang
S3		5°19'50.97"N 103° 7'53.96"E	Near Hotel Seri Malaysia Kuala Terengganu
S4		5°19'30.28"N 103° 7'21.55"E	Near Jambatan Pulau Duyung, Masjid Crystal, and Taman Tamadun Islam
S5		5°20'43.93"N 103° 8'09.34"E	Near Masjid Seberang Takir and Jeti Seberang Takir
S6		5°20'31.43"N 103° 7'30.47"E	Between Pulau Che Long and Che Hassan
S7		5°19'29.40"N 103° 6'25.14"E	Near Pulau Pak Mat
S8		5°18'29.12"N 103° 5'13.77"E	Fisheries area
S9		5°17'45.48"N 103° 5'20.70"E	Fisheries area
S10		5°17'01.95"N 103° 5'26.33"E	Fisheries area
S11		5°16'26.93"N 103° 4'21.49"E	Fisheries area
S12		5°16'14.68"N 103° 4'03.38"E	Fisheries area
S13		5°16'06.05"N 103° 2'47.79"E	Near the jetty
S14		5°15'57.56"N 103° 2'29.30"E	Near the newly built bridge
S15		5°15'47.25"N 103° 2'20.89"E	Close to the newly built bridge and near Kalang Warisan

(DO), temperature, and pH. After sampling, the samples were immediately filtered through a 47 mm (1.7 µm pore size) glass microfibre filter. By adjusting the filtrates to pH 2 with 1M hydrochloric acid and storing them in a freezer before extraction and clean-up, samples were then preserved.

Sediment samples were collected using a Ponar grab at 15 different stations selected along the Terengganu River. Each sediment was wrapped in aluminium foil, kept in a plastic bag, and labelled with S1 to S15. The sediment was then stored in an ice box. After arriving at the lab, the sample is then transferred to the freezer. Sediment was spread evenly in an aluminium container and was air-dried at room temperature for a month. During the drying process, the sediment was stirred every two days to avoid hard drying.

To determine the efficiency of extraction and clean-up, the percentage of diclofenac extraction recoveries was examined. The high-performance liquid chromatography (HPLC)

by Shimadzu was used to determine the peak of diclofenac. The calibration curve was constructed for HPLC optimisation at seven concentrations, which were 0.05 mg/L, 1.0 mg/L, 2.0 mg/L, 4.0 mg/L, 6.0 mg/L, 8.0 mg/L, and 10.0 mg/L. In this study, tap water was spiked with 400 µL of 10 µg/mL targeted compound to assess the extraction recovery of the analytical procedures. For sediment samples, the spike extraction procedures were performed to identify the significant peak corresponding to optimal recovery. A 10 µg/mL standard solution, with a volume of 400 µL was used as a spiking level for the sediment sample. The recovery assessment for both water and sediment was carried out in three replicates to obtain statistically significant values. Figure 2 shows the chromatographic separation for diclofenac.

Sediment Analysis

Grain size analysis was done using a sieving method to determine the type of sediment in each station. Several sieves, which were 4 mm,

