

Article

Molecularly Imprinted Polymer with Photocatalytic Activity for the Adsorption and Degradation of Naproxen and Its Application in Real Water Samples

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Abstract

This research focused on the development and characterisation of molecularly imprinted polymers (MIPs) modified with titanium dioxide (TiO₂) for the adsorption and photocatalytic degradation of sodium naproxen (NPX). Different percentages of TiO₂ (5% and 25%) were tested and compared to non-imprinted polymers (NIPs). FT-IR analysis confirmed the interaction between methacrylic acid and TiO₂, promoting the formation of specific binding sites and presenting a good imprinting factor. The results showed that the MIP with 5% TiO₂ had the highest adsorption and retention capacity, attributed to the imprinting effect and the reduced interference from TiO₂. The surface of the MIPs is heterogeneous, as it was indicated by the Freundlich isotherm model. The K_F for the MIP with 25% of TiO₂ was higher than for the materials with 5%; values for the MIP/TiO₂ 5% and the NIP/TiO₂ 5% K_F were 4.808 and 4.163 (mg/g)(L/mg)^{1/n} respectively, while for the MIP/TiO₂ 25% was 6.542 (mg/g)(L/mg)^{1/n} and for the NIP/TiO₂ 25% it was 2.736 (mg/g)(L/mg)^{1/n}. Kinetic studies followed the pseudo-second-order model, suggesting more active binding sites in MIPs. Photocatalytic experiments achieved 60% degradation, demonstrating the degradation performance of MIPs; however, this behavior is restricted by the slow degradation of NPX. The materials were evaluated using a water sample (Querétaro River, México); the sample was preconcentrated and analyzed, detecting a concentration of 0.332 mg/L of NPX. This finding highlights the MIPs' potential application in environmental monitoring and treatment; nevertheless, due to the recalcitrant nature of NPX, MIPs should be used along with other advanced treatment methods to achieve effective removal.

Keywords: molecularly imprinted polymer; photocatalysis; naproxen; contaminants of emerging concern; river water sample



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1. Introduction

Microcontaminants are compounds of anthropogenic origin, for example, they can originate from agriculture, industry, water treatment, and mass consumption. They are defined as substances present in very low concentrations (usually micrograms per liter or nanograms per liter). It is essential to distinguish them from contaminants of emerging concern (CECs), whose presence in the environment has been recently detected or whose risks are only beginning to be recognized, these pollutants are generally unregulated or subject only to partial regulations [1]. Some compounds can be both emerging and micropollutants—for example, pharmaceuticals in wastewater, hormones, PFAS, or pesticides appearing at new trace levels—but the two terms are not interchangeable [2].

The presence of micropollutants has been reported since the 1970s using gas and liquid chromatography [3–5]. However, for many of these compounds—especially polar or non-volatile ones—the detection limits achievable with the detectors available at that time (e.g., UV-Vis, flame ionization, or early mass spectrometers) were insufficient to quantify trace concentrations (typically $\mu\text{g/L}$ or lower). Consequently, reliable monitoring data were difficult to obtain, which hindered the establishment of regulatory frameworks [6–8]. Important research, such as John Warren Smith's doctoral thesis in 1968 [9] described methods for analyzing microcontaminants in wastewater (in the mg/L range), demonstrating effects such as the inhibition of respiratory enzyme activity and the death of exposed fish. Since then, with the introduction of more sensitive analytical techniques, it has become clear that these contaminants, even at the much lower concentrations reported today (ng/L – $\mu\text{g/L}$), exert a wide range of toxic effects. Recent research has documented impacts such as the development of bacterial resistance [10], endocrine disruption effects including feminization of fish and alterations in their reproductive cycles [11,12], decreased egg fertilization, damage to the immune system [13,14], as well as human health problems such as polycystic ovary syndrome, breast cancer, miscarriage, endometrial disorders, and premature births [15–18]. Additionally, it has been highlighted that the combined effects of two or more micropollutants can be significantly different (synergistic or antagonistic) from those of the individual compounds [19,20].

CECs include personal care products, industrial additives and agents, flame retardants, perfluorinated compounds, and pharmaceuticals [21]. These contaminants are frequently detected at low concentrations but can pose significant risks to aquatic ecosystems and human health. Consequently, the scientific community is actively researching advanced analytical methods for their trace detection and innovative remediation technologies for their removal from water [22]. In this context, the development of multifunctional materials combining selective adsorption and catalytic degradation has gained particular interest. A recent perspective article provides a comprehensive overview of adsorbent design strategies, mechanistic interpretation of pollutant removal, and practical implications for water treatment [23]. Recent studies have explored the synergistic potential of integrating molecularly imprinted polymers (MIPs) with titanium dioxide (TiO_2) photocatalysts. For instance, a TiO_2 -based MIP system has demonstrated enhanced selectivity and efficiency for the targeted removal of persistent pollutants like PFAS [24]. Several studies also highlight the significant progress in using MIPs for pollutant capture and photocatalytic degradation, emphasizing their role in advanced oxidation processes [24–27]. Another study focused on a different application by developing a zein-based active packaging film integrated with TiO_2 nanotube arrays, showcasing the versatility of TiO_2 -based composites for active surface applications [28]. A conventional polymer- TiO_2 system [29] has demonstrated that the dispersion of TiO_2 nanoparticles in a polymer matrix can enhance photocatalytic activity; however, the present study goes a step further by using a molecularly imprinted polymer (MIP) as the matrix, which adds selective recognition of the target pollutant.

These combined approaches, which focus on detection, adsorption, and degradation, underscore the potential of the work on a TiO₂-embedded MIP for the selective removal and degradation of naproxen from aqueous environments.

Among the pharmaceutical microcontaminants, non-steroidal anti-inflammatory drugs (NSAIDs) are of special concern due to their easy acquisition and their constant appearance in environmental monitoring studies [30]. Naproxen (NPX) is an NSAID derived from propionic acid with analgesic and antipyretic properties. The NPX chemical structure (Figure 1) consists of two benzene rings fused with a methoxy group on one ring and a propanoate in the other. At first (1974), sodium naproxen was sold only by prescription, and in 1994, it was released for sale over the counter [31].

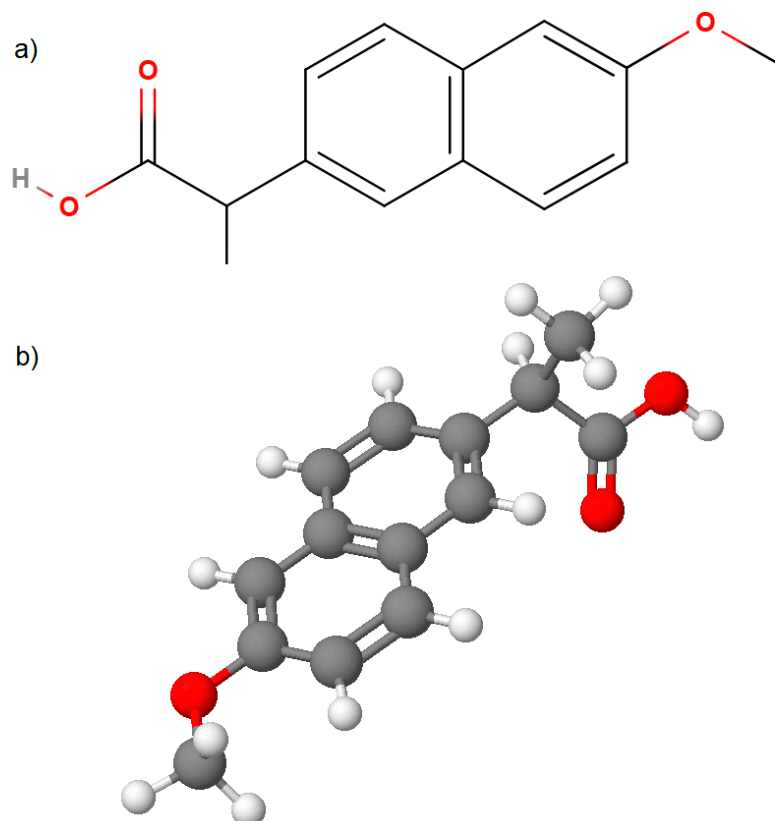


Figure 1. (a) 2D and (b) 3D chemical structure of naproxen. This Figure was made with the web-application MolView found in <https://molview.org/> (accessed on 19 October 2025).

Although NPX is produced for medical purposes, it has now become a risk to aquatic organisms because the compound itself and its metabolites can accumulate in fish due to the suppression of the metabolizing enzyme activity, affect the mRNA expression, cause renal effects, and thyroid disruption. For example, in Jordanella, Floridae NPX exposure caused a decrease in egg fertilization and inhibited *Ceriodaphnia dubia* growth [32]. In México, NPX induced oxidative stress and was considered hazardous for the common carp *Cyprinus carpio* in the Madín reservoir with concentrations of 0.18 to 0.21 µg L⁻¹ [33]. On the other hand, the photolysis byproducts of NPX are more toxic to microorganisms such as bacteria, *Brachionus calyciflorus*, *Thamnocephalus platyurus*, *Ceriodaphnia dubia*, *Vibrio fischeri*, and *Daphnia magna* [33–35].

