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Degradation of Chrysene PAHs by Photocatalytic Remediation in Industrial Wastewaters

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ABSTRACT

Chrysene is a polycyclic aromatic hydrocarbon composed of four benzene rings and has the chemical formula $C_{18}H_{12}$, It occurs in the form of colorless, prismatic crystals, and is suspected to be a human carcinogen, and is known to cause cancer in laboratory animals. Because of the danger of this compound and the possibility of it being carcinogenic, this research aims to treat many treated and untreated waters throw years 2021 and 2022 (5 Farms, Main treatment plant, Tanning factory treated wastewater, Tanning factory non-treated wastewater, Carton factories, Factories Lake, and Grease refining plants) and remediate the Chrysene compound by using the photocatalytic techniques under the influence of UV rays using two wavelengths, 254 and 306 nm in addition to the use of activating catalysts $ZnO/TiO_2/H_2O_2$ (1:1:1) which would speed up the treatment of this compound in the tested waters. Water samples were extracted by QuEChERS methodology and analyzed by GCMSMS/TQD by GCMSMS/TQD using a catalyst for 10 hours. The effect of multiple curing times of photosensitizers containing Chrysene was also studied. The results obtained in this research indicate that the Chrysene detection limit was 5 µg/L, and the recovery was between 96.56 - 99.11%. The impact of photocatalysis on the Chrysene compound disappeared after 3 and 4 hours of treatment using 306 µm after UV radiation with a $ZnO/TiO2/H_2O_2$ catalysis., generally appropriate. the results of the photo treatment were effective and sufficient to break down the Chrysene compound. Conductive electronics, which react with the oxygen adsorbed on the surface to give superoxide radical anions, and finally, produce water starting from HO • this dose oxidizes the target molecule with Chrysene to anthraquinone. Thus, this remediation method can be used to treat contaminated water with Chrysene, which can maintain water sustainability and its variability of reuses.

Keywords: Photocatalysis, Remediation, Chrysene, PAHs, Wastewater, ZnO, TiO2 and H2O2.

INTRODUCTION

Chrysene, a polycyclic aromatic hydrocarbon (PAH), poses significant health risks, particularly regarding respiratory diseases and its carcinogenic effects. Research indicates that Chrysene exacerbates chronic obstructive pulmonary disease (COPD) by promoting inflammation and apoptosis in lung tissues, especially when combined with cigarette smoke exposure. Furthermore, Chrysene accelerates COPD progression, leading to increased collagen deposition and inflammation in the lung tissues. [1] (Gao et al., 2021). Additionally, Chrysene is classified as a human carcinogen, capable of damaging DNA and activating the aryl hydrocarbon receptor (AhR), which influences various cellular processes and gene expressions [2] (Anishya, 2023).

Chrysene exposure results in decreased cell viability and increased apoptosis in Müller cells, indicating its neurotoxic potential, conversely, while Chrysene's health effects are predominantly negative, some studies suggest that certain compounds, like lipoic acid, may mitigate its toxicity, indicating potential avenues for therapeutic intervention [3] (Mansoor et al., 2013).

Chrysene is metabolized into reactive forms that can harm cellular macromolecules, this receptor's activation leads to the upregulation of enzymes involved in xenobiotic metabolism, contributing to its toxic effects [4] (Alqassim et al., 2019).

Chrysene, a polycyclic aromatic hydrocarbon, can be present in wastewater due to the incomplete combustion of carbonaceous materials. It is classified as a high priority pollutant by the U.S. EPA and is considered a human carcinogen. Chrysene, a polycyclic aromatic hydrocarbon (PAH) with four benzene rings, is a significant pollutant found in wastewater, particularly from industrial sources. Its presence in wastewater poses serious environmental and health risks due to its carcinogenic properties and potential to bioaccumulate in aquatic organisms. Chrysene is primarily released into wastewater from industrial activities, notably tanning factories and carton manufacturing, with concentrations ranging from 14.05 to 123.11 µg/l in various wastewater samples. Tanning factory non-treated wastewater exhibited the highest levels of PAHs, including Chrysene [5]) Albuqmi et al, 2023).

