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Zinc oxide-catalyzed UV-photodegradation of cyhalothrin: A kinetic analysis

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ABSTRACT

In this study, we present kinetic studies of the photodegradation of cyhalothrin (CyH) under ultraviolet (UV) irradiation and the photocatalytic activity of zinc oxide (ZnO) in UV-assisted photodegradation. CyH in an acetone solvent was periodically exposed to $UV_{254\ nm}$ radiation light with a surface power density of 48 W/cm^2. The photodegradation experiments were conducted by acquiring periodic wavelength-scan spectroscopic data using a double beam ultraviolet-visible (UV-vis) spectrophotometer and plotting the real-time absorbance data to monitor the reaction coordinate. Under our optimized conditions at room temperature of 26.5 °C, atmospheric pressure of 76.6 mmHg, CyH (5.0 ppm) in the 70% acetone solvent with pH = 6.8 and dose of ZnO (25 ppm) exhibited typical photodegradation efficiencies between 81.9 and 90.3 % within the first 1,200 seconds of UV irradiation. Spectroscopic data showed that the ZnO solution significantly elevated the UV-assisted photodegradation rate of CyH by about 7.03 - 7.18 times more than that of UV-only-mediated CyH photodegradation. The result confirmed and characterized the photocatalytic activity of ZnO. Under the optimized measurement conditions, the rate of the ZnO-catalyzed photodegradation of CyH was found to follow the first-order rate law (RSq. = 0.999).

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1. Introduction

Cyhalothrin (CyH) is a synthetic pyrethroid insecticide widely applied to control several agricultural pests [1]. Generally, pyrethroid pesticides are used to control pests to increase crop yields; however, only about 0.1% of the pesticides sprayed on farms reach the intended targets, while the rest permeates, accumulates, and can persist in ecosystems, leading to environmental contamination of land, surface waters, and air. This portends pollution and toxicity problems. Impairment of the brain and neural systems, reproduction systems, and cancers are some of the long-term detrimental effects arising from exposure to pyrethroid insecticides beyond the threshold limit values [2-4]. To combat the risk of pesticide residue contamination, some researchers have explored photochemical and biological degradation approaches [5-8]. Photochemical methods that exploit metal oxides have been investigated to degrade persistent organic pollutants such as synthetic pesticides [9-11]. Photocatalytic degradation of CyH under visible light irradiation in the presence of ZnO mixed with Bismuth oxide (Bi_2O_3) under visible light has been shown to achieve a degradation efficiency of approximately 85.7% in 120 min [12]. Metal salt cerium nitrate and ZnO nanoparticles in the presence of solar irradiation have also been shown to be capable of degrading CyH by about 63 and 87%, respectively, in terms of efficiency [13,14].

The present study investigates the effect of ultraviolet (UV) irradiation on the photodegradation of CyH and characterizes the photocatalytic effect of zinc oxide (ZnO). Kinetic measurements were conducted via the acquisition of time-dependent wavelength scan spectrophotometric measurements to establish the photodegradation rates of CyH under UV irradiation and to characterize the enhancement of the UV-assisted photodegradation in the presence of ZnO in solution. Kinetic analysis showed that CyH undergoes photodegradation when exposed to ultraviolet (254 nm) radiation. The experiments also indicated that ZnO significantly enhanced the rate of UV-assisted photodegradation of CyH. These results can potentially motivate the development of a decontamination process that uses acidified ZnO and UV radiation to achieve photodegradation of trace levels of CyH as model pesticide residue. The ZnO-UV-mediated photocatalytic decontamination approach can, for example, find important applications in urgent cases where large-scale pollution is detected.

2. Experimental

2.1. Reagents and instrumentation

All reagents used in this study were analytical grade (percentage purity > 99%).