Molecularly imprinted polymers (MIPs) are synthesized materials designed to act as hosts of a target molecule using a covalent or non-covalent approach. The covalent approach consists of the reversible covalent bond formation. On the other hand, the bonding in the non-covalent approach depends on reversible physical interactions such

as hydrogen bonding, van der Waals interactions, π - π stacking, hydrophobic interaction, or metal coordination [36,37]. The non-covalent approach follows the intramolecular phenomena of the molecular recognition fundamentals [38]. The flexibility in the MIP design allows multiple applications, such as separation or isolation of specific compounds, sensors and biosensors, drug delivery, catalysis, and environmental applications [39]. Thus, MIPs with photocatalytic activity and retention properties are viable as a strategy for wastewater treatment.

Molecularly imprinted polymers (MIPs) offer several advantages for trace analysis of CECs: high selectivity for target molecules, reusability, chemical stability under harsh conditions, and compatibility with various detection platforms. They can be used as selective sorbents in solid-phase extraction (MISPE) to reduce matrix effects, or integrated into sensing devices [40,41]. However, MIPs also have limitations: heterogeneous binding sites leading to non-specific adsorption, template leakage (especially in non-covalent approaches), difficulties in water-compatible synthesis, and a relatively low binding capacity compared to non-imprinted polymers. Additionally, the synthesis optimization can be time-consuming. These trade-offs must be considered when designing a MIP-based strategy for pollutant removal or detection [42,43].

This study aims to develop and analyze a molecularly imprinted polymer with high adsorption capacity and excellent photocatalytic performance for NPX degradation. Titanium dioxide (TiO_2) was incorporated into the MIPs to add photocatalytic properties to the polymer matrix. Non-imprinted polymers were synthesized following the same conditions and methods for comparative analysis. Adsorption modeling and kinetics studies were conducted for both MIPs and NIPs. Finally, NPX photodegradation experiments in synthetic and real samples (synthetic samples were prepared using deionized water and real samples were obtained from Querétaro River) were performed to evaluate the effectiveness of the MIPs and NIPs in a photocatalytic reaction.

2. Methods and Materials

2.1. Materials

All reagents, methacrylic acid (MAA) as functional monomer, ethylene glycol dimethacrylate (EDGMA) as crosslinker, azobisisobutyronitrile (AIBN) as initiator, naproxen sodium (NPX-Na) as template, and titanium dioxide (anatase) with 99.8% purity as photocatalyst element, were purchased from Sigma-Aldrich (Darmstadt, Germany). The organic solvents, methanol, and toluene (porogen), were obtained from Tedia Company Inc. (Fairfield, OH, USA).

2.2. Synthesis of Molecularly Imprinted Polymers

Two molecularly imprinted polymers (MIPs) were synthesized using 5% and 25% titanium dioxide (TiO_2). The synthesis was conducted based on the method previously reported by our research group [44], following a non-covalent approach with minor modifications.

First, a mixture of 0.340 mL of MAA, the appropriate concentration of TiO_2 , 3.125 mL of EDGMA, and 4 mL of toluene was prepared. Afterwards, 67.6 mg of sodium naproxen (NPX-Na) (previously dissolved in methanol) was added and stirred for 10 min. Subsequently, 2 mL of AIBN was added to the mixture. This solution was then slowly injected into a flask that had been pre-filled with nitrogen to maintain an inert atmosphere. The reaction was carried out at a constant temperature of 70 °C in a glycerin bath with continuous stirring at 100 rpm for 24 h. Then, the polymer was collected and washed with hot water to eliminate residual materials. Non-imprinted polymers (NIPs) were prepared following the same method without the NPX-Na. The resulting polymers were crushed, followed by ten washing cycles using 10 mL of hydrochloric acid (HCl) (0.1 N) in methanol to remove

the template from the MIPs. In each cycle, the materials were sonicated for 10 min and centrifuged at 2500 rpm for 5 min.

2.3. Naproxen Analytical Determination

NPX-Na determination was performed in a high-performance liquid chromatograph with a photodiode array (Agilent 1260 infinity series, Agilent, Santa Clara, CA, USA). An XDB C18 column was used (4.6×250 mm, $5 \mu\text{m}$ particle size). The mobile phase consisted of acetonitrile and acidic water (60:40), using orthophosphoric acid to reach a pH of 2.6. An isocratic flow of 1 mL min^{-1} was used, the injection volume was set at $10 \mu\text{L}$, and the samples were measured at a wavelength of 220 nm. All samples were filtered using a PVDF (polyvinylidene fluoride) hydrophilic filter with a $0.45 \mu\text{m}$ pore diameter. Previous studies reported a detection limit of 0.018 mg/L and a quantification limit of 0.110 mg/L [45].

2.4. FT-IR

The infrared spectra of the polymeric materials, TiO_2 , and NPX-Na were recorded in the $4000\text{--}500 \text{ cm}^{-1}$ range using an IRAffinity-1S (Shimadzu Kyoto, Japan) Fourier Transform Infrared Spectrophotometer (FT-IR), the samples were analyzed using the total attenuated reflectance method (ATR).

2.5. Point of Zero Charge

MIPs and NIPs surface charge was determined using the acid–base titration proposed by Kuzin and Loskutov [46]. Separate solutions of HCl, NaOH, and NaCl (0.1 N) were prepared, and 50 mg of MIP or NIP were placed in a centrifugal tube. Either HCl or NaOH was placed in the same tube, and NaCl was added for a total volume of 12.5 mL. The neutralizing solutions ranged from 5 to 0.1 mL. The solution was stirred using a vortex and incubated for one hour. Afterward, the pH was measured using a 700 series benchtop pH meter. A blank without MIP or NIP was performed under the same conditions.

The surface charge was calculated using Equation (1):

$$C_S = \frac{C_N(V_B - V_A)}{m} \times F \quad (1)$$

where C_S is the surface charge of the adsorbent at a given pH value (Coulomb/g or C/g), C_N is the concentration of the neutralizing solution (mol L^{-1}), V_A is the volume of the NaOH or HCl solution used in the experiment, V_B is the volume of 0.1 N NaOH or HCl used during the blank experiment, m (g) is the amount of MIP or NIP employed, and, F is the Faraday constant ($96,485.0 \text{ Coulomb/mol}$).

2.6. Adsorption Test

Adsorption equilibrium tests were carried out in a 100 mL flask with 50 mL of an NPX-Na solution (1 to 100 mg/L) and 20 mg of the polymer under constant agitation (300 rpm). Samples were taken and filtered (PVDF hydrophilic filter with $0.45 \mu\text{m}$ pore diameter) at 0, 2.5, 5, 10, 20, 30, 45, 60, and 90 min. The pH of solution was not modified for the analysis (pH = 6). Three replicates were made.

2.7. The Adsorption Capacity, Retention Percentage, and Imprinting Factor

The values obtained in Section 2.6 are useful for calculating adsorption capacity, retention percentage, binding capacity or adsorption capacity, and imprinting factor. The adsorption capacity is determined using the following equation:

$$q_{exp} = \frac{V(C_0 - C)}{W} \quad (2)$$

where q_{exp} is the experimental adsorption capacity or the amount of NPX bound per unit mass of the adsorbent (mg/g), C_0 is the initial concentration (mg/L), C is the equilibrium concentration (mg/L), V is the volume of the standard solution (L), and W is the MIP or NIP weight (g).

The MIP efficiency is determined by the retention percentage (%R), Equation (3) was employed to calculate it:

$$\%R = \frac{C_0 - C}{C_0} \times 100 \quad (3)$$

where C_0 is the NPX-Na initial concentration (mg/L), and C is the NPX-Na equilibrium concentration (mg/L).

The imprinting factor determines the MIP affinity for the target molecule and is a measure of the imprinting grade in the MIP. To achieve a successful imprinting, the IF should be greater than 1 [47]:

$$IF = \frac{q_{MIP}}{q_{NIP}} \quad (4)$$

where q_{MIP} and q_{NIP} can be calculated using Equation (2).

2.8. Adsorption Isotherms Models

To better understand the adsorption behavior of the materials, the Langmuir and Freundlich models were studied.

The Langmuir equation is the following:

$$q = \frac{K_L q_{max} C_e}{1 + K_L C_e} \quad (5)$$

where C_e (mg/L) is the adsorbate concentration at equilibrium, q_{max} is the maximum amount of adsorbate per amount of adsorbent at equilibrium (mg g⁻¹), and K_L (L mg⁻¹) is the Langmuir constant related to the adsorption energy.