Chrysene is classified as a high-priority pollutant by the U.S. EPA due to its carcinogenic and mutagenic effects and It can disrupt aquatic ecosystems, affecting organisms through direct toxicity and bioaccumulation in the food chain [6] (Bastolla et al., 2023). Certain fungi, such as Fusarium sp., have shown promise in degrading Chrysene, achieving up to 48% degradation in laboratory conditions. This highlights the potential for bioremediation strategies to mitigate Chrysene contamination in wastewater. While Chrysene poses significant environmental and health risks, ongoing research into its degradation by microorganisms offers a hopeful avenue for remediation. Understanding the dynamics of Chrysene in wastewater is crucial for developing effective management strategies. [7] (Hidayat & Tachibana, 2015).

The degradation of Chrysene through photocatalytic remediation is a promising approach to mitigate the environmental impact of this persistent polycyclic aromatic hydrocarbon (PAH). Photocatalysis utilizes light-activated semiconductor materials to generate reactive oxygen species, which facilitate the breakdown of organic pollutants like Chrysene into less harmful compounds. The effectiveness of this method is influenced by various factors, including the type of photocatalyst, operational conditions, and the presence of other substances. Photocatalysts such as modified TiO2 and Ag3PO4 generate reactive species like hydroxyl radicals and singlet oxygen, which are crucial for the degradation of Chrysene. Factors like pH, temperature, and ionic strength can significantly affect the degradation rate, with optimal conditions leading to improved efficiency [8-9] (Miad et al., 2024)(Vinu & Madras, 2010).

Degradation Pathways studies indicate that Chrysene undergoes complex degradation pathways, resulting in products like 1,4-Chrysenediol and phthalic acid [10] (Kong & Ferry, 2003).

Catalyst Efficiency use of composite materials, such as graphene oxide enwrapped Ag3PO4, has shown enhanced photocatalytic activity, achieving significant degradation rates under visible light [11] (Yang et al., 2017).

Chrysene, a polycyclic aromatic hydrocarbon (PAH), undergoes various pathways that significantly impact its environmental behavior and toxicity. Understanding these pathways is crucial for assessing the risks associated with Chrysene exposure and its derivatives. The following sections outline the key aspects of Chrysene pathways, including formation, biotransformation, and health implications and primarily formed through the combustion of

organic materials, such as fossil fuels and biomass, which release PAHs into the atmosphere [12 -13] (Nikitha et al., 2017) and (Kameda, 2011).

Chrysene undergoes biotransformation in living organisms, leading to the production of reactive metabolites that can bind to DNA, potentially causing mutations and cancer. The aryl hydrocarbon receptor (AhR) plays a significant role in mediating the toxic effects of Chrysene, linking environmental exposure to adverse health outcomes [14] (Peng et al., 2023)

Long-term exposure to Chrysene and its derivatives is associated with serious health risks, including respiratory issues and carcinogenic effects [15-18] (Venkatraman et al., 2024), Avani et al (2020), Myriam et al (2019), and Kazuichi et al (2023).

2. Materials and Methods

2.1. Wastewater Samples

Treated and untreated wastewater samples were collected from Eleven wastewater sources industrial treated wastewater during the years 2021 and 2022, farms (F1 to F5), main treatment plants (6), tanning factory treated wastewater (7), tanning factory non-treated wastewater (8), Carton factories (9), Factories Lake (10), and Grease refining plants (11) in 1st industrial city, Riyadh, the capital of Saudi Arabia. One liter of each sample was taken in dark glass container in ice blocks and transferred to the lab on the same day to extract the targeted PAHs using the QuEChERS Methodology.

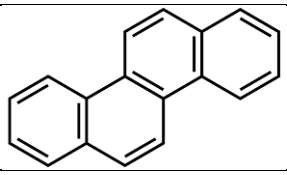
2.2. Standards and Reagents

Calibration and injection standards of **Chrysene** (Table 1) with 99.9% purity, were purchased from Accu-Standard, 153 Inc., New Haven, CT, USA as an individual (50 mg) or mixture standards at a concentration of 100 µg/ml. Internal standards are ^{13}C 12-labelled; the use of the ^{13}C-labelled compound is preferable because the analysis can be quantified without clean-up. All solvents (Methanol, dichloromethane, hexane and acetonitrile) used for the extraction and analysis of Chrysene were residue-analysis grade 99.9% purity and obtained from Fisher Scientific (Fair Lawn, NJ, USA). QuEChERS kits were purchased from Phenomenex, Madrid Avenue, Torrance, CA, USA.