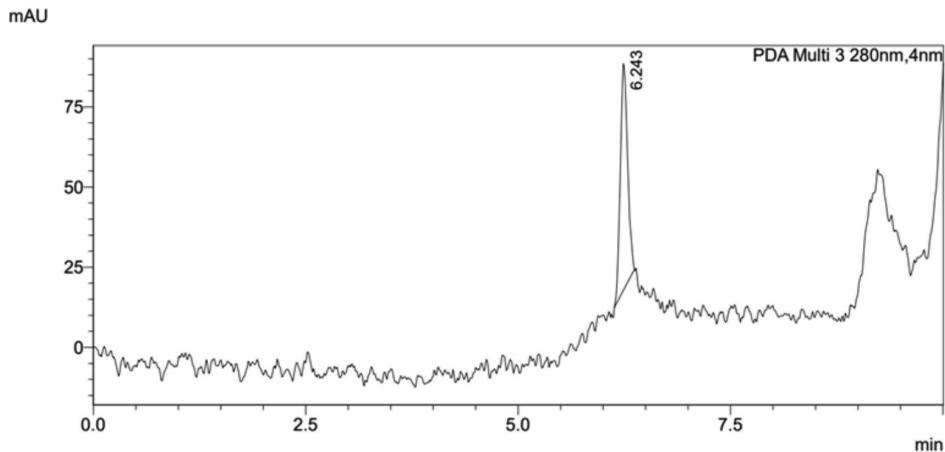


Figure 2: Chromatographic separation for diclofenac

2 mm, 1 mm, 0.5 mm, 0.25 mm, 0.125 mm, 0.09 mm, and 0.063 mm were used in the Grain Size Analysis test and the sediment classification based on particle size range in Unified Soil Classification System (USCS) (Daryati *et al.*, 2019). The S1 dry sediment was weighed, the sieves and pan used for analysis were recorded. Samples were placed in a top sieve, shaken for 10 minutes, and weighed again before data calculation. The processes were repeated for the other samples.

Sediment pH was measured by adding 10 g of air-dried sediment to 20 mL of distilled water, shaking and stirring, and allowing it to sit overnight before reading. The pH meter was regularly calibrated and the electrode was cleansed with distilled water between samples to ensure accurate readings.

The total organic carbon was analysed using SSM-5000A for solid and TOC-L (CPH) for liquid from Shimadzu at every station. The sample boats were weighed with 0.1 mg of sediment. A boat was placed inside a TOC analyser total carbon chamber. The sediment was then run through carbon analysis. Subsequently, a second one was placed inside an inorganic carbon chamber and hydrochloric acid (HCl) was added for inorganic carbon analysis. The procedure was repeated for each of the 15 stations.

Sediment Clean-up

The sediments collected from grain size analysis were sieved and ground into finely divided particles, with each particle weighing approximately $0.500 \text{ g} \pm 0.001 \text{ g}$ (Omar *et al.*, 2021). The materials were ground and mixed with $1.00 \text{ g} \pm 0.001 \text{ g}$ of Aluminium Oxide (Al_2O_3) using a mortar and pestle until the mixture was evenly distributed. The samples were transferred into a polypropylene tube. The tube was filled with 10 mL of ultrapure water (UPW) and 10 mL of acetonitrile. After the tube was sealed with a cap, it was vortexed for 60 seconds. At a temperature of 30°C , the tube was subjected to sonication for 10 minutes. The sample was then centrifuged for 60 minutes at 1,000 rpm. After centrifugation, the extracted material was carefully transferred into a 60 mL conical flask. To acquire 60 mL of extracts from the identical tube, the experiment was replicated twice and subsequently combined in a single conical flask and covered with aluminium foil.

Solid Phase Extraction

The C18 cartridges (SEClute HLB 10 mg/1 mL, 50 pk) for water samples were conditioned with 5 mL of methanol (MeOH), 5 mL of Milli-Q water, and Milli-Q water at pH 2. Water samples (200 mL) were filtered with a 47 mm ($1.7 \mu\text{m}$

particle size) glass microfibre filter and placed into the cartridges. Then, the extraction was accomplished using the suction pump in a series of vacuum manifolds. The process proceeded with washing using 15 mL of Milli-Q water. The sequential elution was accomplished with 10 mL of MeOH and 5 mL of MeOH: ACN (50:50), as the target analytes were all eluted during the elution procedure. The extract then proceeded into the rotary evaporator and was further concentrated with a gentle stream of nitrogen (N₂) blow until almost dry. Before HPLC analysis, the extracts were reconstituted with ultrapure water/ACN (70:30, v/v) and filtered through a 0.2 µm nylon membrane filter. The procedure was replicated three times for the water sample.

The sediment extraction was based on Omar *et al.* (2021), where the extraction begins with conditioning the Strata-X polymeric reversed-phase C18 cartridges with 5 mL of acetonitrile (ACN). This was followed by 5 mL of UPW:ACN (90:10) and 5 mL of acetonitrile: Methanol ACN:MeOH (70:30), respectively. Then, the extracted samples were loaded into the cartridge and 10 mL of UPW:ACN (90:10) was used to clean up the matrix interferences in the space and wall of the cartridge until the samples were completely drawn through the cartridge. The analytes were then eluted with 15 mL of ACN:MeOH (70:30). The extract was then concentrated to approximately 1-2 mL volume by a rotary evaporator and further concentrated until nearly dry under a gentle stream of nitrogen.

