



Advanced Oxidation Process for the Removal of Pesticides Pollution in Water Sample Obtained from River Gongola.

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ABSTRACT

Advanced Oxidation Process is an advance technology that was discovered dating back to 1987 when Glaze introduced the concept, there are different methods and applications but however, many researchers has emphasize in the method that involve the generation of a sufficient quantity of hydroxyl radicals by irradiating H₂O₂ with a UV light to purify water and wastewater for its effectiveness in oxidizing pesticides pollutants. In this research three samples of water were collected from river Gongola and labelled ABC, treated and was sent to the laboratory for analysis of pesticides pollutants using GC-MS. The sample undergoes an extraction using the QUeCHER method after which then GCMS was run for the first time labelled (A1, B1 and C1) and a total 18 pesticides pollutants were detected including Organochlorine, Organophosphate, Neonicotinoids, Bipirydyls, at different concentration and were expressed in PPM and Retention time in minutes which includes the followings Methamidophos, Dichloropropene, Trichlorfon, Diazinin, Trichlorfon, Imidacloprid, Paraquat, Glyphosate, Diuron, Dichlorvos, Dichlorvos, Dichlorvos, Dimethoate, Methomyl, Chlorpyrifos, Endosulfan (α), DDT, Aldrin, Heptachlor epoxide, Heptachlor epoxide and another isomer of Endosulfan (α) after running the first GCMS, the sample was irradiated with UV in the presents of H₂O₂ with concentration of 8Mm and at a wavelength of 254nm after which the sample undergoes a second GCMS and a significant reduction was recorded in the concentration of the Pesticides level and removal of about 98.40% to 100% was achieved which proves that advance oxidation process can serve as an alternative solution to pesticides degradation as can be seen with level of pesticides removal efficiency recorded in the sample of water obtained from river Gongola and this has proven the use of AOPs base on experimental, theoretical and result analysis of the research can enhance its practical application for environmental contamination control.

Keywords; Pesticides, Degradation, Advanced Oxidation Processes (AOPs), Ultra Violet (UV), Radiation, Pollutants, H₂O₂

1. INTRODUCTION.

1.1Background.

Pollution removal is a crucial aspect of environmental protection and sustainability, particularly in the field of water and wastewater treatment. Advanced Oxidation Processes are key technology in removing pollutants by utilizing oxidation principles to break down contaminants through radical generation. Advanced oxidation processes usually abbreviated as (AOPs) are vital for converting pollutants into less harmful substances through breakdown and mineralization mechanisms. The efficacy of AOPs lies in their ability to generate highly reactive and nonselective free radicals like hydroxyl radicals ($\bullet\text{OH}$), superoxide radical (O_2^-), hydroperoxyl radical ($\text{HO}_2\bullet$), and alkoxy radical ($\text{RO}\bullet$) (Duarte and Amorim., 2020). These AOPs processes have emerged as an important approach to waste water treatment and majority of the processes are physicochemical in nature. As one of the advanced methods, AOPs are based on a basic principle that entails the generation and use of a hydroxyl free radical (HO^*) as a strong oxidant for the destruction of compounds which cannot be oxidized using conventional oxidants. AOPs have shown premises in various water treatment sectors due to its superior efficiency in pollutant elimination. Contemporary Types of AOPs are a suite of chemical treatment technologies designed to degrade persistent organic pollutants, pharmaceuticals, and industrial contaminants in water and wastewater. These processes rely on the generation of Reactive Oxygen Species (ROS), particularly hydroxyl radicals, which non-selectively oxidize complex pollutants into simpler, less harmful compounds. Their versatility in generating ROS through diverse mechanisms such as ozonation, electrochemical reactions, and photocatalysis makes them indispensable for addressing modern environmental challenges (Satyam and Sanjukta 2025). Problems associated with the use of pesticide can be extend beyond the farming area when residential areas are nearby, also health concerns associated with the handling and use of pesticides is increasing by day due to most farmer especially in the rural are often unable to read labels that are usually the only source of safety areas instructions and also do not have personal protective equipment (PPE) that are often for farmers as well as lack of knowledge and awareness to the farmers. Pesticides pose a particular danger to children, not only through their use but through their storage within the household. Statistics show that pesticides are involved in a significant number of suicides every