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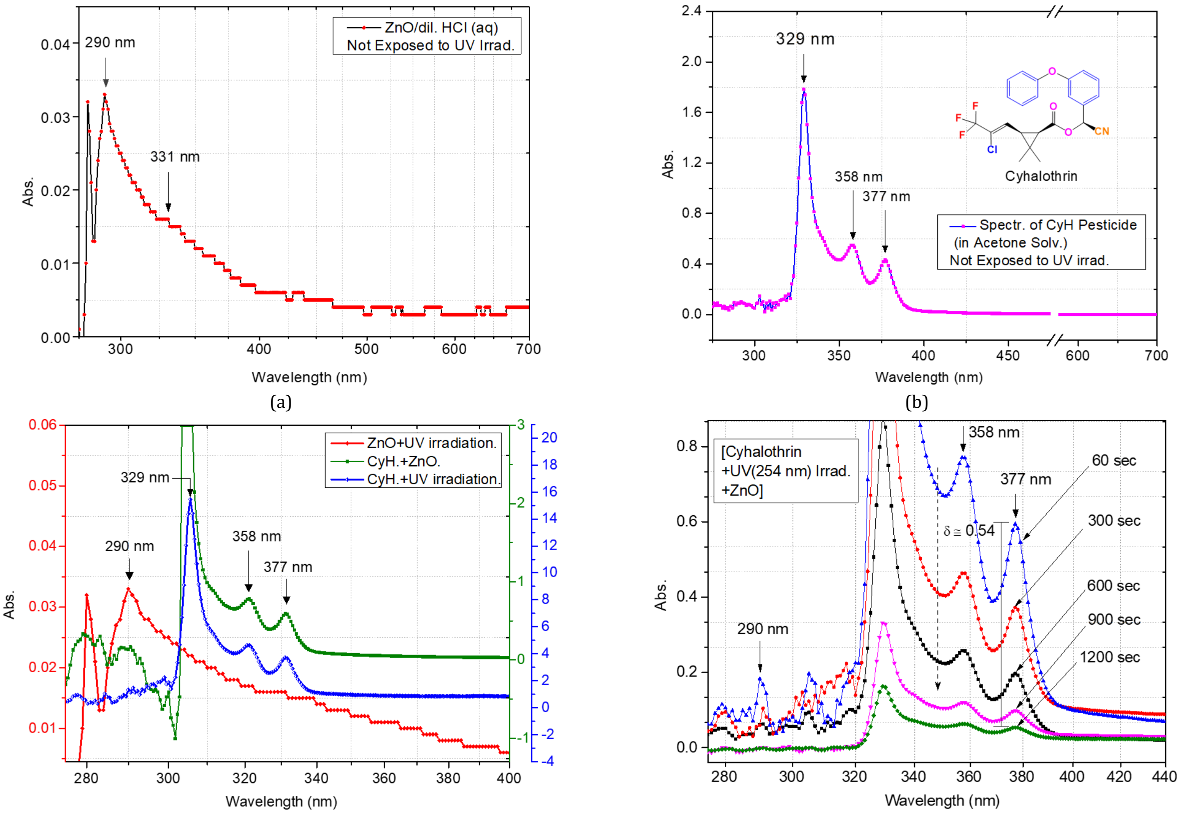

(a) (b) (c) (d)

Figure 1. (a) UV-vis spectrum of pure ZnO, (b) UV-vis spectrum of pure CyH, (c) UV-vis spectra of [pure CyH + UV irradiation] (blue) and [pure ZnO + UV irradiation] (red) and [CyH + ZnO] (green), and (d) time-resolved electronic absorption spectra based on the kinetic experiment: [CyH + UV_{Irrad} + ZnO].

Acetone, zinc oxide (ZnO) and deionized water (DI) were purchased from Kobian Scientific, Nairobi, Kenya. Lambda-cyhalothrin pesticide (50 g/L) was purchased from Elgon Kenya Limited. The source of irradiation energy used was a UV lamp (LF-204.LS model) (UVITEC). A double-beam UV-visible spectrophotometer (UV-VIS K9000 model) was used to acquire the spectroscopic wavelength scans for all kinetic experiments.