A separation factor (R_L) can be calculated using K_L . When $R_L = 0$, the adsorption is irreversible; when $R_L < 1$ implies that adsorption is favorable; $R_L = 1$ indicates linear adsorption, and finally, adsorption is unfavorable when $R_L > 1$:

$$R_L = \frac{1}{1 + K_L C_0} \quad (6)$$

The Freundlich model describes the adsorbent as a heterogeneous surface with a possible interaction among particles; thus, the stacking of these particles allows the formation of a multilayer onto the adsorbent surface [48].

The Freundlich model follows the next equation:

$$q = K_F C_e^{\frac{1}{n}} \quad (7)$$

where K_F [(mg g⁻¹)(L/mg)^{1/n}] is the Freundlich constant, $1/n$ is the adsorption intensity or surface heterogeneity, if $0 < 1/n < 1$ adsorptions are favorable, and, if $1/n$ is close to zero, the MIP surface will be more heterogeneous. On the contrary, if $1/n$ is close to 1, the surface will be more homogenous and the absorption tends to be irreversible [49,50].

The adjusted R^2 (adj. R^2) was the statistical parameter used to determine which model better described the MIPs and NIPs adsorption behavior. The best fit is when the adj. R^2 is equal to or closer to 1 [51].

Data fitting was performed with the OriginPro 2021 software.

2.9. Adsorption Kinetics

A kinetic assay was performed to study the change in the pollutant adsorption through time.

Equations (8) and (9) show the pseudo-first-order (PFO) and pseudo-second-order (PSO) non-linear models used in the kinetic assays [52].

PFO:

$$q_t = q_e \left(1 - e^{-k_1 t}\right) \quad (8)$$

where q_e (mg g^{-1}) and q_t (mg g^{-1}) are the amounts of adsorbate uptake per mass of adsorbent at equilibrium and in a given time t (min), respectively, and k_1 (min^{-1}) is the constant rate.

PSO:

$$q_t = \frac{q_e^2 K_2 t}{q_e K_2 t + 1} \quad (9)$$

where q_e (mg g^{-1}) and q_t (mg g^{-1}) are the adsorbate amount adsorbed at equilibrium and at any time t (min), respectively, and K_2 ($\text{g/mg} \cdot \text{min}$) is the PSO equation constant rate.

A non-linear regression was used to calculate the apparent adsorption rate (K) in both models.

2.10. Photodegradation Test

Twenty mg of each synthesized polymer and an NPX solution (5 mg/L) were placed in a 50 mL quartz tube. In addition, a test was performed by adding 0.1 g of TiO_2 under the same conditions [53]. An adsorption time of 30 min was considered for all materials, and a sample of 2 mL was taken at 0, 2.5, 5, 10, 20, 30, and 45 min. The readings were obtained using a photoreactor with a 15 W UV lamp (200 to 325 nm).

2.11. Determination and Photodegradation of Naproxen in a Real Sample

The retention and degradation capacity of the MIPs was determined using a water sample collected in the Queretaro River, Mexico ($20^\circ 35' 45.6''$ N $100^\circ 24' 46.8''$ W) on 3 April 2024, at 20:49 h. The sample was preserved at 4°C until further analysis. The retention test was carried out using manifold equipment, provided with 50 mL columns packed with 20 mg of MIP. Fifty mL of the sample was placed in the column for one hour to induce contact between the MIP/25% TiO_2 and the NPX molecules. Then, an elution with 2 mL of methanol was performed, letting it rest for 15 min. This process was repeated once more before filtering the solution with $0.45 \mu\text{m}$ PVDF filters and placing it in HPLC vials for further analysis. The degradation assay was performed according to Section 2.10.

3. Results and Discussion

3.1. FT-IR

The infrared analysis spectra of NPX-Na and TiO_2 are shown in Figure 2. The bands at 3061 and 2899 cm^{-1} in Figure 2a are due to the stretching of aliphatic C-H, while at 1632 cm^{-1} , the stretching of the C-C from the aromatic ring is observed. At 1580 and 1480 cm^{-1} , the symmetrical and asymmetrical stretching of the COO^- is found, and the two bands at 1389 and 1362 cm^{-1} belong to the CH_3 bending. The stretching of C-O from R-O- CH_3 is observed at 1252 cm^{-1} , and the band at 1203 cm^{-1} represents the C-O stretching from the carboxylate [54,55]. The Ti-O-Ti links from TiO_2 are observed from 681 cm^{-1} to 500 cm^{-1} [56].

Figure 3 shows the spectra of MIPs and NIPs. The wide band observed from 2990 to 2900 cm^{-1} indicates the vibrational movement of the C-H groups. The band at 1729 cm^{-1} corresponds to the C=O band from EGDMA [57], at 1169 cm^{-1} is located in

the C-O-C bending from the EGDMA [58]; and the band located at 1388 cm^{-1} represents the carbonyl group of the methacrylic acid [59]. The signals at 1514 and 1463 cm^{-1} are related to the COO^- asymmetric and symmetric stretching, respectively, corresponding to the complexation of the carboxylate anion from the methacrylic acid with the surface Ti centers [60].

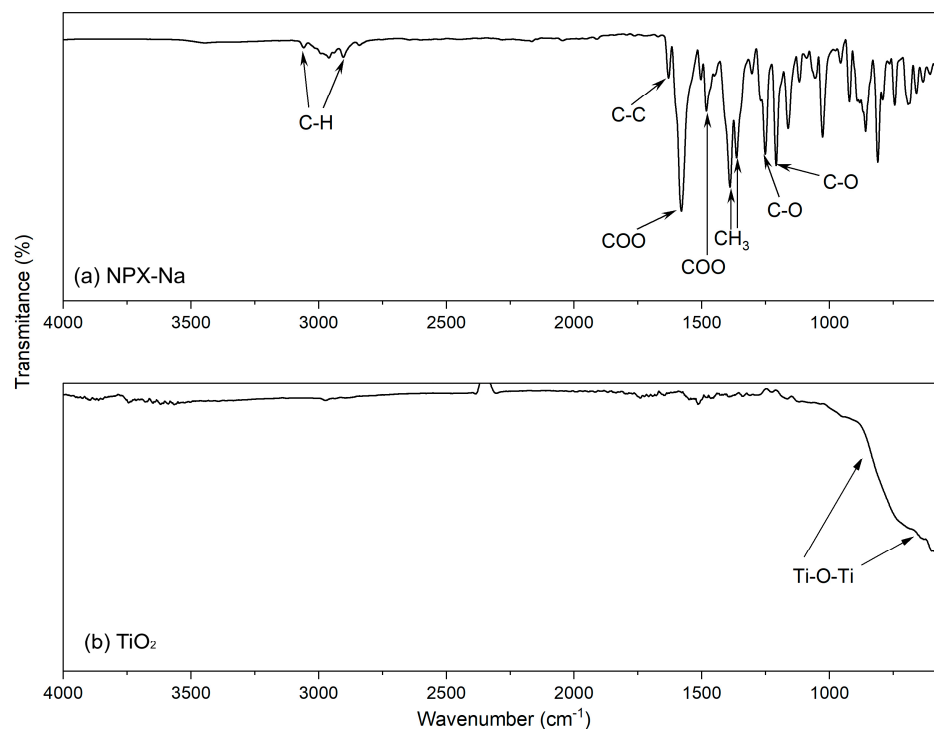


Figure 2. Infrared spectra of (a) sodium naproxen and (b) titanium dioxide.

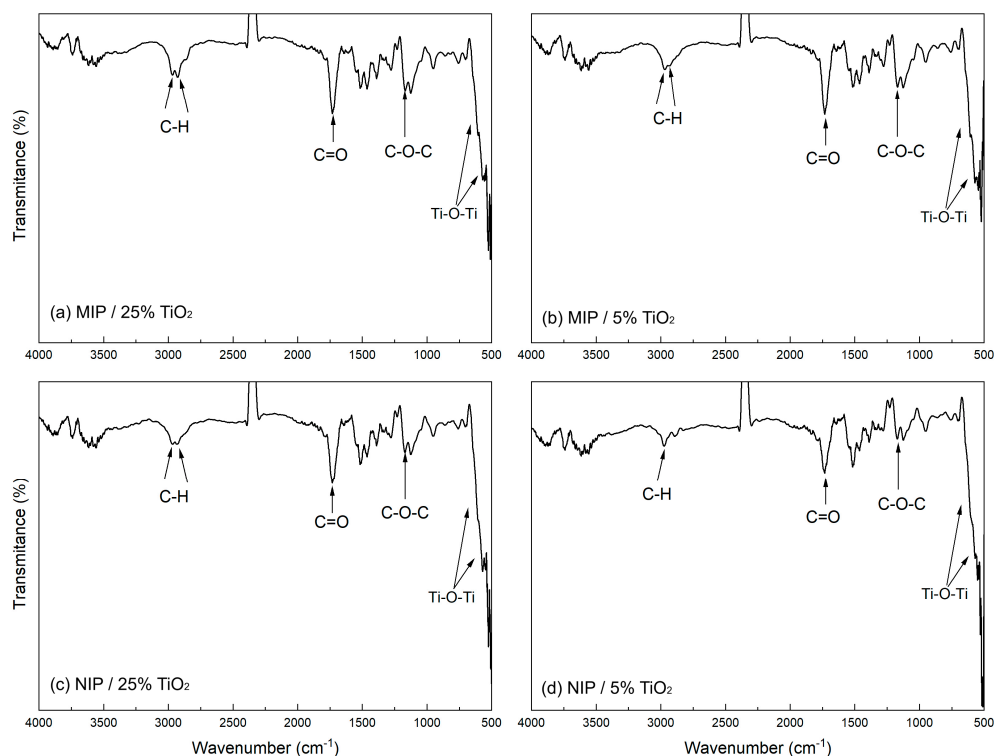


Figure 3. Infrared spectra of (a) MIP/5% TiO₂, (b) MIP/25% TiO₂, (c) NIP/5% TiO₂, and (d) NIP/25% TiO₂.