year. Pesticide residues have been proven by many researchers to constitute a significant source of contamination of environmental factors such as air, water and soil. This phenomenon if not control could become a continuous threat to the co-existence of plant and animal communities of the Ecosystem (Jayaraj *et al.*, 2016). pesticides pollutants are mostly man-made chemicals produced for agricultural and industrial uses and these chemicals end up contaminating all region of the world. The Safe and readily available water that is crucial for life and public health has become contaminated due to the growing defilation of various freshwater sources by modern agricultural practice, industrial effluent, human activity and oil spillage. industrial and municipal wastes have created multiple environmental hazards for mankind, irrigation, drinking and sustenance of aquatic life. The drainage water contains heavy metals in addition to biological contamination. This water pollution infects our food in addition to groundwater contamination when used to irrigate crops. (Mashmood and Arsalam., 2011).

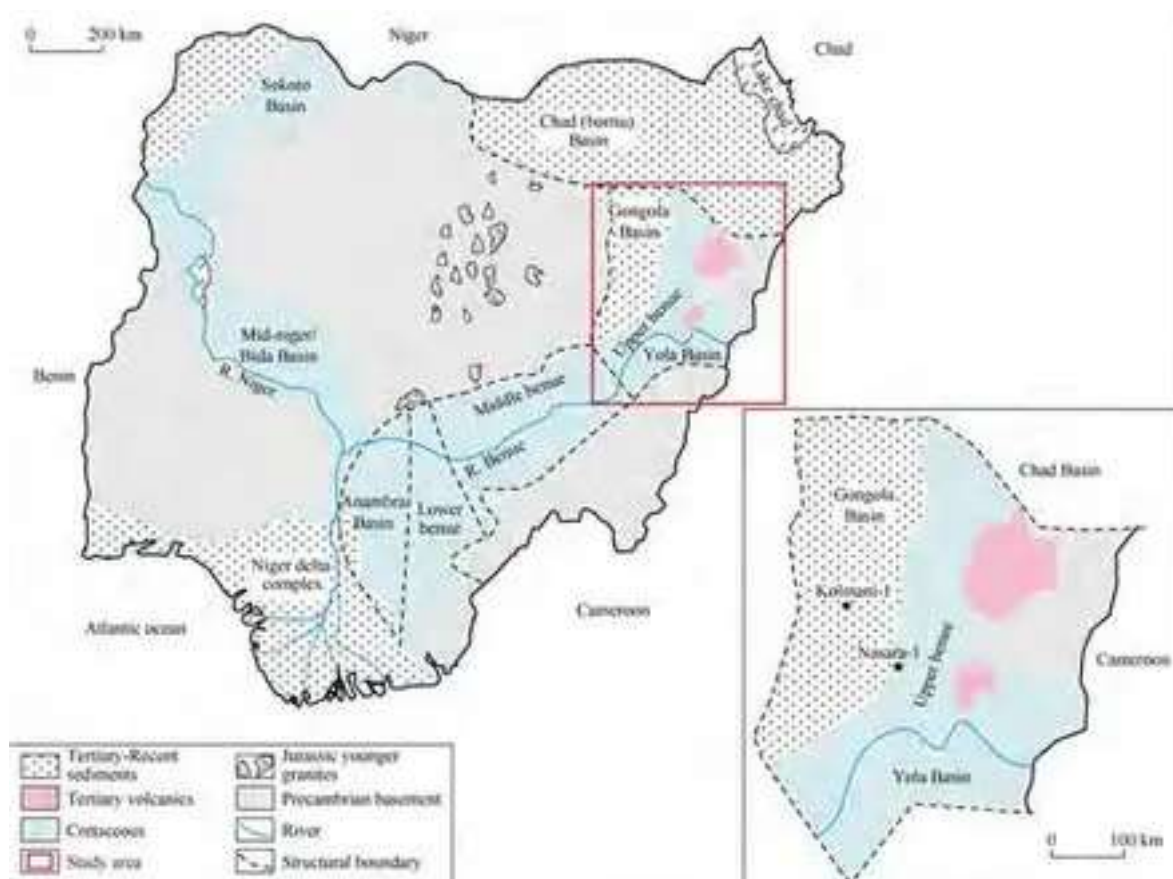
METHODOLOGY

All materials and reagents were of standard analytical grade had a purity of >97% and these includes: 2.5mL dichloromethane, 4g of anhydrous magnesium sulfate (MgSO₄), 1.5g Sodium acetate, anhydrous sodium sulfate, distilled water 2litres, vortex mixer, a weighing balance, fume chamber, centrifuge machine, micro syringe.

Study area

The research shall be carried out in River Gongola, situated at Yola, Adamawa state, Northeast Nigeria. It is located on the Benue River and has a population of 336,648 as at 2010. It is bounded by the coordinates 9013'48'' N 120 27'36'' E. River Gongola is the principal tributary of the Benue river. The upper courses of the river are seasonal streams, but fill rapidly in August and September. The Gongola runs south to the Benue River, joining it to the opposite town of Numan. Figure 1.2. shows the map of Yola showing the river Gongola (study site)