The reconstitution of the extract was done with UPW:MeOH (70:30) to 1 mL. Finally, the extract was filtered with a 0.2 µm nylon membrane filter before being analysed by HPLC-DAD. The procedure was replicated three times for the sediment sample. Different physical states, compounds, and matrices in sediment and water samples influence SPE methods, requiring variations in sample preparation, extraction solvents, and cartridge conditioning steps for optimal recovery (Backe & Field, 2012).

Instrumental Analysis

The diclofenac was identified by peak area and respective retention time at different concentrations. Using HPLC (Agilent Series 1200) equipped with a diode array detector (DAD), the compound was quantified and detected. The reversed-phase C18 analytical column (Supelco C18, 15 cm × 4.6 mm, 5 µm) was chosen with the chromatographic conditions developed and optimised. With a 20 µL injection volume and 10-minute runtime, the mobile phase flow rate was set at 0.8 mL/min and a wavelength of 280 nm. The specific retention period of each standard was used to establish the presence of diclofenac and to quantify it using external standard calibration.

Quality Assurance and Quality Control

The experimental procedures were conducted under stringent quality assurance and Quality Control (QC) protocols to ensure the reliability and accuracy of the results. Laboratory apparatus and glassware were pre-treated to minimise potential cross-contamination by soaking and washing them with Decon 90, a specialised laboratory detergent, followed by rinsing with deionised water and an organic solvent. The materials were subsequently dried in an oven at 90°C to eliminate residual water and solvents. High-performance liquid chromatography (HPLC) grade chemicals and solvents were used for sample preparation while high-purity native and labelled standards (> 95% purity) were used for standard preparation.

Each batch of samples included the analysis of procedural blanks and QC spikes to detect potential cross-contamination and evaluate the efficiency of sample extraction and cleanup processes. The extraction efficiency for the targeted compound yielded satisfactory recovery rates, ranging from 70% for surface water samples to 98% for sediment samples. HPLC optimisation was achieved using a calibration curve constructed with seven concentrations of diclofenac, which exhibited excellent linearity ($R^2 = 0.9996$). The method detection limit,

determined based on a 3:1 signal-to-noise ratio was 0.02 µg/L and 3.50 ng/g for surface water and sediment, respectively.

Statistical Analysis

The statistical analysis was done using IBM SPSS, which uses Pearson correlation. The Pearson correlation measures the strength of the linear relationship between two variables. The two-tailed Pearson correlation was used in IBM SPSS Statistics to find the correlation. At α less than 0.05, the differences between groups were statistically significant.

Results and Discussion

Concentration of Diclofenac in Surface Water Samples and Sediment Samples

The concentration of diclofenac in surface water samples and sediment samples is shown in Table 2. For surface water samples, the compound was detected along the surface water of the Terengganu River with the concentration ranging from < 0.02 µg/L to 6.49 µg/L, as shown in Table 2. It was observed that diclofenac was

found in most sampling stations and was present at higher levels, with the highest level being 6.49 µg/L in the lower reaches of the river. However, this concentration is higher than a study by Praveena *et al.* (2018), which found that the concentrations of diclofenac along the Lui River, Gombak River, and Selangor River were 4.29 ng/L, 15.07 ng/L, and 15.49 ng/L, respectively.

The highest concentration of diclofenac in sediment was in Station 10 at 60.96 ng/g and the lowest in Station 15 at < 3.50 ng/g, which is higher than the study by Omar *et al.* (2018) who stated that diclofenac (13.88 ng/g dry weight) was found in tropical coastal sediment of anthropogenically impacted Klang River estuary, Malaysia. The mean concentration of diclofenac in Terengganu River sediment was 35.54 ng/g. The values of diclofenac concentration range from < 3.50 ng/g to 60.96 ng/g. The highest concentration of diclofenac was detected in Station 10 at the fisheries area and the lowest concentration was in Station 15 near Kalang Warisan. Amran *et al.* (2018) indicated that the water quality in the