2.3. Chrysene fact sheet

Table 1: Characteristics of Chrysene Compounds.

Properties	Value
CAS#	218-01-9
Formula	C18H12

Number of rings	4
Structure	
Molar mass	228.28 g/mol
Melting point	254 °C
Boiling point	448 °C
Density	1.27 g/cm³
Classification	Organic compound, polycyclic aromatic hydrocarbon
General ingredient:	Coal tar
Risks	Suspected to be a human carcinog and is known to cause cancer in laboratory animals

2.4. Photocatalysis Procedure of Chrysene

Treated or untreated water samples collected from 12 various sources were treated using a mixture of ZnO/TiO2/H2O2 catalysts in a 1-1-1 ratio to the water samples to treat the tricyclic Chrysene compound. 20 ml of the water sample was taken, then 1 ml of the catalyst mixture was added, and then exposed to UV rays using two wavelengths, 254 and 306 nanometers, for 10 hours (one hour), along with taking a sample to analyze the Chrysene compound according to the time mentioned previously. Water samples that were photo-remediated with catalysts were extracted using the QuEChERS method, and then analyzed using GC-MS/MSTQD 8000/SRM, which will be explained.

2.5. Extraction of CHRYSENE in Wastewater Sample by QuEChERS Method.

To extract the Chrysene from treated and untreated wastewater samples, 10 ml of each sample (3 replicates) was added to a 50 mL centrifuge tube and then add 10 mL of acetonitrile solvent was added to each sample. Shake (manually or mechanically) or vortex samples for 3 minutes to extract the Chrysene. Add 1 g of NaCl and 2 g of magnesium sulfate, then immediately shake samples and vortex for 2 min to complete the extraction of Chrysene and then centrifuge for 5 minutes at ≥ 3500 rcf. Transfer a 1.5 mL aliquot of supernatant to a 2 mL CUMPSC18CT (MgSO4, PSA, C18) dSPE tube and Vortex samples for 1 min.

Centrifuge for 2 min at high rcf. (e.g. ≥ 5000) . Transfer 1 ml of the aliquot of supernatant to a filter and purify the supernatant through a 0.2 µm syringe filter directly into a 1.8 ml brown GC vial to be analyzed by GC-MS/MS TQD [19-20].

2.6. Analysis of CHRYSENE by GC-MS/MSTQD 8000/SRM

All measurements have been carried out using the latest Thermo Scientific™ TSQ 8000™ triple quadrupole GC-MS/MS system equipped with the Thermo Scientific™ TRACE™ 1310 GC with SSL Instant Connect™ SSL module and Thermo Scientific™ TriPlus™ RSH autosampler. Injection mode was spiltless, Spitless Time 1.0 min GC Column DB5 MS, 30 m × 0.25 mm × 0.25 µm. Carrier gas was He99.999%, flow rate 1.2 mL/min, constant flow, temperature program 100°C, 1 min; 10°C/min to 160 °C, 4 min and 10°C/min to 250 °C, 2 min, transfer line temperature 280°C, total analysis time 31 min, Tri-Plus RSH Autosampler Injection volume 2 µL. Ionization mode EI, 70 eV, Ion source temperature 250 °C, scan mode SRM using timed SRM transition setup automatically build-up by Auto SRM software. GC-MS/MSTQD 8000 SRM Transition conditions are shown in Table 2.

Table 2. GC-MS/MSTQD 8000 / SRM Instrumental conditions of Chrysene analysis in wastewater samples.

