

# Evaluation of Physicochemical Parameters and Pesticide Pollution of Surface Water in the Oued Martil Watershed, Tetouan, Morocco

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**Abstract** Monitoring surface water safety at the watershed-scale is paramount amidst multiple contamination sources. This article investigates surface water quality and assesses pesticide contamination in surface water of Oued Martil watershed, marking a seminal contribution as the inaugural assessment of its kind. It examined pesticide contamination and 6 physicochemical parameters at four locations, primarily mitigating the shortage of Moroccan research in this field within the Oued Martil watershed. The pesticide residue samples were analyzed using liquid and gas chromatography coupled with tandem mass spectrometry. Results revealed that 16 pesticides studied were below the quantification limits. The pH was generally neutral, except at station S4 in June and July where it was alkaline. Water temperature ranges from 19.7°C to 33°C, influenced by local factors. Chemical Oxygen Demand fluctuates between 11.6 mg/l and 30 mg/l. Nitrites were typically absent, except at station S1 in April with 0.19 mg/l. Orthophosphates, originating from domestic and agricultural runoff, were present at low concentrations, exceeding 0.005 mg/l in most stations. Surface water quality of sampling sites was therefore predominantly good. These findings further water quality knowledge and pesticide behavior, establishing a crucial baseline for forthcoming monitoring studies.

**Keywords** Morocco, Oued Martil Watershed, Water Quality, Surface Water Contamination, Physico-Chemical

Parameters, Pesticides, Pesticide Residue Analysis

## 1. Introduction

Water is regarded as a fundamental and paramount component of the earth and its most significant natural resource. Nevertheless, anthropic activities have harmed water quality through the dispersion of pollutants into the environment [1]. Morocco, a North African country, although it has a comparatively significant freshwater resource reserve of about  $22 \times 10^9$  m<sup>3</sup> compared to other North African countries [2], its water resources are regarded as being severely limited because of long-lasting droughts [3]. Moreover, in the setting of limited, fragile and threatened water resources in the Mediterranean region, the matter of water quality resources is also an increasingly critical topic in Morocco as worldwide [4]. Water quality assessment requires the monitoring of numerous parameters such as physicochemical parameters, toxic substances (pesticides, heavy metals) and others, in order to ensure that water quality is effectively maintained and appropriate on an ongoing basis. Surface water pollution is a major problem that is constantly addressed in the Mediterranean region and worldwide. The Mediterranean countries, of which Morocco is a part, have developed a

judicious policy to preserve water resources (surface and groundwater). They also seek to reduce the rate of pollutant load that reaches the various rivers, mainly in the Martil watershed. It is one of the Mediterranean watersheds and is considered the biggest and most important pool in northwestern Morocco [5]. Various studies carried out in Morocco have documented surface water pollution in many watersheds as a result of different human activities [6-10]. Although there is a wealth of research on physicochemical parameters, microbiological markers and metal pollution, there is a significant deficiency in the literature concerning the contamination of surface waters by pesticides that is not adequately documented at the watershed scale.

Pesticides are chemical substances employed throughout the world for various purposes. They rank among the most worrying persistent organic pollutants and among the most detrimental to water environments, owing to their high level of toxicity, lipophilicity, ability to bioaccumulate, and environmental resistance [11-14]. Pesticides are becoming increasingly detectable in water resources globally, leading to severe consequences for both water security and human health. Water pollution by pesticides is a serious global problem, and pesticide residues have become a sort of pervasive pollutant [15,16]. Pesticide residues exhibit low persistence, rapid breakdown and may be adsorbed onto sediments, and some of them are resistant to biodegradation, making it difficult to sample and determine their presence in water sources [17]. Consequently, detecting pesticide traces in aquatic settings is a matter of considerable interest. Monitoring this kind of pollution has become a common necessity for both developed and developing countries due to its dangerous effects in the long term and in light of extreme climate change. A comprehensive understanding of the different routes and mechanisms by which pesticides stabilize and travel off-site is essential for comprehending their fate and, consequently, for implementing risk assessment techniques.

Pesticide application and its environmental implications may be significantly impacted by changing environmental conditions brought on by climate change, notably heightened frequency of drastic weather or temperature increases. As a result, a comprehensive awareness of pesticide occurrences in surface water is becoming increasingly important [18]. The residues of pesticides don't remain confined only to the point of application, but they can be transported and spread to neighboring areas due to spray droplet evaporation, aerial movement, and volatilization during the application [19]. Additionally, residues may spread through water runoff, aeolian transport, or leaching and erosion of soil [20,21]. This sort of pollution could seriously threaten aquatic ecosystems and jeopardize human health [22-24]. As a result, several countries regularly monitor and evaluate the concentration of pesticides in surface water [25], with the aim of

minimizing pesticide impacts on the environment and safeguarding the quality of water resources [26,27]. Pesticide residue monitoring is highly significant, and its ongoing analysis is also necessary to evaluate its presence in surface water samples. Utilizing chromatographic methods comprising HPLC (high-performance liquid chromatography) and GC (gas chromatography), analysis of various pesticides classes in water has been performed effectively [1]. Furthermore, the combination of mass spectrometry and chromatography is also commonly recognized for its capacity to yield positive results [28]. Previous studies demonstrate that both (GC-MS and LC-MS) techniques can produce methods with low limits of detection (LOD) and quantification (LOQ), as well as very high accuracy and precision values [29-31].