Finally, the presence of TiO_2 is observed with the broad band between 681 cm^{-1} and 500 cm^{-1} .

3.2. Point of Zero Charge

The point of zero charge (PZC) indicates the point where the total amount of positive and negative charges is the same and, hence, equals zero; above this point, the surface material is considered negative. Knowing this point helps to understand how the materials are binding through electrostatic exchanges and how to modify the environment where the materials are placed to interact to promote their union.

Figure 4 shows the surface charge distribution of MIPs and NIPs, where all materials remain positive across almost the entire pH scale and report a PZC of 7.9. Previous studies have reported NPX-Na pka values between 4.15 and 4.20, meaning that it exists in its deprotonated form and is a weak acid [54,58,61]. In this study, all experiments were performed without modifying the pH of the solution ($\text{pH} \approx 6.7$). Therefore, NPX-Na is confirmed to be in its negative form at the pH of the solution, and the MIPs surface is positively charged, causing the attraction between the NPX-Na and the MIPs. As part of this research project, other materials without TiO_2 were synthesized. The synthesis and materials were the same except for the addition of the semiconductor [45]. The PZC found for those materials was 3.18 for the MIP and 3.12 for the NIP; thus, TiO_2 increased the PZC of the materials. MIPs can behave like composites since they are a mixture of different materials. The polymer's ability to support the photocatalyst is probably due to the composite principles that apply to these materials. Therefore, combining the properties of the matrix and the reinforcement restructures the composite's properties, leading to their enhancement [62].

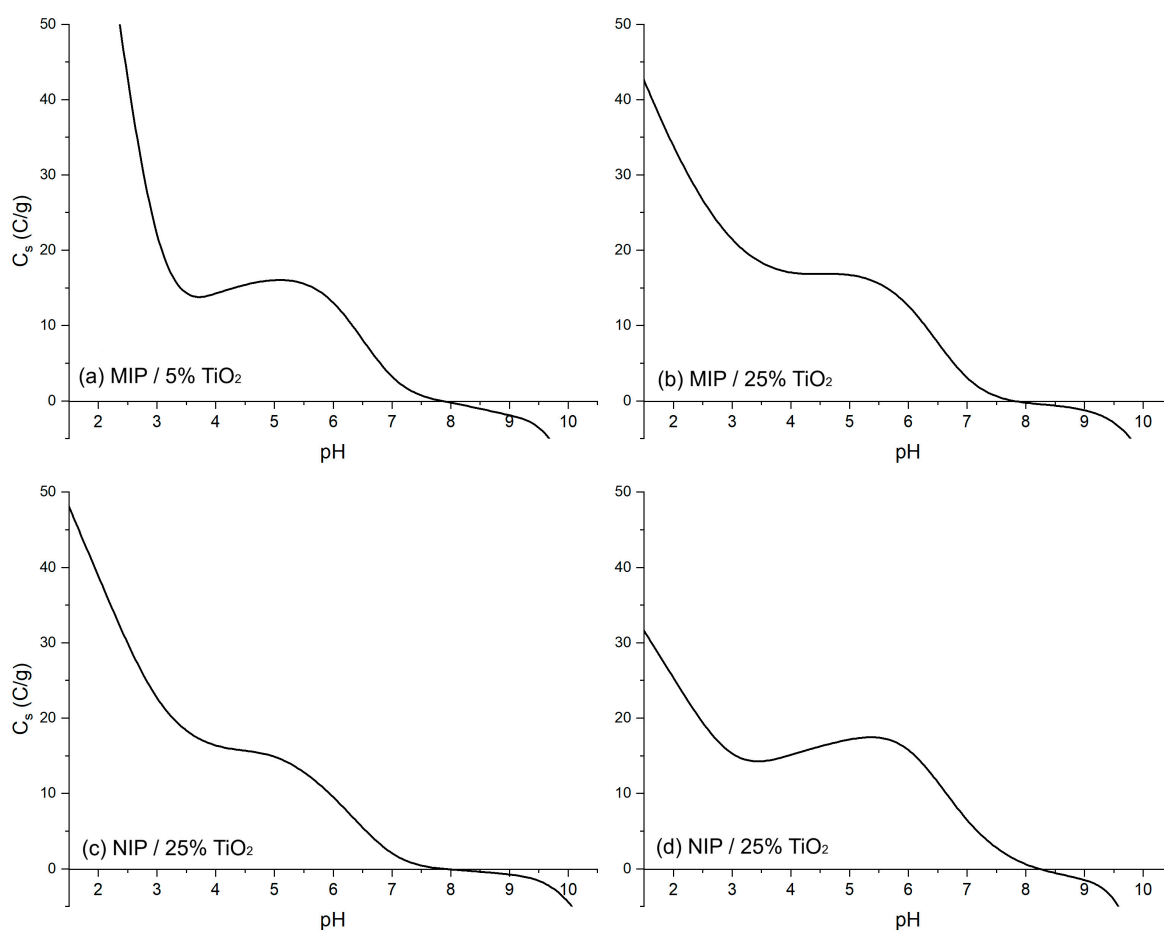


Figure 4. Zero-point charge of MIPs and NIPs.

3.3. Retention Percentage (%R), Binding Capacity (Q) and Imprinting Factor (IF)

According to Figure 5, MIPs have the highest %R. This result was expected due to the imprinting effect that generates cavities with greater affinity for NPX-Na. Significant differences are observed between the MIP with 5% TiO₂ and 25% TiO₂. The MIP with the lower semiconductor achieved the highest %R, meaning that the TiO₂ is associated with a retention percentage reduction [63,64] since there is no significant contribution from TiO₂ to the %R. Also, the semiconductor occupies a space in the polymer matrix, reducing the area available for pore formation. Therefore, it is possible to affirm that the higher the content of TiO₂, the lower the retention percentage.

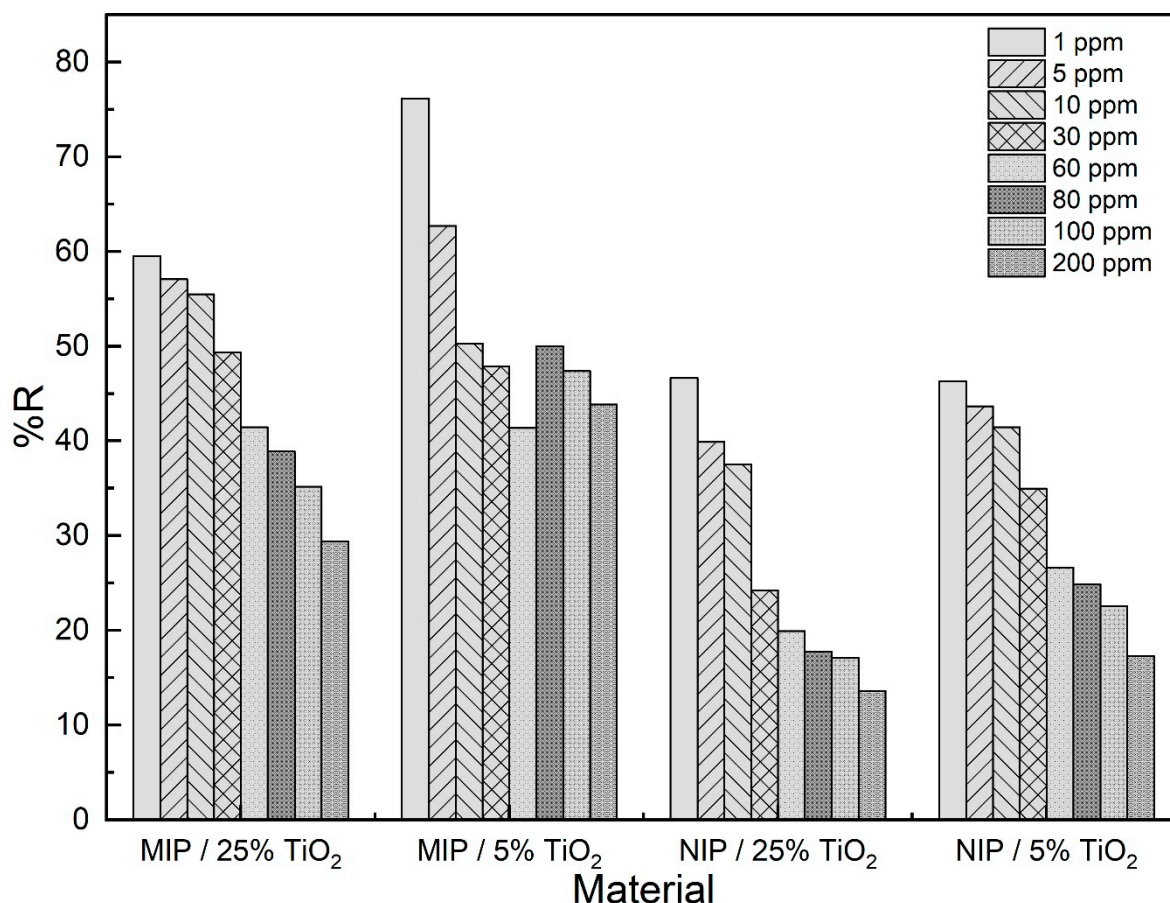


Figure 5. Retention percentages for MIPs and NIPs according to the initial concentration.