2.2. Photodegradation experiments and set-up

The experiment was carried out in a covered homemade compartment in dark conditions. The methodology used to achieve the objectives set in this study involved three distinct steps. The control experiment was prepared by mixing 25 ppm of zinc oxide solution in dilute HCl and 10 ppm of pesticide [CyH + ZnO] in dark conditions. The initial photodegradation experiment was a composition of 10 ppm CyH in acetone and then exposed to a UV lamp at 254 nm [CyH + UV_{254nm}]. The subsequent experiment involved an investigation of the effect of ZnO on the photodegradation kinetics of CyH. In this case, the reaction mixture combined 5 ppm CyH in acetone, 25 ppm of zinc oxide solution in dilute HCl and UV irradiation [CyH + ZnO + UV_{254nm}]. For the photodegradation experiments, the irradiation time was 1200 seconds, and the periodic measurements were taken after every 300 seconds of UV irradiation. Spectroscopic and kinetic data were acquired by performing wavelength scan measurements using a double beam UV-vis spectrometer in the range 190-1100 nm. All data sets generated in this study were analyzed using Origin Pro®v.9.0 software.

3. Results and discussion

3.1. Photodegradation of CyH (UV irradiation and ZnO)

Figure 1a shows the UV-visible spectrum of the ZnO solution as a reference spectrum of the photocatalyst to be investigated. As is evident in Figure 1a, ZnO exhibits a broad absorption band within the UV region with a maximum of 290 nm and a shoulder peak at 329 nm, according to previous spectroscopic characterization [15]. The spectrum acts as a reference to distinguish ZnO peaks from those related to the CyH absorption bands in the preceding experiments. As shown in the spectrum in Figure 1b, CyH alone in acetone solvent exhibits maximum absorption at 329 nm and two other strong absorption bands with peaks at 358 and 377 nm in the UV region. The peak at 331 nm in Figure 1b overlaps strongly with that of CyH at 329 nm in Figure 1a. However, the latter two peaks, *i.e.,* at 358 and 377 nm, do not overlap strongly with those of ZnO in Figure 1a, hence their choice in conducting kinetic studies. Figure 1c shows the spectra obtained from pure CyH + UV irradiation (blue), pure ZnO + UV irradiation (red), and of CyH + ZnO (green). Compared to the respective spectra in Figures 1a and 1b, UV irradiation, under the time scales of our measurements, was observed to not alter their characteristic absorption bands.

Figure 1d demonstrates the wavelength scans obtained from the kinetic experiment set up to monitor the photodegradation of CyH in the presence of ZnO and UV irradiation. It can be observed that the intensity of the absorption bands related to CyH decreases with time upon periodic UV exposure. This indicates the gradual decrease in the concentrations of absorbing chromophores related to CyH following the Beer-Lambert law [16].