There is a substantial deficiency of research inquiry regarding the phenomenon of contamination in Moroccan surface water. As part of the quality evaluation of surface water resources, the Oued Martil watershed has been the focus of several studies throughout the years. These studies have focused on evaluating the site's physicochemical and biological quality as well as biodiversity. On the other hand, other studies have examined the presence of polluting products emitted by different activities, and the results have indicated the presence of minor heavy metal pollution at levels that do not exceed established limits [5,32,33]. A gap was identified regarding the identification or contamination of surface water in the Oued Martil watershed by pesticides. Therefore, the Oued Martil River was selected as the aquatic environment for this study mainly because: (a) it is a significant river in northern Morocco, and (b) surface water in this Moroccan region had never been tested for the presence of pesticides before. This prompted us to evaluate pesticide contamination in the water by analyzing samples taken from four different locations. To address this informational gap, this article endeavors to carry out a diagnosis of the state and to formulate a preliminary assessment of pesticide pollution within the Oued Martil watershed by presenting findings pertaining to surface water contamination by pesticides. It considers the first study of its kind in the Martil watershed in this context. Therefore, it is deemed to be an augmentation to scientific research and represents a pioneering endeavor of its kind within the confines of the Martil watershed.

## 2. Materials and Methods

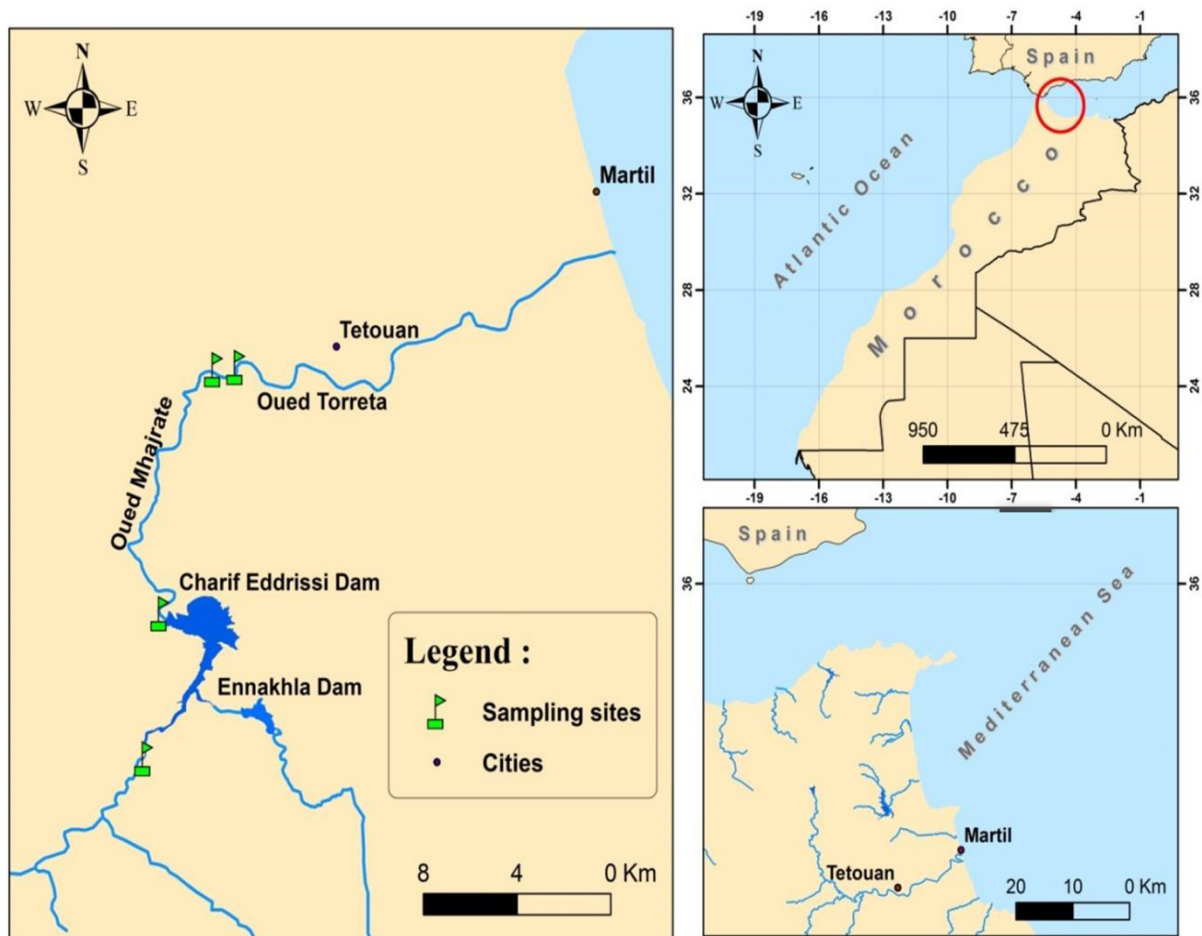
### 2.1. Study Area

The Oued Martil catchment exists in the northern part of Morocco, northwest of western Rif, specifically between the Lambert coordinates  $X_{\max}= 510026,386035$ ,  $Y_{\max}= 544222,33586$  and  $X_{\min}= 484200,139029$ ,  $Y_{\min}= 516578,078511$  [34] (**Figure 1**). It has a fairly dense hydraulic network, making it form a pivotal

hydrogeological area in the Rif Mountain range and also representing one of northern Morocco's biggest hydraulic systems [35]. Among the most prominent and largest tributaries to this watershed are Oued Khmiss, Oued Chekkour, and Oued Mhajrate (**Figure 2**). This water is transported by the major river known as the Oued Martil River, which passes through Tetouan and eventually pours into the Mediterranean at the level of the southern portion of Martil, which are two of northern Morocco's towns that have a particular and distinguished geostrategic position that connects the African and European continents. They are situated in the lower valley of the Oued Martil watershed, on both sides of the major canal, surrounded by the Ghorghiz and Dersa mountains [35,36]. The oued Martil River's length is 22 kilometers, extending a wealth of agricultural, tourist and industrial activities from its borders to its mouth [37,38].