Fig 1.1, Map of Nigeria showing the Location of Adamawa State and the Gongola Basin



file:///C:/Users/Hp/Downloads/wps_download/map%20of%20gongola%20basin.webp

Fig. 3.3, Map of Adamawa state showing yola north local government area**Map of Adamawa state Showing the local government of the study area**

Obtain from google.

Water sample collection.

Standard water sampling procedure as described by (Staare, *et al.*, 2000) was adopted using Grab sampling techniques for collection of all the water samples. water sample which was collected from the river bank of river gongola, using amber glass bottles to prevent photodegradation of pesticides and filtered through a 0.45- μ m cellulose membrane filter to remove suspended particles. The pH of the filtered sample was measured using a calibrated pH meter (Hanna Instruments HI98107), and turbidity was determined with a turbidimeter (Hach 2100Q).

The water samples were stored in a labelled plastic bottle and immediately preserved after collection in ice under (4°C) in order to minimize degradation of the pesticides (POPs) before been transferred to the laboratory the samples was filtered with Whatman filter paper to remove particulate matter, and then acidified with dilute hydrochloric acid to pH 2.5 and stored at room temperature between 0°C to 4°C prior to extraction.

QuEChERS method for water extraction.

Water sample extraction was carried out using 10mL of water that was placed in a 15mL centrifuge tube and 2.5mL dichloromethane was added and the tube was capped and vortex for 3mins before adding 4g of anhydrous magnesium sulfate (MgSO_4). 1.5g Sodium acetate. The tubes was vortex vigorously for 1min and centrifuged for 10mins at 7000RPM. One milliliter of the supernatant was transfer into a 2mL vial containing 500mg MgSO_4 . The vials was shaken and subsequently centrifuged. After which The extract was transfered into a PTFE capped and was Analyze (*QuEChERS procedure for multi-residue pesticides analysis*).