Table 2: Concentration of diclofenac in surface water of Terengganu River

Sampling Point	Surface Water (µg/L ± %RSD)		Sediment (ng/g ± %RSD)		
	Mean	±	Mean	±	Mean
S1	< 0.02		6.05	±	1.16
S2	1.64	±	5.67	±	19.54
S3	1.99	±	2.57	±	2.28
S4	6.49	±	14.45	±	18.17
S5	3.22	±	1.37	±	14.16
S6	0.58	±	2.49	±	9.77
S7	6.33	±	12.19	±	5.62
S8	< 0.02		51.91	±	12.39
S9	< 0.02		58.68	±	16.05
S10	1.62	±	0.72	±	5.24
S11	1.58	±	12.95	±	3.22
S12	1.60	±	5.65	±	1.16
S13	1.57	±	7.60	±	19.54
S14	1.19	±	13.71	±	2.28
S15	1.45	±	7.48	±	18.17

Terengganu River Basin has been negatively impacted, with the most significant degradation occurring in the downstream and central areas. This decline is primarily attributed to various anthropogenic activities, including waste disposal, municipal and industrial effluents, and untreated agricultural runoff entering the river system (Amran *et al.*, 2018).

Physicochemical Parameters in Surface Water and Sediment of Terengganu River

The physicochemical parameters recorded for water samples were temperature, pH, salinity, Dissolved Oxygen (DO), and Total Organic Carbon (TOC). These parameters were collected using the YSI parameter with an average of three replicates. The TOC for the water sample was analysed using a total organic carbon analyser. The averages of temperature, pH, and salinity at the Terengganu River were 28.80°C, 6.03°C, and 0.29°C, respectively.

The pH value of sediment collected from the riverbed of the Terengganu River ranged between 5.76 and 7.65, with a mean of 6.79 ± 0.52. The highest and the lowest pH were at Stations 11 and 12 around the fisheries area, with the values of 7.65 and 5.76, respectively. Station 6, which was located between Pulau Che Long and Pulau Che Hassan had a neutral value of 7.00. Stations 2, 3, 5, 7, 10, 12, 13, 14, and 15 had pH values below 7.00, which were 6.49, 5.91, 6.46, 6.95, 6.77, 5.76, 6.80, and 6.68, respectively. Stations 1, 4, 8, 9, and 11 had pH values higher than 7, which were 7.48, 7.20, 7.20, 7.03, and 7.65, respectively. The highest TOC percentage was observed at Station 3, near Hotel Seri Malaysia Kuala Terengganu, at 1.81%, while the lowest was at Stations 12 and 13 at 0%. The physicochemical parameters for surface water and sediment of the Terengganu River are shown in Table 3.

Particle Size Analysis (PSA) for river sediments of the Terengganu River showed

Table 3: Physicochemical parameters for surface water and sediment of Terengganu River

Stations	Water Sample					Sediment Sample	
	Temperature (°C)	pH	Salinity (ppt)	Dissolved Oxygen (mg/L)	Total Organic Carbon (ppm)	pH	Total Organic Carbon (%)
S1	29.00	6.13	1.10	4.60	2.29	7.48	1.47
S2	29.10	5.96	1.27	4.40	5.56	6.49	1.52
S3	29.10	5.88	1.34	4.30	2.39	5.91	1.81
S4	28.90	5.89	0.16	4.33	2.85	7.20	0.22
S5	28.90	6.12	0.35	4.67	1.91	6.46	1.18
S6	29.20	6.31	0.02	4.40	6.15	7.00	0.21
S7	29.10	6.00	0.01	4.50	4.81	6.95	1.26
S8	29.00	6.04	0.01	4.60	3.32	7.20	0.11
S9	28.90	6.03	0.01	4.60	2.93	7.03	0.21
S10	28.80	6.34	0.01	4.67	6.46	6.77	0.41
S11	28.50	5.94	0.01	4.70	2.25	7.65	0.09
S12	28.70	6.05	0.01	4.40	12.77	5.76	0.00
S13	28.30	5.94	0.01	4.63	5.66	6.80	0.00
S14	28.30	5.94	0.01	4.70	3.53	6.68	1.43
S15	28.20	5.92	0.01	4.63	1.50	6.44	0.78