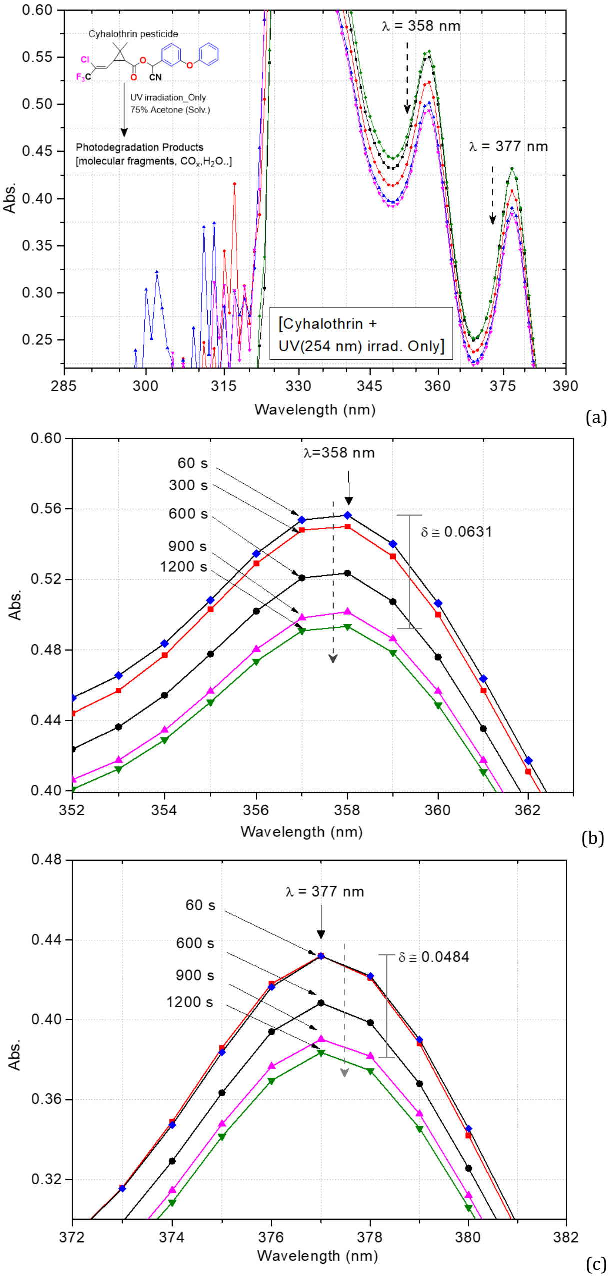

Figure 2. (a) Wavelength-scan electronic spectra obtained from the [CyH + UV_{Irrad}] kinetic experiment, (b) Absorbance band with max = 358 nm, and (c) Absorbance band with max = 377 nm.

This result demonstrates that the organic CyH pesticide substrate undergoes fast photodegradation when exposed to ultraviolet (UV) radiation in a ZnO solution.

The efficiency of CyH photodegradation under the measurement conditions was calculated using Equation 1 based on the absorbance values obtained from the spectroscopic data obtained in Figure 1d. Under our optimized measurement conditions of ZnO+UV-assisted degradation experiments, a substitution of the values A_t = 0.5994 and A_0 = 0.0537 obtained from the absorption band at 377 nm into the formula yields a photodegradation efficiency of 90.3%. A substitution of the values of A_t = 0.7714 and A_0 = 0.0631 obtained from the absorption band at 358 nm gives an estimated value of photodegradation efficiency of 81.9 %.

$$\text{Photodegradation efficiency (\%)} = \left(\frac{Ct}{Co - Ct}\right) \times 100 = \left(\frac{At}{Ao - At}\right) \times 100 \quad (1)$$

where: C_t = Concentration at time t, A_t = Absorbance at time t, C_0 = Initial concentration, A_0 = Initial absorbance.

3.2. Photodegradation of CyH (using UV irradiation only)

Figure 2a depicts the time-resolved wavelength-scan electronic spectra obtained from [CyH + UV_{Irrad}]. Kinetic experiment for the first 1200 s where only UV irradiation was applied. Figures 2b and 2c inset are extracted from Figure 2a and highlight the characteristic absorption bands of CyH with peaks at 358 and 377 nm, respectively. The decrease in intensities over time (monitored from 60th to 1200th s) indicates that UV radiation causes some photodegradation of CyH in the absence of ZnO.

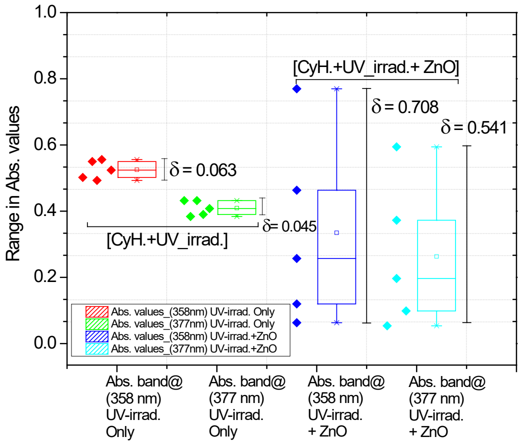

Figure 3. Box plot describing the spread/range in absorbance values recorded during the [CyH + UV_{Irrad}] and [CyH + UV_{Irrad} + ZnO] kinetic experiments.

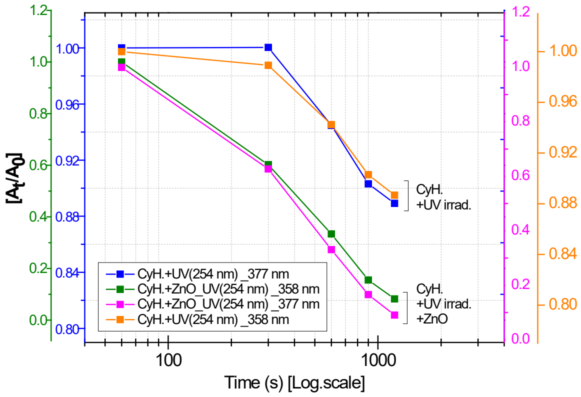

Figure 4. Multi-curve time-lag comparing the variation of absorptions during the [CyH + UV_{Irrad}] and [CyH + UV_{Irrad} + ZnO] kinetic experiments.