The imprinting factor (IF) values presented in Table 1, show that MIP/TiO₂ 5% exhibits higher selectivity than MIP/TiO₂ 25% at low and intermediate concentrations (1–100 mg/L), while MIP/TiO₂ 25% reaches comparable or slightly higher IF values at 30 mg/L and 200 mg/L. This suggests that the imprinting effect is more consistent in the MIP with lower TiO₂ content, although both materials present IF values above 1, indicating successful imprinting. Thus, while the MIP with 25% TiO₂ shows imprinting, the effect is less pronounced at low concentrations compared to the MIP with 5% TiO₂, which maintains a higher IF across a broader concentration range.

Although the imprinting factors (IF > 1) indicate that the MIPs have some selectivity for NPX-Na, a full evaluation of selectivity against other structurally related NSAIDs (e.g., ibuprofen, diclofenac) and against common water matrix interferents (e.g., humic acids, inorganic salts) is not provided here. Such studies are necessary to validate the molecular recognition capability of the MIPs and are recommended for future work.

Table 1. Imprinting Factor.

Material	NPX-Na Initial Concentration							
	1 mg/L	5 mg/L	10 mg/L	30 mg/L	60 mg/L	80 mg/L	100 mg/L	200 mg/L
	IF	IF	IF	IF	IF	IF	IF	IF
MIP/TiO ₂ 25%	1.737 ± 0.155	1.620 ± 0.077	1.317 ± 0.033	2.285 ± 0.080	1.675 ± 0.031	1.721 ± 0.006	1.995 ± 0.015	2.033 ± 0.005
MIP/TiO ₂ 5%	2.260 ± 0.138	1.701 ± 0.080	1.651 ± 0.055	1.176 ± 0.010	1.689 ± 0.008	2.279 ± 0.005	2.374 ± 0.005	2.300 ± 0.025

The observed fluctuations in IF values, particularly for MIP/TiO₂ 5% at 30 mg/L (IF = 1.18) and 60 mg/L (IF = 1.69), may be attributed to several factors: (i) heterogeneous distribution of binding sites [65], (ii) incomplete equilibrium at certain concentrations, or (iii) experimental variability. Although the IF values remain above 1 in most cases (indicating successful imprinting), the lack of a monotonic trend highlights the need for further optimization of the synthesis and adsorption conditions. Future work should include replicate measurements and statistical analysis to assess the significance of these variations.

3.4. Adsorption Experiments

Isotherms reveal information about the equilibrium behavior of an adsorbent toward an adsorbent. The data that constructs these graphs can be introduced into established models to obtain more evidence about the materials involved in adsorption. The models of Langmuir and Freundlich have two parameters that imply chemisorption and physisorption, respectively. These models were applied to the adsorption data obtained (Figure 6), and the results of the main parameters are shown in Table 2.

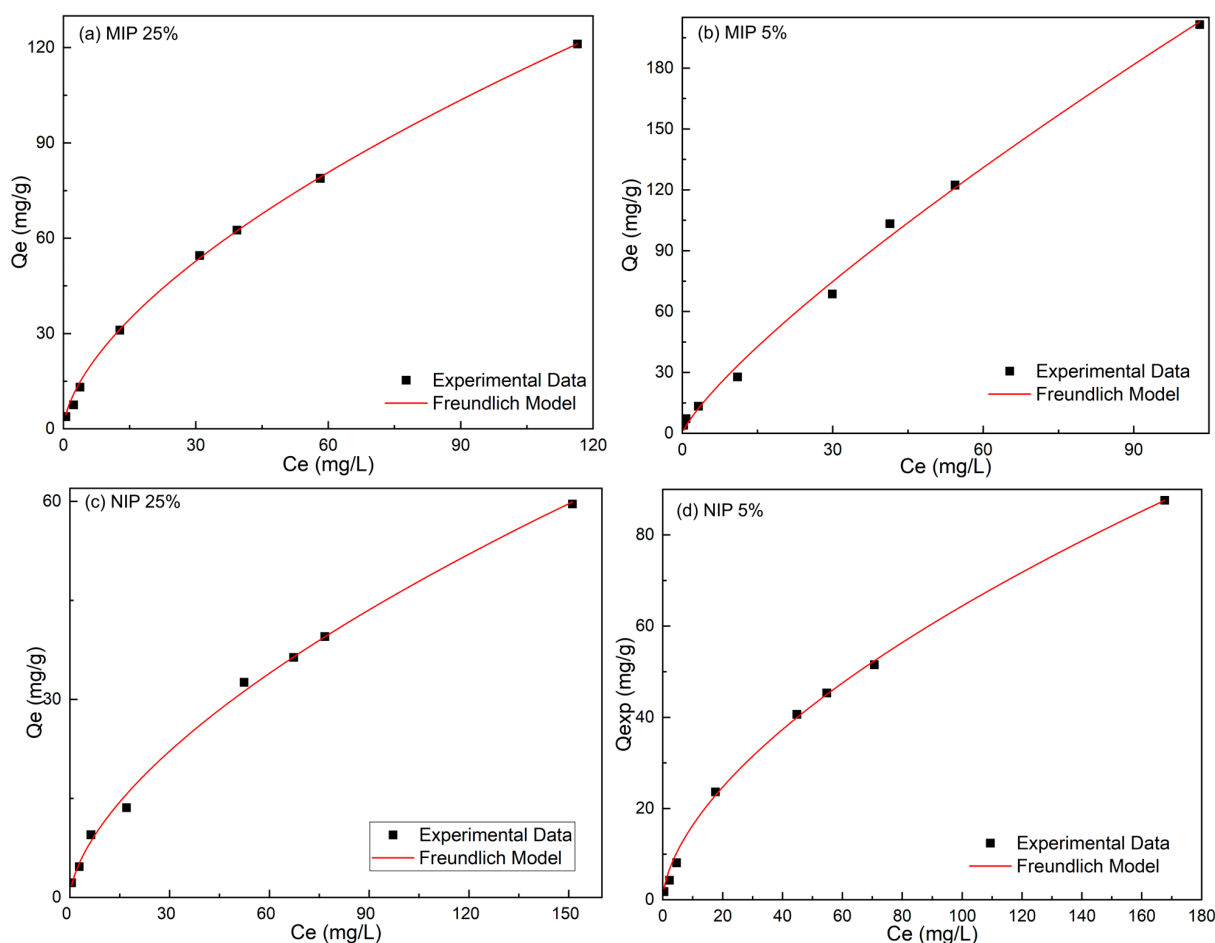


Figure 6. Fitting of Freundlich model for (a) MIP/25% TiO₂, (b) MIP/5% TiO₂, (c) NIP 25% TiO₂ and (d) NIP/5% TiO₂.

Table 2. Calculated parameters for Langmuir and Freundlich models.

Material	$q_{\max}$ (mg g ^{−1})	Langmuir		Freundlich		
		K_L (L/mg)	Adj. R ²	K_F (mg/g)(L/mg) ^{1/n}	1/n	Adj. R ²
MIP/TiO ₂ 25%	46.52	8.64×10^{-3}	0.396	6.542	0.613	0.994
MIP/TiO ₂ 5%	68.56	1.17×10^{-3}	0.738	4.808	0.807	0.999
NIP/TiO ₂ 25%	24.76	7.20×10^{-4}	0.844	2.736	0.615	0.999
NIP/TiO ₂ 5%	32.88	3.24×10^{-6}	0.748	4.163	0.594	0.999

According to Figure 6 and Table 2 (R²), the Freundlich model provides the best empirical fit for all materials, which is consistent with adsorption on a heterogeneous surface. However, model fitting alone does not allow a definitive assignment of the adsorption mechanism. It also demonstrates that the NPX is arranged in layers as it accumulates on the surface of the MIP and that the MIP surface is heterogeneous; hence, the materials have different sites with different types of energy or affinity.