that the sediments were dominated by medium sand with a percentage mean value of 36%. The percentage mean values of fine sand, medium sand, coarse sand, and gravel were 1.45%, 31.79%, 31.94%, and 0%, respectively. Table 4 shows the result of the particle size analysis of sediment in the Terengganu River. PPCPs like sulfamethoxazole, carbamazepine, triclosan, and ciprofloxacin remain more persistent in sediment than in water (Conkle *et al.*, 2012). The ongoing release of these organic chemicals from sediments into the overlying water is conceivable, potentially leading to adverse effects on benthic organisms that are persistently exposed to PPCPs present in the sediments, interstitial water, and overlying water (Gilroy *et al.*, 2011).

Correlation Analysis of Parameters Analysed in Surface Water and Sediment

Table 5 shows the correlation analysis of the parameters analysed in surface water for diclofenac. The correlation shows only one significant correlation, which was between dissolved oxygen and temperature. The data showed a moderate correlation between the two

parameters ($r = -0.582, p < 0.05$). However, based on this correlation analysis, diclofenac does not have any relationship with other physicochemical parameters. A similar pattern was also observed for sediment samples. Therefore, the physicochemical parameter does not affect diclofenac in surface water and sediment.

Malaysia National Water Quality Standards (NWQS) were used in this study to determine the physicochemical parameters of water. Based on the in-situ determination (Table 3), the pH value (5.88-6.34) was still in the range set by NWQS (pH range = 5-9). Dissolved oxygen (4.30 mg/L – 4.70 mg/L) of the surface water of the Terengganu River was found within the range of the NWQS (3-5 mg/L). Physicochemical parameters are good indicators of water quality and excellent attributes of human impact on the environment.

Distribution of Diclofenac in Surface Water and Sediment

Figure 3 and Figure 4 show the distribution of diclofenac in surface water samples and sediment samples, respectively. It was observed

Table 4: Particle Size Analysis (PSA)

Stations	Particle Size Analysis (%)	Sediment Classification
S1	49	Fine sand
S2	45	Fine sand
S3	67	Medium sand
S4	65	Fine sand
S5	61	Medium sand
S6	70	Medium sand
S7	63	Coarse sand
S8	55	Medium sand
S9	54	Medium sand
S10	70	Coarse sand
S11	52	Fine sand
S12	56	Coarse sand
S13	57	Medium sand
S14	49	Medium sand
S15	60	Medium sand

Table 5: Correlation analysis of parameters in surface water and sediment of Terengganu River

	Temperature	pH	Salinity	DO	TOC	DCF
Water						
Temperature	1					
pH	0.364	1				
Salinity	0.456	-0.180	1			
DO	-0.582*	0.167	-0.435	1		
TOC	0.056	0.312	-0.205	-0.312	1	
DCF	-0.025	-0.365	-0.002	-0.340	-0.101	1
*Correlation is significant at the 0.05 level (2-tailed)						
Sediment						
pH	n.a	1	n.a			
TOC	n.a	0.28	n.a	n.a	1	
DCF	n.a	0.17	n.a	n.a	0.33	1

**Correlation is significant at the 0.01 level (2-tailed)
 n.a = not analysed

that diclofenac was found in most sampling stations and was present at higher levels, with a concentration of 6.49 µg/L in the lower reaches of the river. It showed that the concentration was detected highest at S4 for the water sample as shown in Figure 3 and lower, which was < 0.02 at S1, S8, and S9. Based on the site description, the presence of diclofenac in this area is due to the urban area near the river. Generally, the concentration in Figure 4 remained constant between the stations. The compound presence in the water body might be due to the Wastewater

Treatment Plants (WWTPs), facilities, and runoff. A study by Omar *et al.* (2019) found that the concentration of diclofenac at the Klang River estuary is 10.80 µg/L, which is higher than that at the Terengganu River, which is 6.49 µg/L.