GC Trace Ultra Conditions		**TSQ Quantum MS/MS Conditions**	
Column	DB5 30 m × 0.25 mm × 0.25 µm	Operating mode	Selected Reaction Monitoring (SRM)
Injector	Splitless	Ionization mode	EI
Injected volume	1 µL	Electron energy	70 eV
Injector temperatu	215 °C	Emission current	50 µA
Carrier gas	Helium, 1.2mL/min	Q1/Q3 resolution	0.7 u (FWHM)
Oven program	70 °C hold 1 min 15 °C/min to 160 ° hold 1 min 2.2 °C/min to 220 °C hold min 5 °C/min to 280 °C hold 5 min R Time 30.00 min	Collision gas	Argon
Transfer line temperature	275 °C	Collision gas pressure Polarity	1 mTorr Positive

2.7. Method Performance and Validation of Chrysene.

Precision and accuracy of the extraction and analysis method were conducted by 3 replicates of blank wastewater samples spiked with the labeled Chrysene standards. Limit of detection:

Instrument Detection Limit (IDL), Sample Detection Limit (SDL), Method Detection Limit, accuracy, and precision (Table 3).

Table 3. The average recovery of Chrysene and relative standard deviations RSD.

Organic solvents	Recovery%	RSD%	Detection Limit DL
Acetonitrile	97.84	2.6	5 µg/l
Acetone	96.56	2.7	5 µg/l
Dichloromethane	98.72	2.2	5 µg/l
Ethyl acetate	98.83	2.9	5 µg/l
Methanol	99.11	2.8	5 µg/l

2.8. QAQC Strategies

Quality control samples were prepared and analyzed the duplicate sample, blank, and spiked samples, and/ or Certified Reference material CRM was prepared for this purpose and processed with every 5 samples. QuEChERS and GC-MS/MS TSQ 8000 method limit of detection (LOD) and Limit of Quantification (LOQ), repeatability, reproducibility, accuracy, and precision were also determined for the CHRYSENE compound.

3. Results

Chrysene PAH isomers are among the most toxic organic pollutants from numerous industrial wastes, especially the coal processing ones, as heavy carcinogenic substances for aquaculture and the human population. Classical methods for these pollutant eliminations, such as physical, chemical, or biochemical treatments, generate some secondary negative environmental effects due to the accumulation of degradation products that are often more toxic and mutagenic than the original pollutant. In the present work, the model research on adsorption and photocatalytic degradation of the model organics from the type Chrysene PAH as components within real samples, like industrial sewage water, was performed using hybrid ZnO and TiO2 nanoscale powders. Both powders functionalized with H2O2 as lower environmentally polluted catalysts showed higher photocatalytic effectiveness under illumination, based on the decrease in recombination processes and the increase in hydroxyl radical numbers. The pollution of natural water resources by organic and persistent substances plays an important role in the heavy degradation of the ecological equilibrium of aquatic environments, with many negative effects on the aquatic ecosystem, for the human

populations that consume such sources, and for the environment since various water pollutants are usually toxic, bacterial, and mutagenic. The significant increase in the amounts of organic pollutants in the aquatic environment was also due to the very different industrial discharges without previous depolluting treatments of the industrial wastewaters, which contain several organic compounds. In the present work, some treatments to eliminate the Chrysene polynuclear aromatic hydrocarbon (PAH) isomers as very carcinogenic organic pollutants from coal industrial wastewaters were detailed, spending limited financial resources but generating a lower pollution degree for the aquatic environment.

3.1. Chrysene Concentration Different Wastewater Samples (µg/l)

The results of Chrysene in the years 2021 and 2022 wastewater samples are shown in Table 4 and Fig. 2. The results revealed that the concentration (µg/l) in tested samples in the year 2021 were 14.56, 17.22, 14.05, 16.83, 17.23, 15.77, 102.33, 123.11, 89.03, 62.05, and 89.51(µg/l) for sources 1 to 11, respectively. Meanwhile, the concentration in the year 2022 was 25.21, 23.07, 21.6, 22.25, 21.49, 19.33, 118.93, 141.22, 93.21, 77.34, and 92.33 (µg/l) for sources 1 to 11, respectively. The results of the Chrysene compound also indicate that the highest concentration was in the Tanning factory non-treated wastewater (8), followed by Tanning factory treated wastewater (7) for wastewater samples tested in 2021 and 2022.

Table 4. Total concentration (µg/l) of Chrysene in different wastewater samples.