K_F for MIP/25% TiO₂ and MIP/5% TiO₂ is 6.542 (mg/g)(L/mg)^{1/n}, and 4.0808 (mg/g)(L/mg)^{1/n}, respectively. The parameter K_F for the adsorption capacity is higher for MIPs than NIPs, meaning that MIPs have a higher adsorption capacity; hence, they absorb more NPX-Na in their pores. According to Ndunda [47] the lack of the target during polymerization in NIPs causes the functional groups destined to interact with the target to interact with each other during the polymerization, causing the self-association of the functional monomer. The union of the target molecule and the monomer during polymerization creates a different structure than the NIP, with more specific sites for the target. Ndunda also explains that there are three binding types: (1) specific adsorption, related to the formation of specific sites in which the target is bound through hydrogen bonding and electrostatic interactions; (2) semi-specific adsorption is related to the interaction of the target with molecules of the functional monomer that are bound to the surface of the matrix and that do not properly form the polymeric network; that is, they are anchored to the surface and with part of their structure outside the matrix, and (3) nonspecific adsorption, which is a less energetic interaction like Van der Waals or hydrophobic links that occur with the functional monomer or the crosslinker. NIPs can only undergo semi-specific and nonspecific interactions; therefore, MIPs have a higher adsorption capacity.

The K_F values for MIP/25% TiO₂ (6.542 (mg/g)(L/mg)^{1/n}) and MIP/5% TiO₂ (4.0808 (mg/g)(L/mg)^{1/n}) suggest that the material with higher TiO₂ content exhibits a larger adsorption capacity according to the Freundlich model. However, these values are derived from nonlinear regression and should be interpreted qualitatively, as a formal statistical comparison (e.g., *t*-test) was not performed.

The parameter 1/*n* describes the heterogeneity of the MIP surface; as this parameter approaches 1, a more homogeneous material is obtained. On the contrary, when 1/*n* approaches zero, a more heterogeneous material is obtained. MIP/25% TiO₂ got a 1/*n* = 0.613, and 1/*n* for MIP/5% TiO₂ was 0.807; this result means that the content of TiO₂ in the matrix alters the adsorption sites of the MIP, and with a higher TiO₂ content, the MIP becomes more heterogeneous.

The 1/*n* parameter for MIP/25% TiO₂ (0.613) is lower than that for MIP/5% TiO₂ (0.807). This indicates that the material with higher TiO₂ content has a more heterogeneous surface, as TiO₂ nanoparticles embedded in the polymer matrix create additional binding sites with different affinities. Both 1/*n* values are below 1, confirming favorable adsorption in all cases.

A similar behavior was found by Huang et al.; they synthesized a MIP with different amounts of TiO₂ (5, 10, 20, 30, and 40 mg, in this investigation 17 and 85 mg of TiO₂) for specific adsorption of kaempferol using 4-vinylpyridine as a functional monomer and EGDMA as a crosslinker. In the research work conducted by Huang et al., increases as

the amount of semiconductor rises, and the heterogeneity in the MIP also grows with the increase in TiO_2 [66].

While the Freundlich model provides an excellent description of the adsorption behaviour ($\text{adj. } R^2 > 0.994$), the lack of confidence intervals and residual analysis is a limitation of this study. Future work should include a more detailed statistical evaluation, such as the calculation of 95% confidence intervals for the KF and $1/n$ parameters, to further validate the model.

We acknowledge that the present study did not systematically examine the effects of pH, ionic strength, or the presence of competing organic species on the adsorption performance. These factors are likely to influence the electrostatic interactions between NPX-Na and the MIP surface (as suggested by the PZC results) and should be addressed in future work.

3.5. Adsorption Kinetics

The adsorption kinetics describe the uptake rate of the adsorbate onto the MIP. Figure 7 shows that the pseudo-second-order (PSO) kinetic model fits all materials. This model assumes that the rate-limiting step is the chemisorption of the analyte to the adsorbent and that the adsorption rate depends on the adsorption capacity and is independent of the initial concentration. In the PSO model, the adsorption capacity refers to the total empty sites available for the analyte to be bound; in adsorption processes, these sites decrease exponentially throughout time [67]. The PSO model also implies a material with abundant active sites, and the adsorption of the adsorbent onto these active sites drives and controls the adsorption kinetics [68].

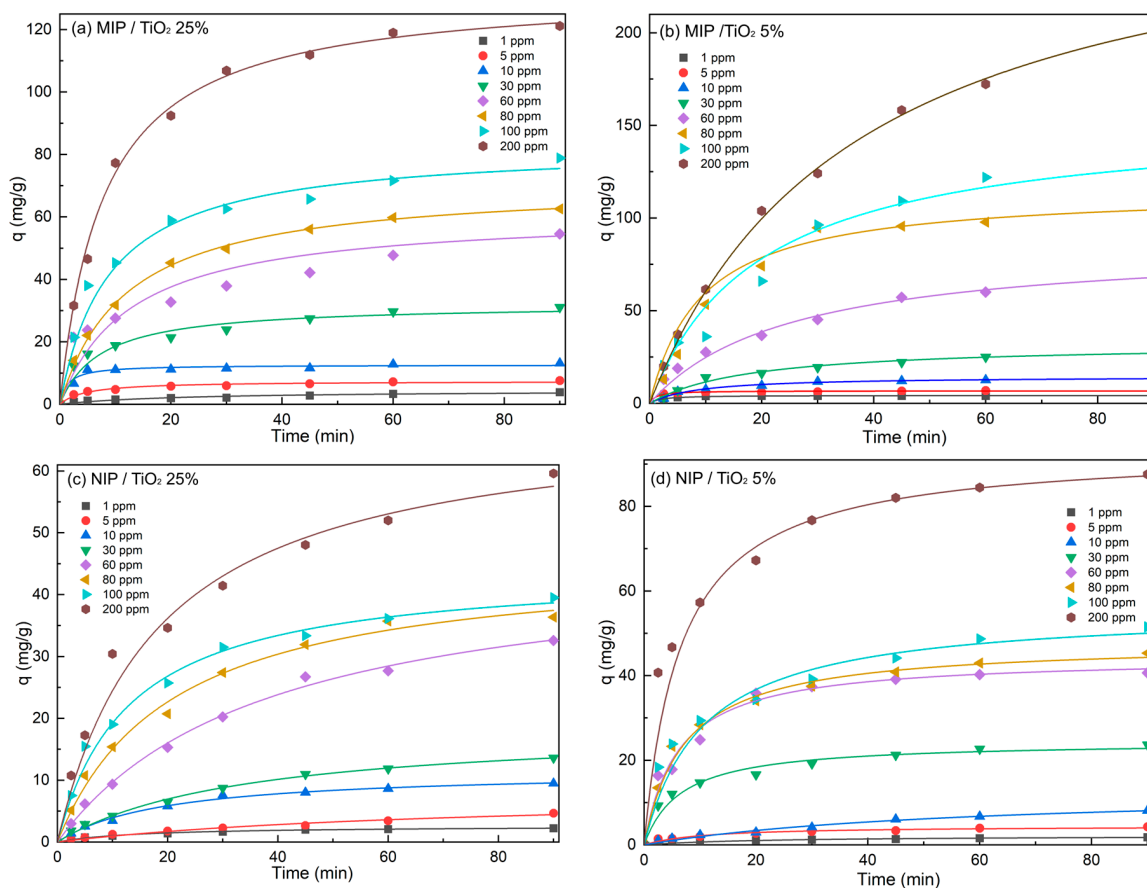


Figure 7. (a) MIP/25% TiO_2 , (b) MIP/5% TiO_2 , (c) NIP/25% TiO_2 , and (d) NIP/5% TiO_2 pseudo second order adsorption kinetic results (initial pH 6, 25 °C, $V = 50$ mL, MIP = 10 mg or 20 mg) 10 mg for 1 mg/L of NPX-Na solution.

In Figure 7, it is observed that the experimental data of the MIPs fit the PSO model better than those of the NIPs because a higher number of points fall within the curve. According to Wang and Guo [68], the PSO model better fits the modified materials to promote a better linkage between the adsorbent and the adsorbate; that is, the formation of active sites has been promoted. Therefore, the imprinting process generates more active sites in MIPs. Although the pseudo-second-order model is often associated with chemisorption, it can also adequately describe physisorption processes when the adsorption rate is limited by the number of available binding sites and by competition between adsorbate molecules, especially on heterogeneous surfaces such as those of MIPs. The Freundlich isotherm (Section 3.4) confirms that the equilibrium is governed by multilayer physisorption on a heterogeneous surface, and the PSO kinetic model simply reflects the rate behaviour under these conditions.

Although the adsorption time of 90 min was sufficient to reach apparent equilibrium for concentrations ≤ 100 mg/L (as judged from the kinetic profiles in Figure 7), for the highest concentration (200 mg/L) the adsorption may not have reached complete equilibrium. Therefore, the IF values at 200 mg/L should be interpreted with caution, and future work should employ longer contact times for high-concentration assays.