The photodegradation effect of UV radiation on the decomposition of persistent organic pollutants, pesticides, and their residues has previously been studied by several researchers in the recent past [12-14,17]. The set of results shown here characterizes the photodegradation of CyH under the set of measurement conditions.

The rate of UV-assisted, however, is lower than that observed in the case of UV-photodegradation in the presence of ZnO solution, illustrated earlier in Figure 1a. This can be seen by comparing the magnitudes of the intensity changes in the absorbance values recorded in the two kinetic experiments within the 60 - 1200 s durations.

3.3. Photocatalytic activity of ZnO on CyH degradation

Figure 3 shows a box plot that describes the spread and/or range of the absorbance values obtained from the two kinetic experiments; [CyH + UV_{Irrad}] and [CyH + UV_{Irrad} + ZnO] performed in a similar manner and for similar periods. The absorbance values analyzed here are those extracted from the wavelength scan measurements depicted in Figures 1 and 2 for the absorptions at 358 and 377 nm in each experiment. The box plot gives an idea of the dispersion or variability of the absorbance values among the two data sets.

It can be observed that the spread in the absorbance values obtained for the [CyH + UV_{Irrad}] experiment is smaller than those obtained in the [CyH + UV_{Irrad} + ZnO] experiment. The range values in absorbance data recorded for the [CyH + UV_{Irrad}] experiment was 0.063 and 0.045 at the corresponding peaks of 358 and 377 nm, while, for the [CyH + UV_{Irrad} + ZnO] kinetic experiment, they were 0.708 and 0.541, as summarized in Figure 3. The interquartile range of the absorbance values obtained in the [CyH + UV_{Irrad} + ZnO] kinetic experiment is also significantly higher than those obtained in the [CyH + UV_{Irrad}] experiment.

The significant change in the spread and range in absorbance values observed in the kinetic experiment [CyH + UV_{Irrad} + ZnO] as opposed to those observed in the [CyH + UV_{Irrad}] experiment demonstrates the greater extent of photodegradation of the CyH substrate. This characterizes the photocatalytic activity of ZnO in the degradation of CyH in our case. The photocatalytic activity of ZnO has also been demonstrated in previous degradation studies in some pharmaceuticals and pesticide contaminants [7,18,19].

Figure 4 shows a multicurve time-lag plot where the absorbances at 358 and 377 nm are plotted against time on a logarithmic scale. It compares the change in absorbance recorded during the two separate kinetic experiments: denoted as [CyH + UV_{Irrad}] and [CyH + UV_{Irrad} + ZnO] respectively in the chart. It can be seen from the graphs that in the case where ZnO is used as a photocatalyst, there is a steeper drop in CyH concentration between 60-300 s, as opposed to the case where no ZnO is used, that is, in the [CyH + UV_{Irrad}] experiment. In both cases, the progressive drop in absorbance values observed from 300 - 1200 s indicates degradation of the CyH substrate under UV irradiation.

Table 1. Average first-order photodegradation rate constant parameters compared by the two kinetic experiments based on the basis of the computed gradients.

Absorption bands of CyH in the UV region	**Average 1st-order rate constants (k_{av}) (Computed from the gradients)**		**Average rate incremental factor**
	[Cyhalothrin + UV irradiation] (α)	**[Cyhalothrin + UV irradiation + ZnO] (β)**	**(β/α)**
358 nm	1.090×10^{-4}	7.831×10^{-4}	7.184
377 nm	1.113×10^{-4}	7.831×10^{-4}	7.036