The Oued Martil watershed stretches over a relatively large surface area of more than 1000 km<sup>2</sup> and is chiefly marked by mountainous landscapes. It is distinguished by diverse lithological morphologies like flysch, colluvium, and limestone. The watershed's altitudes differ tremendously, with an average altitude of 424 m, as do its

slopes, which also vary due to its location between the Mediterranean Sea and the Rif chain. Despite a few areas having lower relief and some very narrow plains, the bulk of the basin is characterized by extremely difficult and complex mountainous terrain. Additionally, this watershed has a substantial vegetation cover; notwithstanding, it is steadily decreasing significantly in its northern half [34,35,39,40]. The perimeter was chosen to be studied because it has an irrigation perimeter of MHAJRAT/AJRAS for huge regions meant for agricultural activity. This perimeter is located approximately 7 kilometers west of Tetouan on the road connecting Tetouan to Tangier, and more accurately in the confluence zone between the KHEMISS and MHAJRAT valleys, that is, downstream of the Oued Martil dam. Around 1500 hectares are irrigated at this location, which contains cereals, onions, cucumbers, tomatoes and fruit trees. The pesticides used in the study area fluctuate depending on the pest and type of crop, however there is no in-depth information about the types of pesticides used or the timing of their application. **Table S1** presents the physicochemical characteristics of the investigated pesticides.



**Figure 1.** Location of the Oued Martil watershed



**Figure 2.** Hydrographic Network of the Oued Martil watershed

**Table S1.** Physicochemical characteristics of the investigated pesticides. Source: Pesticide Properties Data Base (PPDB)

S. N°	Pesticides used	Solubility (20 °C)	DT <sub>50</sub> , water	Log Kow	Mobility
1	Prosulfocarb	13.2	Stable	4.84	Slightly
2	Azoxystrobin	6.7	Stable	2.5	Moderately
3	Cyprodinil	13	Stable	4	Slightly
4	Deltamethrin	0.0002	Stable	4.6	Non-mobile
5	Cyantranilprole	14.2	61	2.02	Moderately
6	Difenoconazole	15.0	Stable	4.36	Slightly
7	Fludioxonil	1.8	Stable	4.12	Non-mobile
8	Malathion	148	6.2	2.75	Moderately
9	Thiamethoxam	4100	Stable	-0.13	-
10	Tribenuron-methyl	2483	31.0	0.42	Mobile
11	Clofentezine	0.0342	3.1	4.09	Non-mobile
12	Hexythiazox	0.1	Stable	2.67	Non-mobile
13	Glyphosate	100000	Stable	-6.28	Non-mobile
14	Cypermethrin	0.009	Stable	5.55	-
15	Trifloxystrobin	0.61	40	4.5	Slightly
16	Difenoconazole	15.0	Stable	4.36	Slightly

DT<sub>50</sub>, water: half life by aqueous hydrolysis at 20°C and pH 7.0;

Kow: Octanol-water partitioning coefficient at pH 7 and 20 °C

## 2.2. Sampling

The sampling method depends on the objective of the investigation. In fact, target sampling involves taking samples from the pelvis's most vulnerable points, or from areas where the presence of contaminants is likely [41]. In order to find out the pesticide concentrations in the surface water of the Martil catchment, water samples were taken at 4 sampling sites chosen scattered in the Martil watershed: A (35,487340 / -5,439685), B (35,437053 / -5,444275), C (35,560505 / -5,412892), D (35,566673 / -5,407383). Sampling was performed every month, where water samples were collected over the period from January 2023 to July 2023, which included drought and low rainfall periods. Water samples were also used to identify physicochemical parameters. These sampling points were chosen with the aim of: i) providing an overview of the Oued Martil watershed, ii) considering the level of surface water contamination with pesticide at the study site. The first sampling campaign was carried out in January 2023, where samples were collected at the 4 points in the oued Martil watershed.

The water samples were collected using 1-liter glass bottles. To prevent air bubbles, the bottles were water-sealed. The water samples were kept under dark conditions in isothermal boxes with ice at 4°C until they reached the laboratory (on the same day) for analysis. In the laboratory, the water samples were filtered to 0.45µm, and stored in a refrigerator at -32°C in glass vials previously cleaned with hexane.

## 2.3. Physicochemical Parameters Analysis

Measuring physicochemical parameters is inevitable in any environmental study, since they can affect the composition and behavior of pollutants in the aquatic environment [42]. The physicochemical parameters

assessed in this study encompass pH, water temperature. These parameters were measured in the field with a specialized portable device (EUTECH CyberScan PCD 650), whereas other parameters such as nitrite (NO<sub>2</sub>-), nitrate (NO<sub>3</sub>-), orthophosphate (PO<sub>4</sub><sup>3-</sup>) and Chemical Oxygen Demand (COD) (Table 1) were analyzed in accordance with [43] recommendations.