Table 3 shows the kinetic parameter results for all materials, demonstrating that the imprinting effect leads to a higher amount of NPX-Na bound to the MIPs than NIPs. According to the results, the parameter q for MIPs is always higher; however, the content of TiO₂ interferes with q at concentrations above 60 mg/L since this parameter remains similar at low concentrations (between 1 and 30 mg/L). However, from 60 mg/L q for MIP/5% TiO₂ is considerably higher than MIP/25% TiO₂. The effect that TiO₂ has on q is also evident in NIPs, except that the q values diverge starting at 30 mg/L.

Table 3. MIPs and NIPs pseudo second order kinetic parameters.

MIP/TiO ₂ 25%					MIP/TiO ₂ 5%			
C ₀ (mg/L)	q _{exp} (mg g ⁻¹)	q _{cal} (mg g ⁻¹)	k ₂ (g mg ⁻¹ min ⁻¹)	R ²	q _{exp} (mg g ⁻¹)	q _{cal} (mg g ⁻¹)	k ₂ (g mg ⁻¹ min ⁻¹)	R ²
1	3.867	3.595	0.0098	0.9999	4.089	4.201	0.1196	0.99996
5	7.569	6.603	0.1628	0.9996	7.287	6.681	0.1377	0.99986
10	12.765	11.470	0.0065	0.9996	13.418	13.265	0.0079	0.99976
30	31.086	25.293	0.0043	0.9976	27.836	26.955	0.00171	0.99906
60	54.578	54.284	1.36×10^{-3}	0.9921	68.718	67.920	4.60×10^{-4}	0.99841
80	62.588	63.212	8.02×10^{-4}	0.9980	103.338	104.387	9.39×10^{-4}	0.99693
100	78.868	75.493	1.36×10^{-3}	0.9971	122.339	127.099	3.29×10^{-4}	0.99588
200	121.172	122.277	9.34×10^{-4}	0.9993	201.505	200.712	9.64×10^{-5}	0.99977

NIP/TiO ₂ 25%					NIP/TiO ₂ 5%			
C ₀ (mg/L)	q _{exp} (mg g ⁻¹)	q _{cal} (mg g ⁻¹)	k ₂ (g mg ⁻¹ min ⁻¹)	R ²	q _{exp} (mg g ⁻¹)	q _{cal} (mg g ⁻¹)	k ₂ (g mg ⁻¹ min ⁻¹)	R ²
1	2.227	2.228	0.0191	0.9999	1.809	1.756	0.0201	1.0000
5	4.672	4.425	0.0016	0.9999	4.283	4.004	0.0207	0.9999
10	9.491	9.540	0.0044	0.9999	8.125	8.076	0.0013	0.9999
30	13.605	13.583	0.0016	0.9999	23.667	22.788	0.0062	0.9989
60	32.592	32.675	3.79×10^{-4}	0.9974	40.679	41.589	3.72×10^{-3}	0.9989
80	36.365	37.427	1.05×10^{-3}	0.9991	45.346	44.311	2.77×10^{-4}	0.9996
100	39.528	38.668	1.74×10^{-3}	0.9993	51.541	50.024	1.73×10^{-3}	0.9969
200	59.616	57.533	7.91×10^{-4}	0.9976	87.616	87.219	4.296×10^{-4}	0.9999

Although the PSO model provides a good overall fit ($R^2 > 0.99$ for most concentrations), some deviations between experimental and calculated q values are observed, particularly at intermediate concentrations. These deviations reflect the inherent heterogeneity of the MIP surface and the limitations of the simple PSO model to describe all concentration ranges with the same rate constant. Therefore, the kinetic parameters should be interpreted as semi-quantitative estimates, and future work should consider more sophisticated models (e.g., mixed-order or diffusion-based models) to better capture the adsorption dynamics.

Previous research synthesized MIPs without TiO_2 , reporting a kinetic assay with 20 mg of MIP or NIP (same as in the present investigation) with naproxen (10 mg/L) under acidic conditions [45].

Table 4 compares previous works with our investigation for similar naproxen concentrations (10 mg/L). Results show that q is higher for MIPs with TiO_2 , but equilibrium is reached slower according to k_2 .

Table 4. Comparative of results found in this investigation and our previous work.

Material	MIP-NPX	MIP/5% TiO_2	MIP/25% TiO_2
q_{exp} (mg g ⁻¹)	0.481	13.418	12.765
k_2 (g mg ⁻¹ min ⁻¹)	0.0847	0.0079	0.0065

Compounds with carboxylic groups are often used as compatibilizers to link TiO_2 with other substances, like dyes, because these functional groups have strong and specific interactions with TiO_2 in aqueous solution [69]. In this investigation, the formation of complexes between methacrylic acid and TiO_2 was demonstrated in Section 3.1 using FTIR analysis. This complex formation results in crosslinking between methacrylic acid and TiO_2 , even when the methacrylic acid is in polymer form [70–72]. Therefore, the significant difference between the present results and those obtained in our previous work lies in the increased crosslinking and, consequently, the number of pores with active sites in the MIP.

The observed decrease in k_2 with increasing initial NPX-Na concentration is physically explained by the progressive saturation of binding sites. At low concentrations, adsorption occurs mainly on high-affinity sites, resulting in a high k_2 . As the concentration increases, these sites become occupied, and the adsorption becomes limited by competition among NPX-Na molecules and by intraparticle diffusion, leading to a lower apparent k_2 . This behaviour is typical for heterogeneous adsorbents such as MIPs and is fully consistent with the Freundlich isotherm model, as discussed by Plazinski et al. [73], who related the pseudo-second-order model to intraparticle diffusion.

3.6. Photocatalytic Experiments

Photodegradation tests were performed to verify the photocatalytic activity of all materials (Figure 8). The initial concentration was determined considering a specific adsorption period to avoid errors due to possible adsorption at the beginning of the reaction and to prove that the reaction occurs on the catalyst surfaces when the contaminants are adsorbed [74].

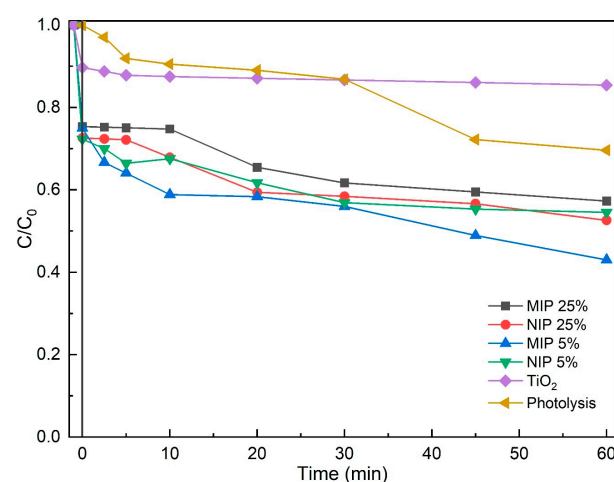


Figure 8. Photodegradation of NPX-Na (NPX-Na = 5 mg/L, initial pH 6, V = 50 mL, MIP and NIP = 20 mg, TiO_2 = 0.1 gL⁻¹, λ_{max} = 325 nm).

The uptake after the adsorption time for MIPs was 0.75 and for NIPs 0.72. This behavior was expected since, in Section 3.5, q is similar for both MIPs at low concentrations. In addition, for TiO_2 , C/C_0 is 0.90, showing lower NPX adsorption under these conditions. According to Rosal et al. [63], the equilibrium time for NPX adsorption onto TiO_2 is quite long, requiring 1 h to reach a $C/C_0 = 0.85$ and up to 25 h to reach a C/C_0 of 0.65. On the other hand, thirty minutes of UV light exposure was selected for the photolysis experiment, although a minimum time exposure was not required. For the MIP/25% TiO_2 , the NPX concentration remains constant for the first 10 min; afterward, the concentration diminishes slowly, reaching 0.57. Similar behavior was observed with NIP/25% TiO_2 , except that the drug concentration was constant for the first 5 min, slowing down to 0.53 after 60 min. MIP/5% TiO_2 reached the highest degradation since $C/C_0 = 0.43$. In this material, the concentration decreased from the first minutes, but from 10 to 30 min, the concentration remained constant, and after 30 min, the concentration decreased again. For NIP/5% TiO_2 , the fraction of NPX-Na remains constant after 30 min, reaching 0.54. NPX-Na degradation by TiO_2 is slower, starting at 0.90 and ending at 0.85. Finally, photolysis reaches 0.70 after 60 min; in this case, the reaction remains constant between 10 and 30 min, as in the c experiment.

Similar results were found by Méndez-Arriaga et al. [75] regarding the results of photolysis and degradation by TiO_2 ; they found that despite the high degradation achieved by photolysis, mineralization is low, whereas with TiO_2 , higher percentages of mineralization are achieved but with lower degradation percentages. However, the toxicity of NPX in N aqueous medium does not diminish, since the byproducts are even more difficult to degrade.