WW Sources	Chrysene Concentration (µg/l)	
	2021	2022
Farm1	14.56	25.21
Farm2	17.22	23.07
Farm 3	14.05	21.6
Farm 4	16.83	22.25
Farm 5	17.23	21.49
Main treatment plant 6	15.77	19.33
Tanning factory treated wastewater 7	102.33	118.93
Tanning factory non-treated wastewater 8	123.11	141.22
Carton factories 9	89.03	93.21
Factories Lake 10	62.05	77.34
Grease refining plants 11	89.51	92.33

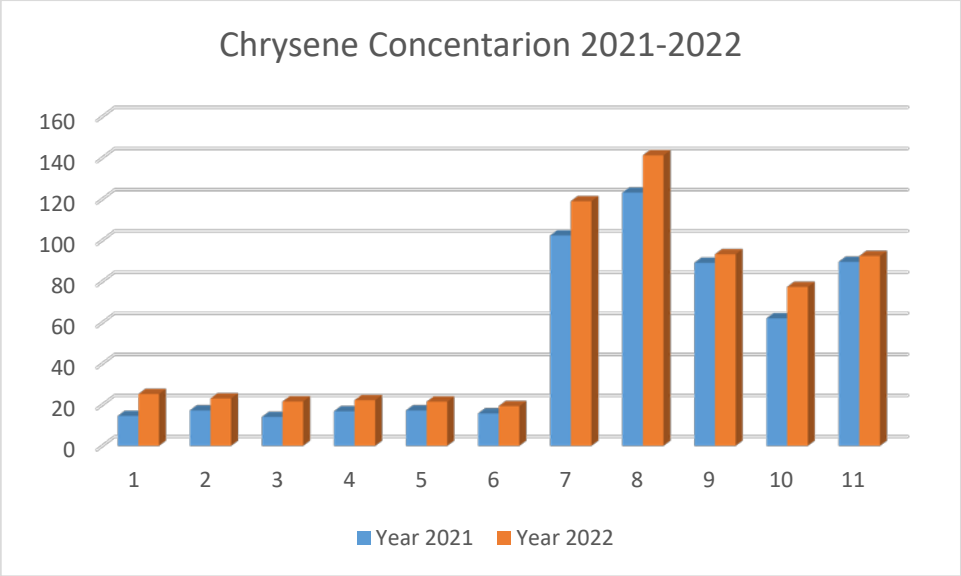


Figure 1: Concentration (µg/l) of Chrysene in different treated and untreated wastewater during the years of 2021/2022.

3.3. Effect of UV Remediation (254nm) on Chrysene with ZnO / TiO2 / H2O2 (1:1:1) in different Wastewater.

The effect of photocatalytic remediation (254nm) on CHRYSENE with ZnO + TiO2 + H2O2 (1:1:1) using 1, 3, 5, and 8h (Table 5 and Figs 2 and 3) in different tested wastewater samples. The results of this study showed that the effect of the mixture of catalysts used was very effective and positive in treating the Chrysene compound in the tested water samples, as showed in (Table 5) where it was not detected ND after 3 hours in the water samples of farms 1 to 5, and Main treatment plant 6. Its concentration reached 17.61 micrograms/liter in the Tanning factory treated wastewater 7 sample, and it was noted that the Chrysene compound was not detected at a treatment time of 8 hours for all the previously mentioned samples. The results also showed that the Chrysene compound was not destroyed after 5 hours of treatment for each of the samples, Factories Lake 10 and Grease refining plants wastewater 11, where the concentrations were 7.33 micrograms/liter, still remaining in the Tanning factory non-treated wastewater 8 at 10 hours of the photocatalyst remediation.

Table 5. Effect of UV remediation (254nm) on Chrysene (4 rings) PAH (µg/l) with ZnO + TiO2 + H2O2 catalysts.