GC-MS analysis of pesticides pollution

The GC- MS was the analytical instruments used to determine the pesticides concentration before and after the UV analysis. Extracted sample was analyzed for pesticide content using gas chromatography-mass spectrometry (GC-MS; Agilent 7890B/5977B). The GC-MS was equipped with a DB-5MS capillary column (30 m \times 0.25 mm \times 0.25 μ m). The oven temperature program was set as follows: initial temperature of 50°C (held for 2 min), ramped at 20°C/min to 280°C (held for 5 min). The mass spectrometer was operated in selected ion monitoring (SIM) mode for better sensitivity. Pesticide standards (e.g., glyphosate, atrforce, sniper 1000 EC, DD force, Glycot, Herbizin, atrazine, chlorpyrifos) were used to calibrate the instrument, and the limit of detection (LOD) was determined to be 0.1 μ g/L. The Gas Chromatography-Mass Spectrometry (GC-MS) results of the experimental water sample (Figure 4.1) reveal the presents of 18 different pesticides pollutants in the water sample, belonging to different class of pesticides such as Organochlorine, Organophosphates, Pyrethroid, Neonicotinoid insecticides contamination in terrestrial environments.

UV-AOP Treatment Setup:

A UV sterilization chamber (6W, 254 nm) was used for batch treatment. A 500-mL Quartz reactor was filled with the GC pre- analyzed water sample, and hydrogen peroxide (H₂O₂, 30% w/v) was added at a concentration of 8 mM (optimized from preliminary tests). The solution was continuously stirred using a magnetic stirrer (500 rpm) to ensure uniform exposure to UV light. The sample was irradiated for 60 minutes, with 5-mL aliquots collected at 60 minutes. During UV irradiation, the pH of the solution was monitored and maintained at 7.0 \pm 0.5 using dilute (0.01M) NaOH/H₂SO₄. Residual H₂O₂ concentrations were measured at each sampling interval using a titanium sulfate colorimetric method (Bader et al., 1988). Briefly, 1mL of sample was mixed with 0.1 mL of titanium sulfate reagent, and the absorbance was measured at 405 nm using a UV-Vis spectrophotometer (UNICO UV-5120).

The UV/H₂O₂ analysis for the decontamination of pesticides pollutants in water sample.

The UV/H₂O₂ analysis of the analyzed water sample (previously analyzed by GC-MS) was placed under a UV lamp setup to a wavelength of 254nm and H₂O₂ was added with the concentration 8Mm and properly installed and configured in a reactor vessel as obtained from (*Ultraviolet (UV) Disinfection in water: Ensuring Safe and Clean Water*), which from other literature is optimal for pesticides removal and after, the UV/H₂O₂ another GC-MS was run to ascertain the level of decontamination and a remarkable decontamination efficiency was recorded with degradation of up to 98-100% was achieved across the various pesticides detected.

Post-treatment analysis

After UV treatment, residual H₂O₂ was quenched by adding sodium sulfite (Na₂SO₃) at a 1:1 molar ratio. The final pesticide concentrations in the treated water were analyzed using GCMS as described in Step GCMS analysis step the removal efficiency (%) for each pesticide was calculated using the formula:

$$\text{Removal Efficiency(\%)} = 1 - C_t \times 100 / C_0$$

Where C₀ is the initial concentration and C_t is the concentration at time t

Results

From the result of the Analysis, 18 different pesticides were detected in the three water samples at different concentrations as expressed in (PPM) and different retention time in minutes and percentage recovery was expressed in (%). and the 18 pesticides detected belongs to the following classes of pesticides pollutant; including; Organophosphate; five class of organophosphate including (Methamidophos, Diazinin, Dichlorvos, Dimethoate, Chlorpyrifos) they all acts as insecticides used for the purpose of pest control but however end up in the water as pesticides pollutant and this class of pesticides pollutants are known to be toxic for human health as they potentially affects the nervous system including cholinesterase inhibition as stated in the literatures published by (WHO, USEPA AND NIOSH). Another class of pesticides detected were the Organochlorines including DDT, Aldrin and Endosulfan. Which are known for their carcinogenic and endocrine disrupting effects. In addition the two class of pesticides are all Persistent Organic Pollutants and these pollutants are known for their persistent nature and literatures have proven them to resist degradation by photolytic, chemical and biological means but however with UV-H₂O₂ AOPs we have achieved 98 and above percentage degradation in most of these pesticides pollutants as can be seen in the table of results