As reported by Méndez-Arriaga et al. [75], there is a competition between NPX and TiO_2 for the absorption of UV light. A full mechanistic analysis of this competition (e.g., quantum yield calculations or kinetic modelling) is beyond the scope of the present work, which focuses on the overall performance of the MIPs. Nevertheless, our results clearly show that the MIPs with embedded TiO_2 achieve faster degradation than TiO_2 alone, confirming the beneficial effect of the MIP matrix.

Naproxen mineralization appears complicated, since the byproducts are even more toxic than NPX itself [32]; different methods have been tested, for example, a combination of UV radiation and chlorine [76], UV radiation and H_2O_2 [77], sonolysis and Fenton reagent, sonocatalysis [78], UV/ O_3 /peroxymonosulfate [79], among others.

Commonly, the problem is associated with UV radiation and its effect on the degradation products of NPX. A possible solution could be the degradation in the visible light region, as in the research by Schindra et al. [80], where they found that the photocatalyst $\text{AgBr}-\alpha\text{-NiMoO}_4$ reached total NPX mineralization.

A limitation of this study is the absence of identification of NPX photodegradation intermediates, which would require LC-MS or similar techniques. Consequently, the toxicity of the degradation products was not assessed. Future work should address these aspects to confirm the environmental safety of the treated water.

Another limitation of this study is the absence of Total Organic Carbon (TOC) measurements, which would be necessary to quantify the degree of NPX mineralisation. Our degradation data (UV-Vis) indicate the disappearance of the parent NPX molecule, but the formation of organic byproducts cannot be ruled out. Future studies should include TOC analysis and longer irradiation times to evaluate the extent of mineralisation.

On the other hand, there is a lack of information regarding MIPs and degradation (MIPs with a polymeric matrix and not materials that use molecular imprinting). Most reviews and research papers regarding MIPs and environmental issues are focused on solid-phase extraction and adsorption [81–83].

We acknowledge that the present data do not allow a complete separation of the contributions of selective adsorption, direct photolysis, and genuine photocatalysis. Future

work should include quenching experiments to identify reactive oxygen species, TOC analysis to quantify mineralisation, and LC-MS to identify transformation products. Such experiments would provide a more mechanistic understanding of the degradation process.

3.7. Determination and Photodegradation of Sodium Naproxen in a Real Sample

The Querétaro River flows through the capital city of Santiago de Querétaro and two neighboring cities: Corregidora and El Marqués. According to the Mexican National Water Commission (CONAGUA) and the latest National Inventory of Municipal Water Treatment and Wastewater Treatment Plants in Operation from December 2022, there are two active wastewater treatment plants (WWTP) in the area. WWTP Centro is located in Querétaro City, while WWTP Chichimequillas is in El Marqués. The effluents from these plants are released into the Querétaro River. The river is near industrial areas with factories, including commerce, tourism, agriculture, and livestock. As a result, multiple sources of pollution impact the river; thus, efforts are made to identify specific pollution discharge points. A study assessing water quality found that the water near these discharge points is rated as poor based on the Water Quality Index from the National Sanitation Foundation [84].

The recorded sample temperature at 20:40 on the sampling day was 19 °C. The river flow was low, and the sample exhibited turbidity and a dark-brown color caused by suspended particles. The sampling site presented an unpleasant odour, which persisted in the sample. In the laboratory, the pH of the sample was 7.76. Subsequently, the sample was filtered using a Buchner funnel and Grade 2 filter paper with a diameter of 125 mm. The retention test was carried out according to Section 2.11 using manifold equipment. The photodegradation reaction was carried out according to Section 2.10; finally, the MIP/5% TiO₂ was selected for the retention assay and the degradation experiment.

The MIP with 5% TiO₂ was selected for the real-sample analysis because it displayed the highest retention percentages and more consistent imprinting factors in the synthetic water experiments. Table 5 presents the results of the retention assay using MIP 5%/TiO₂ and the degradation results of the sample taken from the Queretaro River. According to the retention test, the concentration found using MIP 5%/TiO₂ was 0.332 mg/L, compared with the value obtained in the degradation experiment (0.245 mg/L), a difference of 0.087 mg/L is found, meaning that the MIP can preconcentrate NPX. It is crucial to highlight that the degradation test was carried out according to Section 2.10, in which a first sample was taken before the MIP was used with the real sample.

On the other hand, during the degradation experiment, the concentration found after the adsorption process was 0.217 mg/L. Then, after UV exposure, the concentration remained constant during the experiment, giving degradation percentages between 86% and 92%. The interaction between the MIP and all the molecules in the sample is expected; therefore, a deeper and broader analysis is needed to find additional contaminants that may interfere with the analysis.

Given the LOQ of 0.110 mg/L, the observed changes in NPX concentration during the photodegradation experiment are close to the quantification limit; therefore, the calculated degradation percentages are approximate and should be considered indicative rather than absolute.

This real-water test is based on a single sample and lacks matrix-effect controls, recovery tests, or replicate analyses. Therefore, the results should be considered as preliminary. Future work should include spike-recovery experiments, replicate field samples, matrix-matched controls, and uncertainty analysis to properly validate the environmental application of these MIPs.

Table 5. Detection and photodegradation of DCF found in real sample.

Assay	Time (min)	Conc. (mg/L) *	% Degradation
Retention	-----	0.332	-----
Photodegradation	C ₀	0.245	-----
	0	0.217	88.26
	2.5	0.213	86.96
	5	0.210	85.61
	10	0.212	86.27
	20	0.215	87.61
	30	0.226	92.19
	45	0.222	90.32
	60	0.217	88.45

* LOD 0.018 mg/L and LOQ = 0.110 mg/L.

Also, NPX is a recalcitrant contaminant, meaning that photolysis byproducts are more difficult to degrade and more toxic [32]; therefore, efforts are paramount to find effective processes to remove NPX from the environment. Our results demonstrate the feasibility of MIPs combined with other materials or processes to achieve water remediation. The MIPs developed can be used for the preconcentration and identification of NPX and as a co-adjuvant in the degradation process of less contaminated samples.

4. Conclusions

This study demonstrated that molecularly imprinted polymers modified with 5% TiO₂ exhibit high affinity and adsorption capacity for naproxen sodium (NPX-Na), attributed to the formation of specific binding sites during polymerization. The Freundlich isotherm model provided an excellent fit (adj. $R^2 > 0.994$), confirming the heterogeneous nature of the MIP surface. Kinetic studies followed the pseudo-second-order model ($R^2 > 0.99$), indicating a high availability of active sites. The MIP with 5% TiO₂ showed the highest retention percentages (e.g., >90% at low concentrations) and a more consistent imprinting factor (IF up to 2.37) compared to the 25% TiO₂ material. Photodegradation experiments confirmed that both MIPs exhibit photocatalytic activity derived from TiO₂, although NPX degradation remained limited (maximum 60% in synthetic water), consistent with NPX's known recalcitrance and the potential persistence of toxic photoproducts. In real water samples from the Querétaro River, the MIP/TiO₂ 5% effectively preconcentrated NPX-Na (from 0.245 to 0.332 mg/L) and enabled its detection at environmentally relevant levels.

However, degradation performance in the real matrix was constrained, likely due to competing species and UV light attenuation. Overall, TiO₂-modified MIPs show promise as multifunctional materials capable of both selective extraction and partial photocatalytic transformation of pharmaceutical pollutants. Their primary value lies in preconcentration and analytical enrichment, with photocatalysis serving as a complementary—not standalone—treatment mechanism. Given the incomplete degradation of NPX-Na and its recalcitrant byproducts, future work should integrate TiO₂-MIPs with other advanced oxidation processes or visible-light-driven photocatalysts to achieve more efficient and safer remediation. Additionally, imprinting using naproxen itself, or dual-template strategies, may enhance specificity and improve adsorption.

While the present study confirms the successful imprinting through adsorption isotherms, kinetics and imprinting factor calculations, we recognise that direct visual evidence of the imprinted cavities (e.g., by TEM) was not obtained. Future work should include TEM characterisation of the MIPs before and after template extraction to further validate the formation of specific recognition sites.

Author Contributions: All persons who meet authorship criteria are listed as authors, and all authors certify that they have participated sufficiently in the work to take public responsibility for the content, including participation in the concept, design, analysis, writing, or revision of the manuscript. Conceptualization, Analytical methods, Writing—Reviewing, and Manuscript Editing D.S.V.-L.; Analytical Methods, Reviewing and Manuscript Editing by V.S.G.-R., C.L.-S. and B.V.L.-G.; Reviewing and Manuscript Editing by K.X.V.-B., R.O.-P. and H.H.-M.; Conceptualization and Funding by R.F.-R. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Data available on request from the authors.

Acknowledgments: The authors also declare that during the preparation of this work the authors used Grammarly (<https://www.grammarly.com/>) in order to check the grammar of the paper. After using this tool/service, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication.

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