WW Sources	Chrysene concentration (ppb) and Irradiation time (h)					
	0 Time (ppb)	1h	3h	5h	8h	10h
Farm1	14.56±1.67	8.34±0.41	4.11±0.83	ND	ND	ND
Farm2	17.22±2.86	9.26±0.21	3.17±0.54	ND	ND	ND

Farm 3	14.05±2.02	6.05±0.33	2.98±0.66	ND	ND	ND
Farm 4	16.83±1.77	6.77±0.81	3.09±0.37	ND	ND	ND
Farm 5	17.23±1.23	7.14±0.44	4.03±0.88	ND	ND	ND
Main treatment plant 6	15.77±1.81	8.17±0.66	4.05±0.99	ND	ND	ND
Tanning factory treated wastewater 7	102.33±1.71	84.88±4.04	49.91±2.05	31.66±3.44	17.61±4.05	ND
Tanning factory non-treated wastewater 8	123.11±3.55	91.71±6.55	55.61±7.23	37.98±6.02	19.66±3.55	7.33±1.29
Carton factories 9	89.03±3.55	68.88±7.14	44.13±3.33	27.77±4.48	10.92±3.03	ND
Factories Lake 10	62.05±2.15	47.33±4.82	19.78±2.76	8.11±1.88	ND	ND
Grease refining plants 11	89.51±3.71	55.81±5.27	30.45±5.12	11.88±2.22	ND	ND

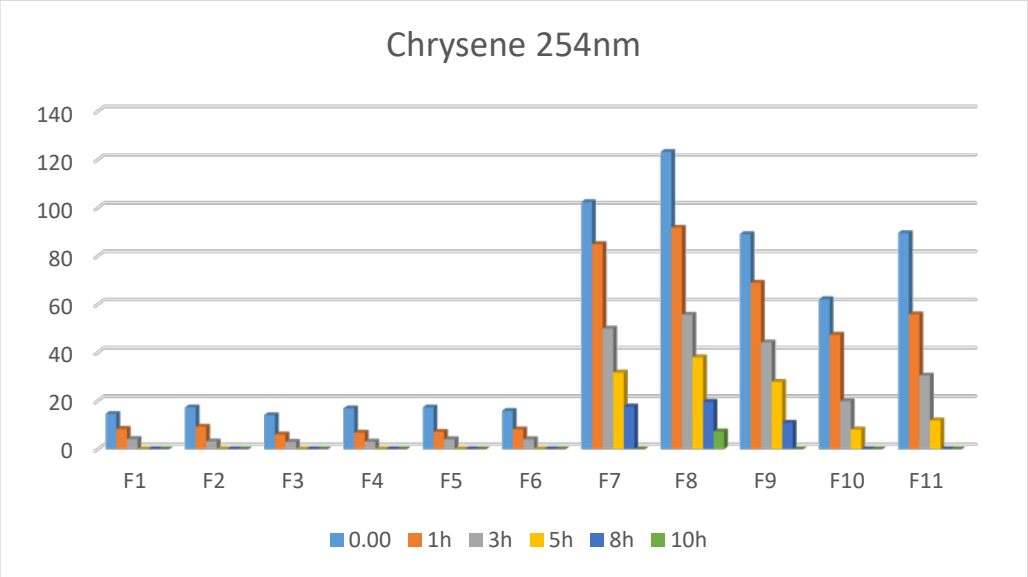


Figure 2: Effect of UV remediation (254nm) on Chrysene (4 rings) PAH with ZnO + TiO2 + H2O2 catalysts.

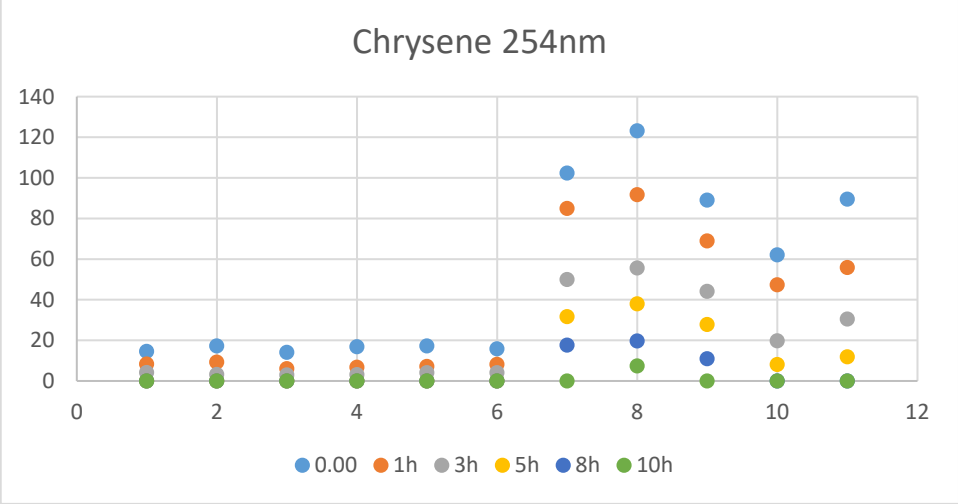


Figure 3: Effect of UV remediation (254nm) on CHRYSENE (4 rings) PAH with ZnO + TiO2 + H2O2 catalysts.