## 2.4. Water Quality Evaluation

Water quality assessment represents a difficult and tricky procedure that depends on multiple descriptors. To resolve this issue, it could be required to employ quality indexes, which have been developed in various ways across the globe. The process of indexes development follows a common pattern throughout the world. A mathematical formula converts the concentrations of specific descriptors into a dimensional sub-index [44]. In our instance, the system created by Morocco's Directorate of Water Resources and Planning was adopted, founded on the notion of the weight quality index, which is calculated for every alteration and parameter [4]. Each parameter's index is computed via weighting, whereas each alteration's index is obtained by computing average of the weighted parameter values related to that alteration (IP) [45,46]. This index matches the relevant quality class in the general surface water quality evaluation grid, encompassing five classes (Table 2). The weighting index of every parameter was determined using this formula:

$$IP_{pa} = I_i + [(I_s - I_i) / (bs - bi)] * (bs - pa)$$

IP<sub>pa</sub>: Weighted index of the analyzed parameter

pa: Analyzed parameter

I<sub>i</sub>: Lower index

bi: Lower bound

I<sub>s</sub>: Upper index

bs: Upper bound

**Table 1.** Analytical Methods Used for the Determination of Studied Parameters

Parameters	Unit	Chemical Analysis Method
Nitrates (NO <sub>2</sub> -)	mg/l	Spectrophotometric titration using sodium hydroxide solution and sodium potassium tartrate.
Nitrites (NO <sub>3</sub> -)	mg/l	Spectrophotometric titration using sulfanilamide solutions and N-(1-naphthyl)-ethylenediamine 0.1%.
Orthophosphates (PO <sub>4</sub> <sup>3-</sup> )	mg/l	Colorimetric method using ascorbic acid.
COD	mg O <sub>2</sub> /l	Closed reflux method using potassium dichromate.

**Table 2.** Standards for the Global Weighted Index of Surface Water

Quality Class	Excellent	Good	Average	Poor	Very Poor
Temperature (°C)	0 - 20	20 - 25	25 - 30	30 - 35	35 - 40
pH	6.5 - 8.5	-	8.5 - 9.2	3 - 6.5 and 9.2 - 10	-
COD (mg O <sub>2</sub> /l)	15 - 30	30 - 35	35 - 40	40 - 80	80 - 2000
PO <sub>4</sub> <sup>3-</sup> (mg/l)	0 - 0.02	0.02 - 0.05	0.05 - 1	1 - 5	5 - 20
NO <sub>3</sub> . (mg/l)	< 10	10 - 25	25 - 50	> 50	-
NO <sub>2</sub> . (mg/l)	< 0.03	0.03 - 0.3	0.3 - 0.5	0.5 - 1	> 1

## 2.5. Pesticides Analysis Method

All of the investigated pesticide standards (**Table S1**) were acquired from Riedel-de Haen (Seelze, Germany). Acetonitrile was used to generate stock standard solutions of each chemical at a concentration of 500 µg/ml. All pesticides were diluted with a mobile phase (methanol and water (1:1)) to create working standard solutions with concentrations ranging from 25 to 400 µg/ml. The standard solutions were kept in storage at 4°C.

## 2.6. Solvents

HPLC grade dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>) (1), acetonitrile (2), methanol (3) and hexane (4), anhydrous sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>) (5) were obtained from Sigma-Aldrich and were employed as received. Using a Milli-Q water purification system supplied by Millipore (Bedford, MA, USA), ultrapure water was created by an ultrafiltration process.

## 2.7. Pesticide Residues Analysis

### 2.7.1. Sample Preparation and Extraction

A 250 ml aliquot of the water sample was accurately measured and introduced into a 1 L separatory funnel. A 30 ml volume of dichloromethane was subsequently added to the funnel. The funnel was then stoppered and agitated vigorously for a period of 4 minutes to facilitate the exhaustive partitioning of pesticides from the aqueous phase into the organic solvent. Following agitation, the mixture was allowed to stand undisturbed, enabling complete phase separation. The lower, denser organic (dichloromethane) layer, containing the extracted pesticides, was carefully collected into a clean receiving vessel. This extraction procedure was replicated two additional times with fresh 30 ml aliquots of dichloromethane, ensuring comprehensive recovery of the analytes.

### 2.7.2. Extract Drying

The three individual organic extracts obtained from the successive extractions were combined. This pooled dichloromethane solution was then passed through a glass filter funnel containing approximately 15 g of anhydrous sodium sulfate. This desiccation step was crucial for the quantitative removal of residual water co-extracted with the organic phase, thereby preventing potential interference in subsequent analytical steps.

### 2.7.3. Concentration and Reconstitution

The dried dichloromethane extract was quantitatively transferred to a round-bottom flask and concentrated using a rotary evaporator operating under reduced pressure and controlled temperature. This process effectively removed the bulk of the dichloromethane, yielding a concentrated "dry extract" containing the pesticide residues. The dry

extract was then precisely redissolved in 1 ml of methanol. This final methanol solution, containing the pre-concentrated pesticide residues, was transferred into small, sealed glass vials and stored prior to instrumental analysis.

## 2.8. High Performance Liquid Chromatography (HPLC) Analysis

For chromatographic separation, a CECIL 1010 HPLC system was used, featuring a C18 reverse-phase column (150 mm × 4.6 mm, 5 µm). The mobile phase, specifically developed for this purpose, was a 1:1 (v/v) mixture of HPLC-grade methanol (with 0.1% formic acid) and distilled water; this was prepared by combining 250 ml of each solvent. The system was purged by pumping the mobile phase at a flow rate of 1 ml/min. To determine the optimal detection wavelength for the analyte, a diluted solution in methanol was scanned. Finally, 10 µl of the extracted residues were injected into the system for analysis.

## 2.9. Analysis by Chromatography-GC/MS

Pesticides were analyzed using an Agilent Gas Chromatograph (GC) coupled with an Agilent Mass Spectrometer (MS) equipped with a Triple Axis Detector. Separation occurred on an Agilent Technologies DB-5MS capillary column (30 m × 0.25 mm × 0.25 µm film thickness), using helium as the carrier gas.

The GC oven temperature program was initiated at 60°C for 5 minutes, and then increased to 210°C at 15°C/min, holding for 5 minutes. The temperature was further elevated to 250°C at 5°C/min, held for 5 minutes, and finally raised to 300°C at 5°C/min, with a 13-minute hold. The inlet temperature was maintained at 250°C, and a 2 µL sample was introduced via an autosampler.