Meanwhile, the concentration of diclofenac at Langat River, which is 0.113 µg/L, as stated by Al-Odaini *et al.* (2011) is lower than that of Terengganu River. Pharmaceuticals' environmental fate is determined by partitioning,

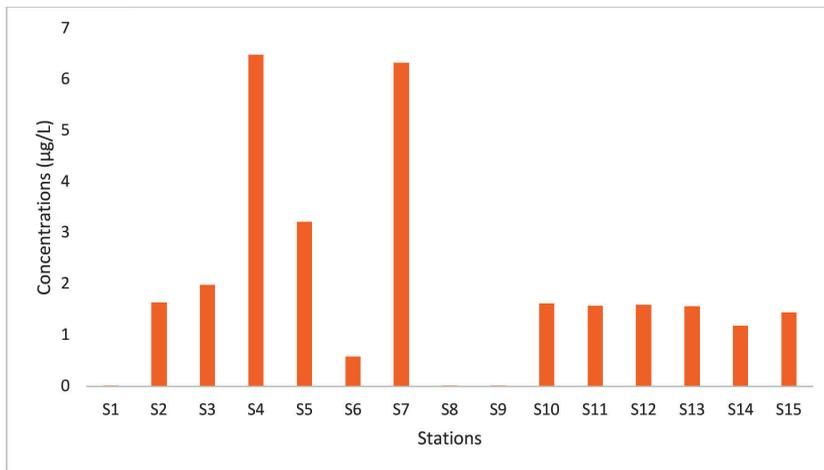


Figure 3: Distribution of diclofenac in surface water samples

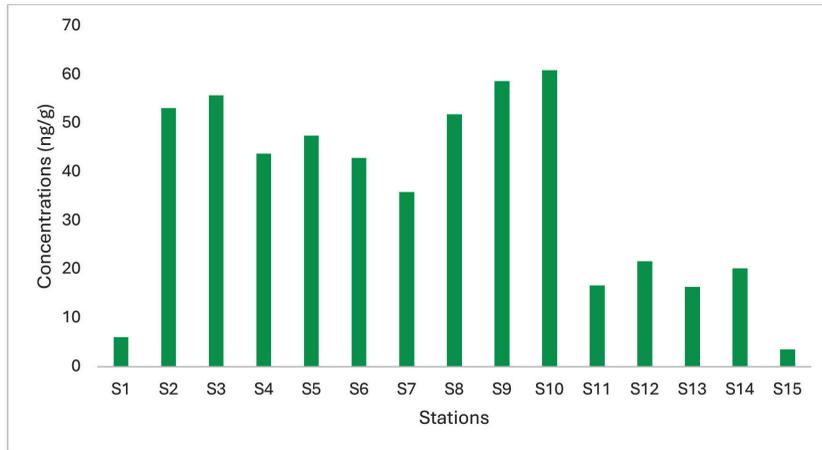


Figure 4: Distribution of diclofenac in sediment samples

photolysis, hydrolysis, and biodegradation, influenced by compound properties and environmental factors like pH and organic matter (Ying *et al.*, 2013). Diclofenac was identified in sediment, likely as a result of its mobility from Wastewater Treatment Plants (WWTPs) and subsequent accumulation within the sediment. Moreover, the pronounced mobility of diclofenac in sediment is attributed to its interactions between the solid and dissolved phases, as indicated by its octanol-water distribution coefficient ($\log K_{ow}$) (Sathishkumar *et al.*, 2019).

Conclusions

This study examined the concentration of diclofenac in the surface water and sediment of the Terengganu River. Diclofenac was detected in 12 out of 15 water sampling stations, with concentrations remaining relatively low. The highest concentrations were found near urban and industrial areas, especially at station S4 downstream. There was no significant correlation between diclofenac levels and physicochemical parameters. Diclofenac was also present in sediment samples in 14 out of 15 stations, with higher concentrations near the river mouth and in fishing and settlement areas.

The concentration of diclofenac increased as it moved downstream, likely due to sedimentation. The usage of anti-inflammatory drugs containing diclofenac contributed to the rise of chemicals in the river. Therefore, an extensive study should be carried out to further evaluate the potential source of diclofenac in this river.