Table 1.1

Additionally, Pyrethroids and Others were also detected even though, none in the list directly belong to pyrethroids, but some have different modes of action. Others includes Herbicides such as; Paraquat: Bipyrindyl herbicide, Glyphosate: Organophosphnoate herbicide, Diuron: Urea herbicide Clopyralid: Pyridine carboxylic acid herbicide. Insecticides with Unique Structures; Imidacloprid: Neonicotinoid insecticide, Methomyl: Carbamate insecticide, Trichlorfon: Organophosphate insecticide (also has some carbamate-like properties). Fumigants as Dichloropropene, Fumigant including; (Methamidophos, Trichlorfon, Diazinon) and Neonicotinoids (Imidacloprid) With other pesticides below the limits of detection indicated by N/A as such

do not have any degradation effects. degradation across different compounds Detected in sample A,B,C with their initial concentration as (A1,B1 and C1) respectively, and final concentration (A2,B2,C2) all in the unit of (PPM), Their Retention time in Minute, their removal efficiency in percentage (%) all as can be seen in **Table 1.1**, This table also shows the GC-MS result of All the pesticides detected has shown a 98% and above degradation efficacy using the UV-H₂O₂ AOPs and this variability in degradation is attributed mostly to different in the pesticides structures, environmental condition and sometime microbial activities of the pesticides. Example, the following pesticides (Diazinon, Imidacloprid, Chlorpyrifos, Endosulfan(α) DDT and Aldrin) shows 100% degradation indicating their high susceptibility to degradation under the UV-H₂O₂ AOPs while other pesticides detected has shown 98% degradations and the following pesticides have initial concentrations of zero as such do no show any degradation effect (Trichlorfon, Methamyl, Heptachlor epoxide, Heptachlor and Dichloropropene) due to having zero concentration at the initial stage of the analysis indicating that its was below the detection limit.

Tables of Results

Table 1.1: Advanced Oxidation Process (AOPs) treatment. The results in table 4.2 demonstrate avarying degrees of pesticide degradation across different compound detected in sample A , before(A1) and after (A2) and the percentage removal of the pesticides as A removal

Peak	RT (min)	Compound	A1 (Before) conc (PPM)	A2 (After) conc (PPM)	A Removal (%)
1	2.9	Methamidophos	0.435	0.007	98.39
2	3.6	Dichloropropene	0.706	0.011	98.44
3	4.2	Trichlorfon	0.960	0.000	100.00
4	4.4	Diazinon	0.560	0.000	100.00
5	5.3	Imidacloprid	0.731	0.000	100.00
6	5.4	Paraquat	0.922	0.014	98.48
7	6.1	Glyphosate	0.689	0.010	98.55
8	6.6	Diuron	0.889	0.013	98.54
9	7.8	Dichlorvos	0.844	0.013	98.46
10	9.2	Clopyralid	0.386	0.006	98.45
11	10.5	Dimethoate	0.779	0.012	98.46
12	10.6	Methomyl	0.350	0.000	100.00
13	11	Chlorpyrifos	0.984	0.015	98.48
14	11.9	Endosulfan (α)	0.873	0.013	98.51
15	15.7	DDT	0.941	0.014	98.51
16	17.2	Aldrin	0.332	0.005	98.49
17	20.1	Heptachlor epoxide	0.940	0.014	98.51
18	20.8	Endosulfan (α)	0.465	0.007	98.49