The mass spectrometer utilized Electron Impact (EI) mode. A temperature of 280°C was selected for the transfer line, 150°C for the quadrupole, and 230°C for the ion source.

# 3. Results and Discussion

## 3.1. Physico-Chemical Parameters

Studying physicochemical parameters is indispensable for acquiring an accurate understanding of the water's quality, as it enables the comparison of the different physicochemical parameter found with standard values (Table S2 and Table 2). To monitor the pollution origin in the surface waters of the Oued Martil watershed, we conducted tests on pH, temperature, chemical oxygen demand (COD), phosphate (PO<sub>4</sub><sup>3-</sup>), nitrate (NO<sub>3</sub><sup>-</sup>) and nitrite (NO<sub>2</sub><sup>-</sup>).

3.1.1.1. pH

The pH of water measures the acidity, alkalinity, or neutrality of an aquatic environment. It varies according to the concentration of CO<sub>2</sub> and CO<sub>3</sub><sup>2-</sup>. It influences a large number of biological and chemical processes. This parameter can undergo significant fluctuations due to the action of pollutants with acidic or basic characteristics. It particularly depends on the water's origin and the geological characteristics of the environment it passes through. The observed values reveal that the pH is generally neutral for all stations, except for S4 in the months of June and July, where alkaline pH values above 7 were noted (**Figure 3**).

3.1.2. Temperature

The minimum temperature recorded in the study area during the two campaigns was 19.7°C, and the maximum reached 33°C. These variations in water temperature follow those of the air, incorporating the region's climate. However, the water temperature is influenced by several primarily non-climatic local factors. Among these morphological factors are slope and altitude, but also the percentage of riparian vegetation cover, which also affects the shading of watercourses. Indeed, we noticed a decrease in water temperature with altitude, slope, and shading of the sampling points. Thus, stations S3 and S4 showed maximum temperatures exceeding 20°C in July (**Figure 4**).

**Table S2.** Descriptive Statistics of Physicochemical Parameters at Four Study Sites

Parameter	Station	Mean	Std Dev	Min	Max
pH	S1	6.70	0.32	6.28	7.20
	S2	6.88	0.38	6.32	7.27
	S3	6.82	0.43	6.22	7.20
	S4	7.24	0.71	6.45	8.40
Temperature (°C)	S1	24.09	3.95	19.70	29.70
	S2	24.01	3.89	19.70	30.00
	S3	24.94	3.82	20.02	31.20
	S4	25.48	4.52	21.00	33.00
COD (mg O <sub>2</sub> /l)	S1	12.89	0.99	11.90	14.50
	S2	14.52	2.24	12.20	18.50
	S3	18.54	7.88	11.64	30.00
	S4	18.12	7.40	11.56	30.00
NO <sub>2</sub> (mg/l)	S1	0.06	0.07	0.00	0.19
	S2	0.06	0.11	0.00	0.30
	S3	0.06	0.11	0.00	0.30
	S4	0.02	0.04	0.00	0.10
NO <sub>3</sub> (mg/l)	S1	3.77	4.53	0.00	11.55
	S2	2.92	3.70	0.00	10.20
	S3	2.41	1.58	0.13	4.30
	S4	1.28	1.85	0.00	5.00
PO <sub>4</sub> <sup>3-</sup> (mg/l)	S1	1.00	0.64	0.33	1.86
	S2	1.17	0.36	0.78	1.65
	S3	0.97	0.50	0.15	1.51
	S4	0.93	0.55	0.01	1.64

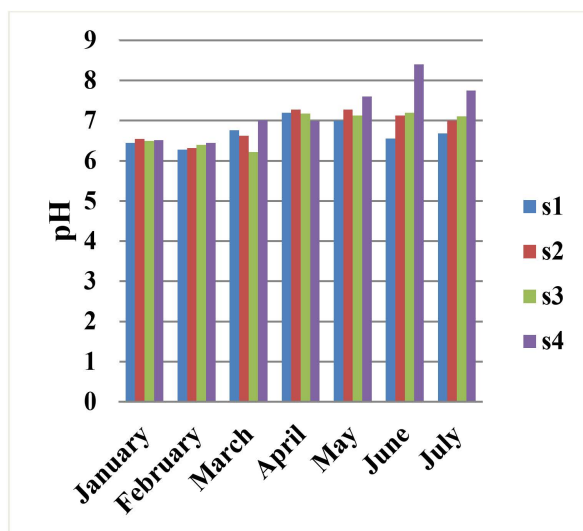


Figure 3. pH variation of the 4 sites

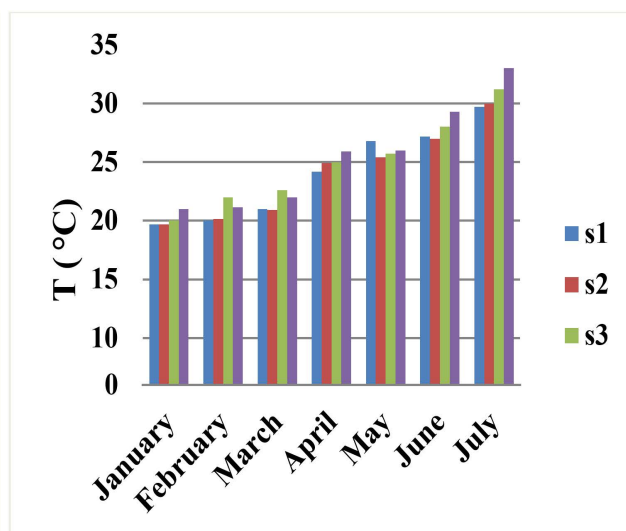


Figure 4. Temperature variation of the 4 sites

### 3.1.3. COD

Chemical oxygen demand is defined as the quantity of oxygen used by the water's chemically oxidizable components contained in the water, regardless of their origin (ferrous iron, nitrates, ammonium, sulfides, and chlorides) [47]. The values recorded in the study area indicate an unstable change in COD at the surveyed station from January to July. We obtained a value range between 11.6 mg/l at S3 in March and 30 mg/l at S4 in June (Figure 5).