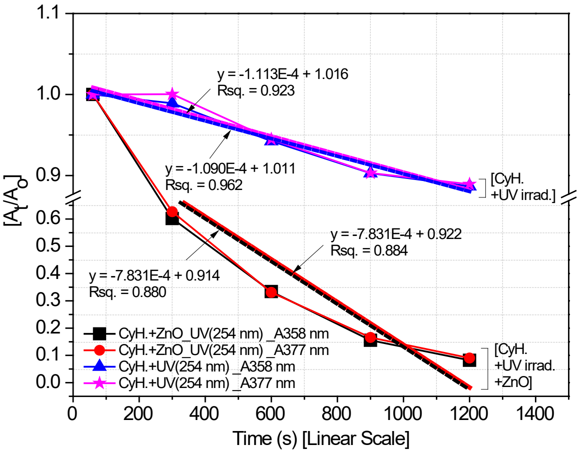

Figure 5. A plot of [A_t/A_o] *versus* time for the photodegradation rates of the kinetic experiments of [CyH + UV_{Irrad}] and [CyH + UV_{Irrad} + ZnO].

The drastic drop in concentration that occurred much earlier in the case of the [CyH + UV_{Irrad} + ZnO] experiment is evidence of an accelerated rate of degradation of CyH, which characterizes the photocatalytic effect of ZnO.

Figure 5 shows a linearized comparison of the photodegradation rates of the kinetic experiments [CyH + UV_{Irrad}] and [CyH + UV_{Irrad} + ZnO]. The absorbance values are those of the wavelength-scan experiments in (Figures 1 and 2) for the peaks at 358 and 377 nm. It can be noted that steeper gradients in the photodegradation rates are observed in the case of the [CyH + UV_{Irrad}] experiment. The coefficient of determination (R^2) derived from the fitted values ranges from 0.923-0.962 in the case of the [CyH + UV_{Irrad}] experiment, while in the case of [CyH + UV_{Irrad} + ZnO] experiment it ranges between 0.880 and 0.884. Although relatively high RSq. Values are observed, and the variation in the values would possibly imply a difference in the photodegradation mechanisms or pathways between the two experiments.

Table 1 gives the average first-order photodegradation rate constant parameters comparing the two kinetic experiments based on the computed gradients. They highlight the experimental photodegradation rates in the case of UV irradiation only (labelled as α in column 2) and in the case of UV irradiation with ZnO (labelled as β in column 3). The slope of the fits compares the average first-order photodegradation rates ($k_{av.}$) for the two experiments. It can be seen that the k_{av} values increase by factors between 7.036 and 7.184 after the introduction of ZnO. This highlights the photocatalytic efficiency of ZnO in the degradation of CyH. Therefore, it can be noted that ZnO under our optimized measurement conditions enhances the photodegradation of CyH by a photocatalytic efficiency factor of between 7.036 and 7.184 relative to the uncatalyzed UV-assisted photodegradation reaction.

3.4. Kinetic analysis of photocatalytic degradation of CyH

Figures 6a-6c show the absorbance data fitted on the models for the zero-, the first-, and the second-order integrated rate laws derived from the kinetic experiment [CyH + UV_{Irrad} + ZnO]. The fitted tests were carried out on based on the maximum absorbance values obtained from the CyH spectroscopic characteristic absorption bands at 358 and 377 nm indicated earlier in Figure 1b. The linear fittings of all kinetic data in all three scenarios are characterized by the relevant linear equations and their respective RSq. values. A cursory check of the kinetic graphs shows that the zero-order and second-order graphs are curves (Figures 6a and 6c), while the graph for the first-order fitting is linear (Figure 6b). Based on RSq. values obtained from the linear fit kinetic analysis, it is evident that the values obtained from the [CyH + UV_{Irrad} + ZnO] experiment best fit with the first-order kinetic rate equation (Figure 6b). This is because, of all three graphical fitted tests, the first-order filling has the highest RSq. values of 0.999-0.998 using the two characteristic absorption peaks of 358 and 377 nm. These results indicate that the ZnO-catalyzed photodegradation of CyH pesticide under our optimized measurement conditions proceeds via first-order kinetics.