Acknowledgments

This research was carried out using a grant provided by the Ministry of Higher Education (MOHE) Malaysia through the Fundamental Research Grant Scheme (FRGS/1/2024/WAS02/UMT/02/8). The authors would like to express gratitude to the Universiti Malaysia Terengganu (UMT), specifically the Faculty of Science and Marine Environment and the Centre of Research and Field Service, for granting permission to use their facilities in completing this study. The first author also wishes to thank the Ministry of Higher Education (MOHE) Malaysia for scholarship under MyBrain 2.0 Program.

Conflict of Interest Statement

The authors declare that they have no conflict of interest.

References

- Al-Odaini, N., Zakaria, M. P., Yaziz, M. I., & Surif, S. (2011). Detecting human pharmaceutical pollutants in Malaysian aquatic environment: A new challenge for water. *ResearchGate*. <https://www.researchgate.net/publication/279913351>
- Amran, N. H. M., Kamarudin, M. K. A., Wahab, N. A., Saad, M. H. M., Rosli, M. H., Samah, M. a. A., Baharim, N. B., Razak, N. a. E. M., & Jusoh, S. (2018). Environmental management on natural river based on water quality deterioration in Terengganu River Basin, Terengganu, Malaysia. *International Journal of Engineering & Technology*, 7(4.34), 132-136. <https://doi.org/10.14419/ijet.v7i4.34.23844>
- Backe, W. J., & Field, J. A. (2012). Is SPE necessary for environmental analysis? A quantitative comparison of matrix effects from Large-Volume Injection and Solid-Phase Extraction Based Methods. *Environmental Science & Technology*, 46(12), 6750-6758. <https://doi.org/10.1021/es300235z>
- Bio, S., & Nunes, B. (2020). Acute effects of diclofenac on zebrafish: Indications of oxidative effects and damages at environmentally realistic levels of exposure. *Environmental Toxicology and Pharmacology*, 78, 103394. <https://doi.org/10.1016/j.etap.2020.103394>
- Boelee, E., Geerling, G., Van Der Zaan, B., Blauw, A., & Vethaak, A. D. (2019). Water and health: From environmental pressures to integrated responses. *Acta Tropica*, 193, 217-226. <https://doi.org/10.1016/j.actatropica.2019.03.011>
- Conkle, J. L., Gan, J., & Anderson, M. A. (2012). Degradation and sorption of commonly detected PPCPs in wetland sediments under aerobic and anaerobic conditions. *Journal of Soils and Sediments*, 12(7), 1164-1173. <https://doi.org/10.1007/s11368-012-0535-8>
- Dame, R., & Libes, S. (1993). Oyster reefs and nutrient retention tidal creeks. *Journal of Experimental Marine Biology and Ecology*, 171, 251-258.
- Daryati, D., Wideasanti, I., Septiandini, E., Ramadhan, M. A., Sambowo, K. A., & Purnomo, A. (2019). Soil characteristics analysis based on the unified soil classification system. *Journal of Physics Conference Series*, 1402(2), 022028. <https://doi.org/10.1088/1742-6596/1402/2/022028>
- Gilroy, È. A., Balakrishnan, V. K., Solomon, K. R., Sverko, E., & Sibley, P. K. (2011). Behaviour of pharmaceuticals in spiked lake sediments – Effects and interactions with benthic invertebrates. *Chemosphere*, 86(6), 578-584. <https://doi.org/10.1016/j.chemosphere.2011.10.022>
- Hanafiah, Z., Wan Mohtar, W. H. M., Abd Manan, T. S. B., Bachi', N. A., Abdullah, N. A., Abd Hamid, H. H., Beddu, S., Mohd Kamal, N. L., Ahmad, A., & Wan Rasdi, N. (2022). The occurrence of non-steroidal anti-inflammatory drugs (NSAIDs) in Malaysian urban domestic wastewater. *Chemosphere*, 287.
- Lee, H. L., Tangang, F., Wahap, M. H., & Yang, S. (2016). Seasonal Hypoxia occurrence at Terengganu estuary, Malaysia and its potential formation mechanisms. *IOP Conference Series Materials Science and Engineering*, 136, 012068. <https://doi.org/10.1088/1757-899x/136/1/012068>
- Lonappan, L., Brar, S. K., Das, R. K., Verma, M., & Surampalli, R. Y. (2016). Diclofenac and its transformation products: Environmental occurrence and toxicity - A review. *Environment International*, 96, 127-138. <https://doi.org/10.1016/j.envint.2016.09.014>
- Nathanson, J. A., & Ambulkar, A. (2024, July 23). *Wastewater Treatment Process, History, Importance, Systems, & Technologies*.