### 3.1.4. $NO_2^-$

Nitrites act as intermediate ions linking nitrate and ammoniacal nitrogen, a property that explains their remarkably low concentrations in aquatic environments [48]. Nitrites are therefore the most oxidized and abundant form of nitrogen as they result from the nitrification process, which is the oxidation of organic and ammoniacal nitrogen. They are generally found in low concentrations in surface waters. In our study area, we recorded a zero value in the majority of stations during the different months and a maximum value of 0.19 mg/l at station S1 in April (Figure 6).

### 3.1.5. $NO_3^-$

Nitrates are the most soluble forms of nitrogen in water. Natural water normally contains nitrates at doses varying with the seasons. The concentrations of these naturally occurring ions in surface water are a few milligrams per liter [49]. Nitrates originate from agricultural activities (fertilizer application or livestock farming) after the leaching of agricultural lands. They also originate from ammonium oxidation and organic nitrogen mineralization. The recorded concentrations are generally low throughout

the different months, with maximum values recorded in January of 11.55 mg/l at station 1 and 10.2 mg/l at station 2 (Figure 7).

### 3.1.6. $PO_4^{3-}$

Phosphorus can be present in water in ionized form (orthophosphates). It is generally responsible for accelerating eutrophication processes. Domestic and industrial effluents, as well as the drainage of fertilized agricultural soils, discharge phosphorus into the water. Typically, their content should not exceed 1 mg/l, and any increase causes a degradation of water quality. The figure shows values recorded at the stations. Analysis of the findings highlights that the orthophosphate concentration in the surface waters of the Oued Martil watershed is greater than 0.005 mg/l for the majority of the studied stations and across the different months (Figure 8).

## 3.2. Pesticides

This is the first study to investigate the detection of pesticides in the surface water of the Oued Martil watershed. In the present research, a total of 16 pesticides were analyzed in surface water of oued martil catchment (Table S1). The concentrations of pesticides were determined in micrograms per liter ( $\mu\text{g/l}$ ) in the water samples. It is imperative to know the extent of pesticide contamination in the surface water within this watershed, given the diverse range of activities in the research area, which includes agricultural production. Moreover, the selected area involves a former public dump and an area near the Paloma hotel in Tetouan. The fact that the study was conducted in this watershed is also significant since it may offer a more accurate depiction of the quantities of pesticides in the study area's surface water given the lack of information in this context.