4. Conclusion

In this study, the kinetics of the photodegradation of CyH in the absence and the presence of ZnO was successfully investigated using UV spectroscopy. The effect of UV_{254nm} irradiation on the photodegradation of CyH and the catalytic activity of ZnO in enhancing CyH UV photodegradation was elucidated by monitoring the decreases in intensities of absorption bands of CyH substrate using time-resolved wavelength scan spectrophotometric measurements. The intensity of the characteristic absorption peaks at 358 and 377 nm that were monitored over time depicted the photodegradation of CyH in the presence of UV radiation alone and in the presence of both UV radiation and ZnO. It emerged that the rate of photodegradation of CyH in the presence of ZnO was about seven times compared to that in the case in which only UV radiation was used to cause photodegradation. The enhancement of the photodegradation of CyH in the presence of ZnO from the spectroscopic measurements characterized its photocatalytic effect. The graphical fitted kinetic analysis tests of ZnO-catalyzed degradation of CyH showed that the photolytic reaction followed the first-order integrated kinetic rate law (RSq. = 0.99). These laboratory photodegradation kinetic studies on a selected trace-level pesticide residue using a ZnO-based photocatalyst can act as a scientific basis that would inform and justify the potential applicability of photocatalysts

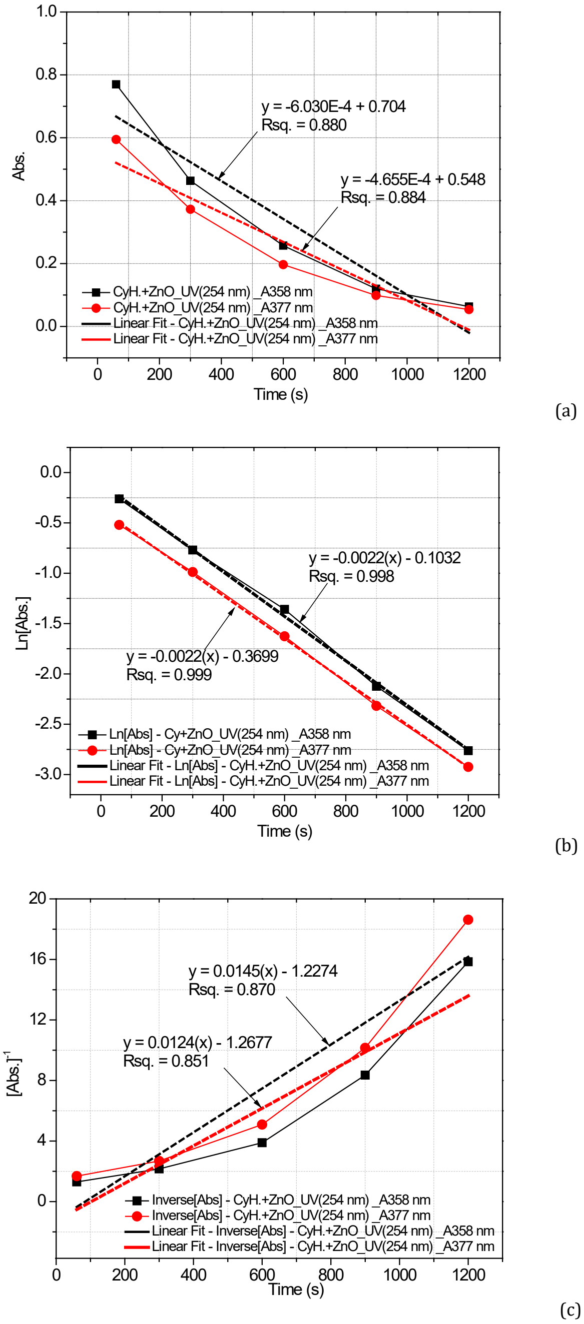

Figure 6. Fitted kinetic models for the [CyH + UV_{Irrad} + ZnO] experiment: (a) Zero-order; (b) first order, and (c) second order.

in the photodegradation of other known pesticide residues toward augmenting environmental remediation efforts.

Disclosure statement

Conflict of interest: The authors declare that they have no conflict of interest.
Ethical approval: All ethical guidelines have been adhered to.
Sample availability: Samples of the compounds are available from the author.

CRediT authorship contribution statement

Conceptualization: Seth Otieno Osumba, John Onyango Adongo; Methodology: Seth Otieno Osumba; Data Curation: Seth Otieno Osumba, John Onyango Adongo, Josiah Ouma Omolo. Writing - Original Draft: Seth Otieno Review: Josiah Ouma Omolo; Osumba Visualization: Seth Otieno Osumba; Project Administration: Seth Otieno Osumba; John Onyango Adongo.

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