- Encyclopedia Britannica. <https://www.britannica.com/technology/wastewater-treatment>
- Omar, T., Aris, A. Z., Yusoff, F. M., & Mustafa, S. (2019). Occurrence and level of emerging organic contaminant in fish and mollusk from Klang River estuary, Malaysia and assessment on human health risk. *Environmental Pollution*, 248, 763-773. <https://doi.org/10.1016/j.envpol.2019.02.060>
- Omar, T. F. T., Aris, A. Z., & Yusoff, F. M. (2021). Multiclass analysis of emerging organic contaminants in tropical marine biota using improved QuEChERS extraction followed by LC-MS/MS. *Microchemical Journal*, 164, 106063. <https://doi.org/10.1016/j.microc.2021.106063>
- Omar, T. F. T., Aris, A. Z., Yusoff, F. M., & Mustafa, S. (2018). Occurrence, distribution, and sources of emerging organic contaminants in tropical coastal sediments of anthropogenically impacted Klang River estuary, Malaysia. *Marine Pollution Bulletin*, 131, 284-293. <https://doi.org/10.1016/j.marpolbul.2018.04.019>
- Praveena, S. M., Shaifuddin, S. N. M., Sukiman, S., Nasir, F. a. M., Hanafi, Z., Kamarudin, N., Ismail, T. H. T., & Aris, A. Z. (2018). Pharmaceuticals residues in selected tropical surface water bodies from Selangor (Malaysia): Occurrence and potential risk assessments. *The Science of the Total Environment*, 642, 230-240. <https://doi.org/10.1016/j.scitotenv.2018.06.058>
- Sabri, M. M., Omar, T. F. T., Ahmad, A., & Suratman, S. (2022). An optimised analytical method to study the occurrence and distribution of Bisphenol A (BPA) and 17 β -Estradiol (E2) in the surface water of Ibai River, Terengganu, Malaysia. *Journal of Sustainability Science and Management*, 17(7), 195-203. <https://doi.org/10.46754/jssm.2022.07.014>
- Sathishkumar, P., Meena, R. a. A., Palanisami, T., Ashokkumar, V., Palvannan, T., & Gu, F. L. (2019). Occurrence, interactive effects and ecological risk of diclofenac in environmental compartments and biota - A review. *The Science of the Total Environment*, 698, 134057. <https://doi.org/10.1016/j.scitotenv.2019.134057>
- Świacka, K., Maculewicz, J., Świeżak, J., Caban, M., & Smolarz, K. (2022). A multi-biomarker approach to assess toxicity of diclofenac and 4-OH diclofenac in *Mytilus trossulus* mussels - First evidence of diclofenac metabolite impact on molluscs. *Environmental Pollution*, 315, 120384. <https://doi.org/10.1016/j.envpol.2022.120384>
- Von Der Ohe, P. C., Schmitt-Jansen, M., Slobodnik, J., & Brack, W. (2011). Triclosan—The forgotten priority substance? *Environmental Science and Pollution Research*, 19(2), 585-591. <https://doi.org/10.1007/s11356-011-0580-7>
- WebMD. (2023). *Diclofenac Oral: Uses, Side Effects, Interactions, Pictures, Warnings & Dosing* (online). <https://www.webmd.com/drugs/2/drug-4284-4049/diclofenac-oral/diclofenac-sodium-enteric-coated-tablet-oral/details>
- Ying, G., Zhao, J., Zhou, L., & Liu, S. (2013). Fate and occurrence of pharmaceuticals in the aquatic environment (Surface water and sediment). In *Comprehensive analytical chemistry* (pp. 453-557). <https://doi.org/10.1016/b978-0-444-62657-8.00014-8>