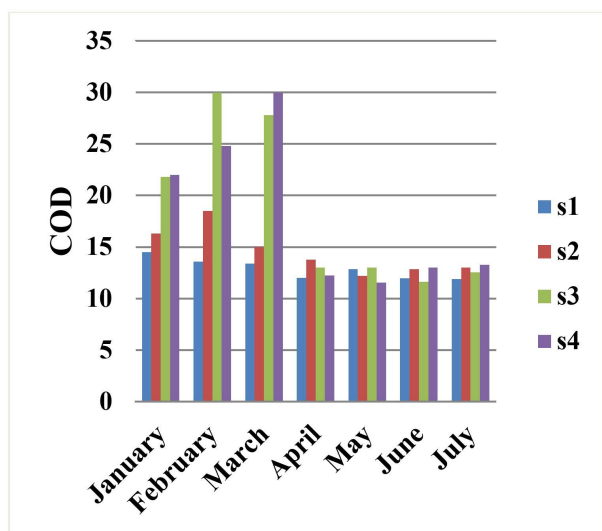


Figure 5. COD variation of the 4 sites

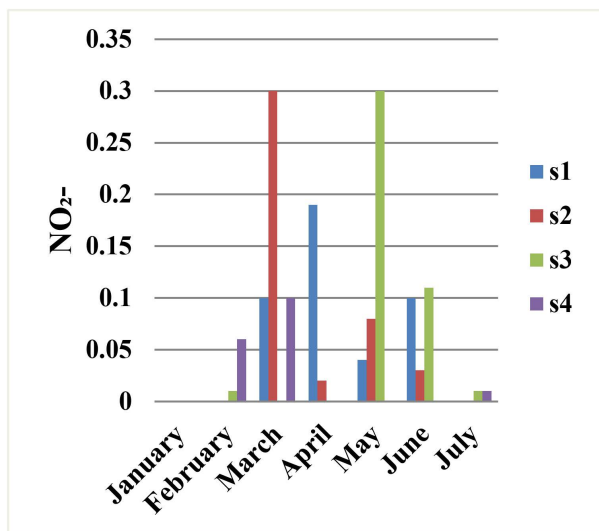


Figure 6. NO<sub>2</sub><sup>-</sup> variation of the 4 sites

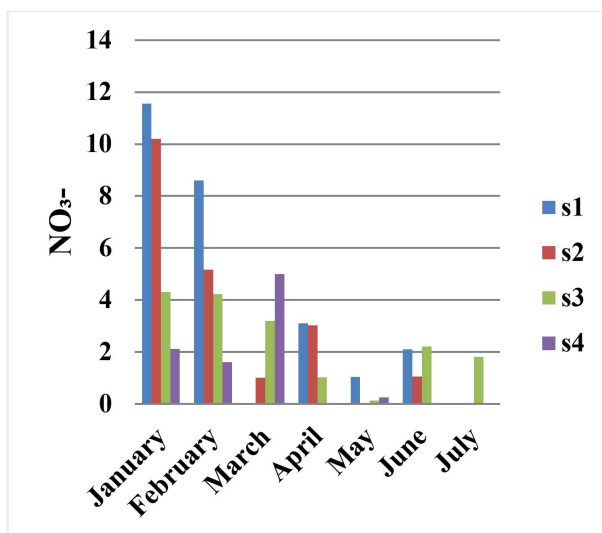


Figure 7. NO<sub>3</sub><sup>-</sup> variation of the 4 sites

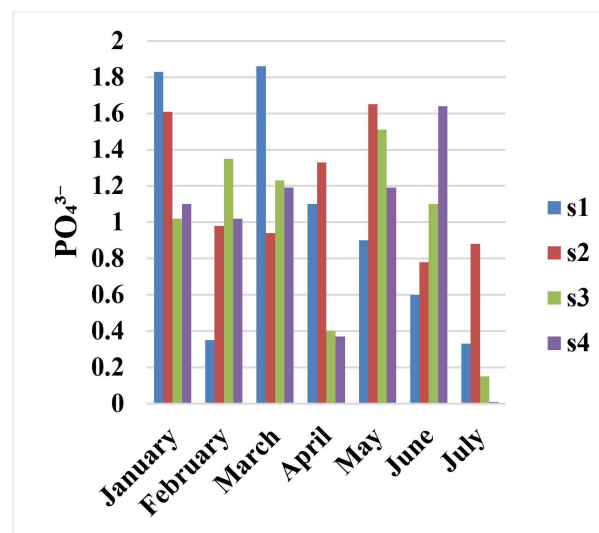


Figure 8. PO<sub>4</sub><sup>3-</sup> variation of the 4 sites

The analysis of the chromatograms of our samples taken along the Oued Martil catchment and comparison of the retention times of the various compounds appearing on the chromatogram with the standards enabled us to observe the total absence of pesticide contamination in our samples. Indeed, no sample peak was recorded at a time equivalent to the standards' injection retention period. Pesticide concentrations were always below the LOD and typically very low in the samples, indicating no evidence of pesticide contamination at all points. While all findings indicated trace amounts (below LOQ) or negative (non-detected) values, certain inferences could explain these negative results.

The non-detection of pesticides in this study may be due to the limited sensitivity of our analytical tools. Consequently, pesticides were not detected because their concentrations may be lower than the detection limit of the instruments used.

From an anthropogenic perspective, the non-detection may also be attributable to the fact that the samples were gathered in a traditional and domestic agricultural area. Unlike large-scale or intensive agriculture operations, these systems frequently use low persistent chemicals or lower rates of pesticide application. Moreover, farmers may not have easy access to controlled agrochemicals, which could lead to irregular or unrecorded use. This is consistent with the literature that points out that the area lacks official information on the use of pesticides, as well as the farming practices and customs practiced in this area do not necessitate the utilization of significant amounts of pesticides [50].

The lack of pesticides in water may be related to their mobility or persistence in soil [51], and it's also possible that the physicochemical features of these pesticides (high volatility, high degradability, short residence time, etc.) contribute to their high susceptibility to degradation.

Enhancing the physicochemical characteristics that may be connected to pesticide persistence in the environment and detection or non-detection is crucial since these attributes may be linked to ongoing detection [52]. However, pesticides' fate is not solely determined by their physicochemical properties, but also by the quantity of treatments applied and irrigation conditions [53].

Pesticides are transported from the target site to other off-target plants or environmental matrices by a variety of mechanisms, including surface runoff, adsorption, etc. [54]. There are also other elements impacting the pesticide movement process and final concentration of pesticides, notably climatic factors (duration, quantity, and intensity of precipitation, air movement, and air temperature). A low precipitation level implies no surface runoff and no pesticide dispersal into the watercourse [52]. Furthermore, according to [55], sunlight and temperature also have an impact on all biotic and abiotic changes in pesticide reactions. Furthermore, because the samples were gathered throughout varying seasons, the pesticides used may also vary according to the target to be attained [52]. It was noted that in comparison to the first campaign, which took place in January 2023, the July 2023 campaign saw greater temperatures and less precipitation. These results are consistent with the period in which the campaign was conducted. Morocco has witnessed a sharp decrease in rainfall and an increase in temperature in recent years. It is relatively difficult to compare these results with other research results due to the paucity of data related to the study of the detection of pesticides and their metabolites or residues in the surface water of the oued Martil watershed. The same findings have been reported in other Mediterranean catchments that are mostly used for small-scale farming, where concentrations of pesticides can surge substantially during autumn-winter storm events but are often below detection limits during dry-season base-flows [56-58]. This hydrological regime supports our finding that  $\text{NO}_3^-$  concentrations were higher throughout the rainy season and lower in the dry season, implying enhanced surface runoff and transport of dissolved pollutants. In addition,  $\text{PO}_4^{3-}$  concentrations remained moderately high throughout most of the sampling period, probably owing to continued inflows from agricultural activities, domestic wastewater within the watershed. This persistent nutrient load highlights the potential for continued contaminant inputs to the aquatic environment. Therefore, the absence of detectable pesticides amid these consistent nutrient signals highlights the role of environmental mitigation processes (e.g. strong soil sorption, volatilization, or rapid degradation, which may effectively remove residues from the water column before they are detected. This reinforces the hypothesis that low

rainfall and high temperatures during the sampling period may have contributed to the absence of detectable pesticide residues in the surface water samples from the oued Martil watershed. Additionally, the disparity between agricultural activities and the lack of measurable contamination could be attributed to the fact that the watershed's vegetation cover and diverse topography may act as natural buffers, thereby reducing runoff directly into rivers. In upper watersheds, riverine vegetation and rugged topography can serve as filters, absorbing pesticide residues before they enter surface water.