From the results in **Table 1.1**, labelled as sample A, a total of 18 different pesticides were detected at different concentrations before and after treatment with UV-H₂O₂ advanced oxidation. the treatments has shows high removal efficacy, with most of the compounds degrading in the presents of UV/H₂O₂ exhibiting removal efficacy of 98% and above with those four compound showing a 100% removal efficacy after treatment in one trial of the including (Trichlorfon, Diazinon, imdacloprid and Methomyl) all other compound shows a significant reduction in concentration ranging from 98.39% to 98.55% . and the following compounds (Methamidophos) shows a removal efficacy of 98.39% with a final concentration of 0.007, also (Dichloropropene) exhibit 98.44% removal efficacy with a final concentration of 0.011% and (Glyphosate) with removal efficacy of 98.55% with it final concentration to be 0.010. the two isomers of endosulfan (α) exhibits different degrees of degradation with one having 98.13% removal efficacy and the other showing a 100% efficacy in removal in a simple breakdown of results analysis its can be conclude that sample (A) have attain a high degradation efficiency as a results of a favorable degradation conditions and chemical structure of the pesticides. with the above breakdown, the UV-H₂O₂ AOPs has proven to be an effective treatment methods in removing wide range of pesticides pollutants and the removal of these pesticides can help in mitigating environmental pollutions which in turns can results in protecting human health.

Table 1.2: showing the results of sample B obtained before (B1) and after (B2) treatments of sample B. and its removal efficacy

Peak NO	RT (min)	Compound Detected	B1(Before) concentration (PPM)	B2 (After) concentration PPM	B Removal (%)
1	2.9	Methamidophos	0.569	0.009	98.42
2	3.6	Dichloropropene	0.000	0.000	N/A
3	4.2	Trichlorfon	0.000	0.000	N/A
4	4.4	Diazinon	0.733	0.000	100.00
5	5.3	Imidacloprid	0.957	0.000	100.00
6	5.4	Paraquat	0.950	0.015	98.42
7	6.1	Glyphosate	0.902	0.014	98.45
8	6.6	Diuron	0.917	0.000	100.00
9	7.8	Dichlorvos	0.870	0.013	98.51
10	9.2	Clopyralid	0.505	0.008	98.42
11	10.5	Dimethoate	0.803	0.012	98.51
12	10.6	Methomyl	0.000	0.000	N/A
13	11	Chlorpyrifos	0.987	0.000	100.00
14	11.9	Endosulfan (α)	0.876	0.000	100.00
15	15.7	DDT	0.944	0.000	100.00
16	17.2	Aldrin	0.435	0.000	100.00
17	20.1	Heptachlor epoxide	0.000	0.000	N/A
18	20.8	Endosulfan (α)	0.609	0.000	100.00

TABLE 1.2, have shown 100% degradation efficiency in eight pesticide out of the total of eighteen with four pesticides below the limits of detection and all other pesticides detected have removal efficiency of 98.42% to 98.51% and this have proven the reliability of advance Oxidation Process to be an effective method in pesticides degradation when treating water pollution

TABLE 1.3 shows the result of analysis obtained before running the GCMS analysis labelled in the table as C1 (before) and C2 (after) refers to result obtained after UV-H₂O₂ irradiation of the sample and second GCMS.

Peak NO	RT (min)	Compound	C1 conc (PPM) (Before)	C2 conc (PPM) (After)	C Removal (%)
1	2.9	Methamidophos	0.000	0.000	N/A
2	3.6	Dichloropropene	0.239	0.000	100.00
3	4.2	Trichlorfon	0.320	0.000	100.00
4	4.4	Diazinon	0.431	0.000	100.00
5	5.3	Imidacloprid	0.000	0.000	N/A
6	5.4	Paraquat	0.633	0.024	96.21
7	6.1	Glyphosate	0.538	0.000	100.00
8	6.6	Diuron	0.606	0.011	98.18
9	7.8	Dichlorvos	0.580	0.022	96.21
10	9.2	Clpyralid	0.301	0.012	96.01
11	10.5	Dimethoate	0.535	0.000	100.00
12	10.6	Methomyl	0.000	0.000	N/A
13	11	Chlorpyrifos	0.000	0.000	N/A
14	11.9	Endosulfan (α)	0.587	0.011	98.13
15	15.7	DDT	0.000	0.000	N/A
16	17.2	Aldrin	0.000	0.000	N/A
17	20.1	Heptachlor epoxide	0.000	0.000	N/A
18	20.8	Endosulfan (α)	0.000	0.000	N/A