3.3. Effect of UV remediation (306nm) on Chrysene with ZnO + TiO2 + H2O2 (1:1:1) in different Wastewater.

The effect of photocatalytic remediation at 306 nm on Chrysene compound with ZnO + TiO2 + H2O2 (1:1:1) catalyst using 1, 3, 5, 8, and 10h in different wastewater (Table 6 and Fig. 4 and 5). The results of the catalytic treatment of Chrysene at 306 nm in the water samples from the test area indicate that it was treated after a three-hour exposure period for each of the water sources 1 to 6, as shown in Table 6, at which time chrysene was not detected. The compound continued to decrease with the continuation of the treatment process for water sources 7, 8, and 9 until the concentration reached 9.21, 11.04, and 7.26 µg/L, respectively, after 8 hours of exposure, but was not detected after 10 hours. On the other hand, water sources 10 and 11 were fully treated after 5 and 8 hours, respectively. Finally, the Chrysene concentration in Factories Lake and grease refining plants wastewater samples was reduced and not detected at the time of 5h after the photocatalytic remediation using (306nm).

Table 6. Effect of UV remediation (306nm) on Chrysene (4 rings) PAH with ZnO + TiO2 + H2O2 catalysts.

WW Sources	Chrysene concentration (ppb) and Irradiation time (h)					
	0 Time (ppb)	1h	3h	5h	8h	10h
Farm1	14.56±1.67	6.79±0.81	ND	ND	ND	ND
Farm2	17.22±2.86	7.21±0.56	ND	ND	ND	ND
Farm 3	14.05±2.02	4.61±0.69	ND	ND	ND	ND
Farm 4	16.83±1.77	4.03±0.77	ND	ND	ND	ND
Farm 5	17.23±1.23	3.66±0.81	ND	ND	ND	ND
Main treatment plant 6	15.77±1.81	4.09±0.81	ND	ND	ND	ND
Tanning factory treated wastewater 7	102.33±1.71	66.29±5.22	32.55±3.17	22.03±1.38	9.21±1.06	ND
Tanning factory non-treated wastewater 8	123.11±3.55	82.44±3.77	39.61±2.87	23.12±4.56	11.04±1.88	ND
Carton factories 9	89.03±3.55	51.76±3.33	34.78±1.87	18.22±2.87	7.26±1.43	ND
Factories Lake 10	62.05±2.15	33.19±2.41	9.93±0.89	ND	ND	ND
Grease refining plants 11	89.51±3.71	38.22±2.79	21.67±2.45	8.62±1.62	ND	ND

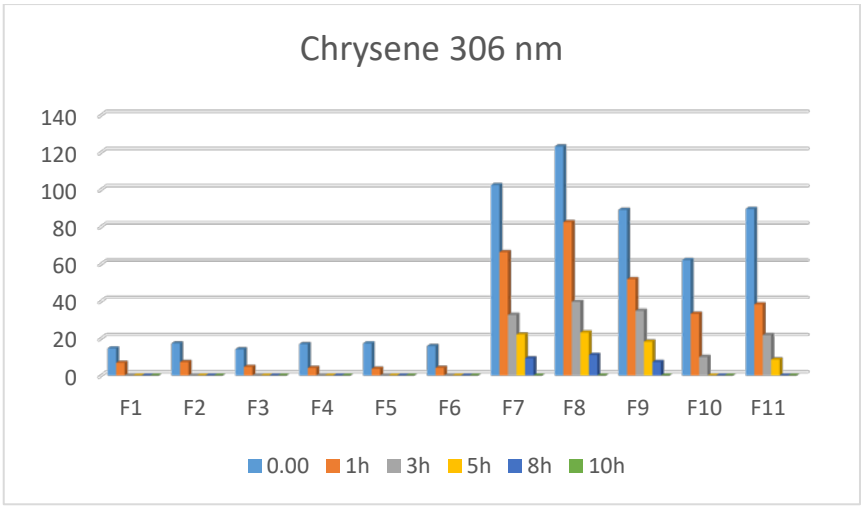


Figure 4: Effect of UV remediation (306nm) on Chrysene (4 rings) PAH with ZnO + TiO2 + H2O2 catalysts.