### 3.3. Descriptive Statistics of Physicochemical Parameters at Four Study Sites

**Table S2** presents the descriptive statistics for the physicochemical parameters across the four study sites from January to July. pH values generally remained stable, with mean values ranging from 6.70 (S1) to 7.24 (S4). Station S4 exhibited the highest mean pH and also the largest standard deviation (0.71), indicating greater variability in pH at this site compared to the others. Mean water temperatures increased from Stations S1 to S4, consistent with expected downstream warming. Chemical Oxygen Demand (COD) showed notable differences, with higher mean concentrations observed at Stations S3 (18.54 mg  $\text{O}_2/\text{l}$ ) and S4 (18.12 mg  $\text{O}_2/\text{l}$ ) compared to S1 and S2. Furthermore, the significantly larger standard deviations for COD at S3 (7.88) and S4 (7.40) suggest more pronounced fluctuations in organic pollution levels at these locations. Nitrite ( $\text{NO}_2^-$ ) concentrations were generally low across all stations, with most means below 0.06 mg/l. Nitrate ( $\text{NO}_3^-$ ) and Orthophosphate ( $\text{PO}_4^{3-}$ ) levels also varied, with some stations showing wider ranges and higher standard deviations, particularly for  $\text{NO}_3^-$  at S1 (Std Dev: 4.53), suggesting more sporadic or localized inputs.

### 3.4. Water Quality Evaluation of the Oued Martil Watershed

The results reported in **Table S3** show that the surface water in the Martil watershed is of good quality, with physicochemical parameters conducive to aquatic life. These findings align with the work of [50]. Additionally, the absence of pesticide traces confirms these positive results, indicating that the water is not contaminated by harmful chemicals from agricultural practices. However, it remains essential to continue monitoring these parameters to ensure that water quality is maintained, particularly in the face of growing environmental challenges.

**Table S3.** Results of the physical and chemical parameters of surface water for the four study sites

Parameters	pH				T (°C)				COD (mg O <sub>2</sub> /l)				
	s1	s2	s3	s4	s1	s2	s3	s4	s1	s2	s3	s4	
Months	January	6,45	6,54	6,49	6,51	19,7	19,7	20,02	21	14,5	16,3	21,8	22
	February	6,28	6,32	6,40	6,45	20,03	20,16	22	21,16	13,6	18,5	30	24,8
	March	6,76	6,62	6,22	7,01	21	20,9	22,6	22	13,4	15,02	27,8	30
	April	7,2	7,27	7,18	6,99	24,2	24,9	25	25,9	12,02	13,77	13,01	12,24
	Mai	7	7,27	7,13	7,6	26,8	25,4	25,7	26	12,86	12,2	13	11,56
	June	6,55	7,13	7,2	8,4	27,18	27	28,04	29,3	11,98	12,87	11,64	13
	July	6,68	7	7,11	7,75	29,7	30	31,2	33	11,9	13,01	12,56	13,27
Parameters	NO <sub>2</sub> - (mg/l)				NO <sub>3</sub> - (mg/l)				PO <sub>4</sub> <sup>3-</sup> (mg/l)				
Station	s1	s2	s3	s4	s1	s2	s3	s4	s1	s2	s3	s4	
Months	January	0	0	0	0	11,55	10,2	4,3	2,11	1,83	1,61	1,02	1,1
	February	0	0	0,01	0,06	8,6	5,17	4,22	1,6	0,35	0,98	1,35	1,02
	March	0,1	0,3	0	0,1	0,01	1,01	3,2	5	1,86	0,94	1,23	1,19
	April	0,19	0,02	0	0	3,1	3,02	1,02	0	1,1	1,33	0,4	0,37
	Mai	0,04	0,08	0,3	0	1,04	0	0,13	0,25	0,9	1,65	1,51	1,19
	June	0,1	0,03	0,11	0	2,1	1,05	2,21	0	0,6	0,78	1,1	1,64
	July	0	0	0,01	0,01	0	0	1,81	0	0,33	0,88	0,15	0,01

### 4. Conclusions

Water monitoring studies are of utmost importance due to the high potential for contamination from the numerous chemicals used in everyday life. This research investigated surface water quality in the Oued Martil watershed. The Oued Martil watershed's physicochemical parameters showed site-specific seasonal oscillations and variations from January to July. Over the course of the study, temperature clearly increased in response to seasonal variations, while nitrate levels generally declined as the months grew warmer. In addition to nitrite and phosphate contents being generally low or varying between stations, pH levels often fell between neutral and slightly alkaline.

It is frequently difficult to know the exact type and quantity of pesticides used by small farmers, and this information is frequently inaccurate because it is not documented and does not adhere to the measures recommended by the National Office for Health Safety of Food Products. The Martil watershed's estuary knows a regular agricultural activity, which is expected to increase over time to meet the needs, namely in light of the population surge, which would lead to the emergence of new population settlements, which could contribute significantly to the pollution of surface water resources. Analysis of surface water in the Oued Martil watershed revealed that all samples taken from the watershed contained residual levels of pesticides that were below the quantification limits. Thus, periodic water analysis and

disseminating pertinent current information remain a fundamental requirement, to monitor and detect the changes that can unbalance the environment, prevent pesticide-related damage, and reduce the pollution of aquatic environments. The evaluation emphasizes the necessity of ongoing pesticide level monitoring in the Oued Martil watershed. The absence of pesticides points to a complicated web of interrelated factors determining pesticide contamination. From the perspective of discovering the causes of this pesticide deficiency, it is advised that more detailed monitoring research be done on the biological effects, sediment analysis, and temporal change in order to fully comprehend the dynamics of pesticides in this system. These studies will enable us to further understand and explain their disappearance in this study area.

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