TABLE 1.3 shows the result of analysis obtained before running the GCMS analysis labelled in the table as C1 (before) and C2 (after) refers to result obtained after UV-H₂O₂ irradiation of the sample and second GCMS and from the table it can be seen that there are eight pesticides that are below the limit of detection with 0.000 concentration, four others with whose concentrations ranges from 0.538 to 0.239 (PPM) have attain 100% degradation efficiency and others with like paraquat, clopyralid and dichlorvos be degraded completely but have achieved 96.01%, 96.21% and 96.21% degradation efficiency and an incomplete decontamination of the pollutants concentration. In conclusion, the result above have shown a remarkable degradation efficiency of from 96.01% to complete degradation of 100% percentage.

SUMMARY

This research aims at evaluating the performance of Advanced Oxidation Process in the degradation of pollutants in water sample and an analytical technique GC-MS was employed to detect the presents of these pesticides pollutants in the sample of water and the GCMS analysis detected 18 different pesticides with their concentration expressed in (PPM) and their retention time in minutes. The sample sets were labelled (A, B and C), and during GC-MS determination the analyzed sample were labelled (A1, B1, C1) before the UV/H₂O₂ at the first GCMS analysis and after the UV/H₂O₂ the GC-MS detection were labelled as (A2, B2, C2) just as seen in table 4.1. The UV-AOP (Advanced Oxidation Process) treatment results have demonstrated varying degrees of pesticide degradation as where an Exceptional Degradation Efficiency was observe The UV-AOP treatment demonstrated remarkable effectiveness, with most pesticides showing >98% degradation across all samples. Complete degradation (100%) was achieved for several compounds including Trichlorfon, Diazinon, Imidacloprid, Methomyl, and others in at least one sample set. Additionally, the treatment showed consistent in performance across different sample sets (A, B, C), with minor variations in degradation efficiency. Sample set B generally showed slightly better degradation than A and C for most compounds. The UV-H₂O₂/AOP method proved highly effective against a wide range of pesticide classes including:

* Organophosphates (Methamidophos, Trichlorfon, Diazinon)

* Neonicotinoids (Imidacloprid)

- * Organochlorines (DDT, Aldrin, Endosulfan)
- * Bipyridyls (Paraquat)
- * Ureas (Diuron)

Conclusion

The GCMS analysis demonstrates that UV-H₂O₂/AOPs treatment is highly effective for degrading a wide range of Pesticides pollutants as can be seen in the table of result, **TABLE1.1.** with most compounds showing >98% removal and many achieving complete degradation. The consistent performance across different sample sets suggest the method's reliability for pesticide remediation applications, as such stakeholder involve in environmental protection and remediation are advice to includes the advanced oxidation process as one of the effective method to be employed when it comes to environmental remediation involving water pollution.

Recommendation

From the result of the data obtained, here are recommendation why stakeholder should include the application of Advanced oxidation process for the remediation of water pollution due to the effectiveness of the technology for the removal of over 97% and above of the pesticides pollutant;

- Efficient degradation; AOPs can degrade a wide range of pesticides pollutants including recalcitrant
- Environmental compatibility: AOPs offers environmental friendly treatments by minimizing secondary pollution
- Versatility AOPs; can be tailored to target a specific pesticides of interest base on their reactivity and structure
- Mineralization; AOPs can completely mineralize pesticides into CO₂, H₂O and inorganic ions thereby reducing toxicity

Contribution to Knowledge

By advancing the knowledge base of AOPs and their application to pesticide pollution and water contamination, this research will provide valuable insights for academics researchers, and students in environmental science and engineering fields and shall fosters further research and development in advance treatment technologies, contributing to the broader scientific community's understanding and innovation.

2. As the need to strictly regulate the environmental contaminants through effective technologies compliance with standards for pollutant removal grows, the findings from this research can help policymakers and regulators giving recommendations base on evidence for integrating advanced oxidation technologies into environmental management practices.

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