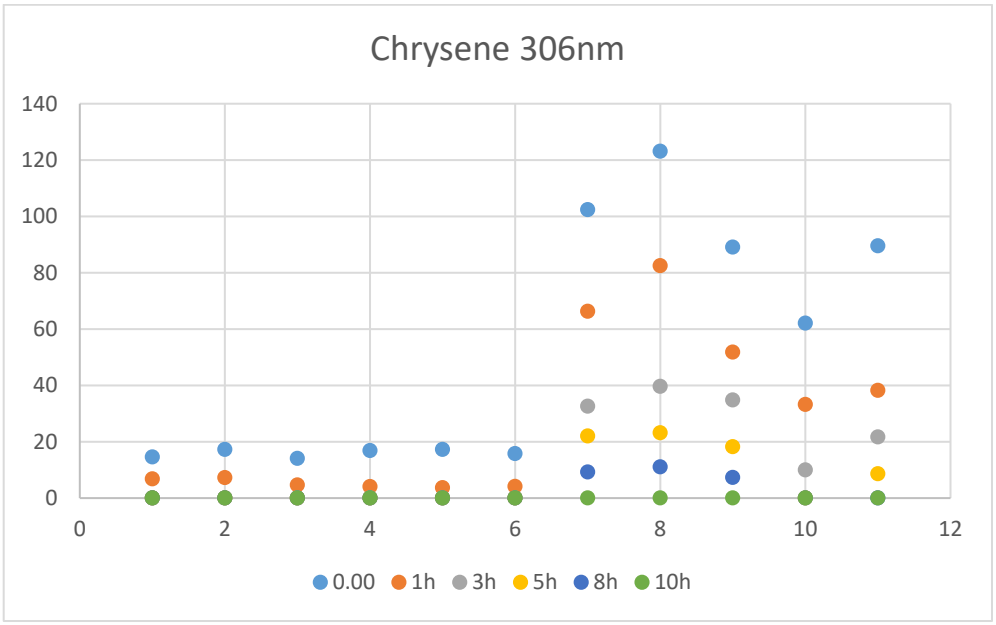


Figure 5: Effect of UV remediation (306nm) on Chrysene (4 rings) PAH with ZnO + TiO2 + H2O2 catalysts.

4. Conclusions

Photocatalytic degradation using ZnO + TiO2 + H2O2 (1:1:1) can play an efficient photocatalyst in the oxidation of Chrysene PAHs and converted to safer compounds, especially with Chrysene artificial or sunlight illumination to safe end, the effect of photocatalytic reactions on the degradation of Chrysene under different experimental

conditions **of catalyst and wavelength.** In this study, Photocatalysis (UV + ZnO + TiO2 + H2O2) successfully remediated the wastewater and subsequently activated the oxidative degradation of Chrysene in different sources of treated and untreated wastewater samples. The photocatalytic remediation with ZnO + TiO2 + H2O2 was effective in removing 94.69 – 100% of Chrysene PAHs within 5 -8 h by using 254nm. Moreover, the photocatalytic remediation with ZnO + TiO2 + H2O2 was effective in removing 97.61– 100% of Chrysene PAHs within 3 -8 h by using 306nm. Chrysene PAHs results also indicate that the highest concentration was in Tanning factory non-treated wastewater (8) followed by Tanning factory treated wastewater (7) for the wastewater tested samples. The average recovery of Chrysene ranged from 96-99%, and the Detection Limit (DL) was 5 µg/l. This study encourages the future application of this method with extraction by the QuEChERS method to estimate the PAHs in real environmental samples for future research. The exploration of the prediction and products of Chrysene PAHs will be helpful to better understand the fate of PAHs in the wastewater.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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