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Photodegradation of Neonicotinoid Insecticides Nitenpyram, Thiacloprid, and Acetamiprid in Water and Soil Environments

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Abstract

The photodegradations of three selected neonicotinoid insecticides nitenpyram, thiacloprid, and acetamiprid were investigated in both water and soil samples under natural sunlight, UVA light, and UVB light. The results indicate that these insecticides undergo significant degradations when subjected to sunlight, whether they are in deionized (DI) water, tap water, and DI water containing 100 mg/L humic acids or in soil. The degradation half-lives of nitenpyram, thiacloprid, and acetamiprid in tap water under sunlight were found to be 3.7, 4.7, and 8.9 h, respectively, in DI water 5.4, 6.3, 9.1 h, respectively, in DI water containing 100 mg/L humic acids 3.6, 3.3, 6.5 h, respectively, and in soil 7.5, 7.9, and 15.9 h, respectively. The degradation due to hydrolysis was found insignificant as compared to photodegradation. The examination of the effects of light source revealed that the UVB in the sunlight plays a major role in the photodegradation of these three neonicotinoids, and the effects of UVA and visible light are negligible. The analysis on the degradation products indicated that the nitroguanidine group in these insecticides is unstable and prone to break up under sunlight. A total of nine degradation products were detected, of which the health effects and the fate and transport in the environment need to be further studied.

Keywords Neonicotinoid · Photodegradation · Sunlight · Soil · Water · Environment

Introduction

Neonicotinoids belong to the nitroguanidine family of systemic insecticides and are widely used in seed treatment at planting to protect plants from early-season root and foliar-feeding pests, as well as later foliar treatment (Jeschke et al. 2011; Huseth et al. 2014; Morrissey et al. 2015). Since the first global commercialization of imidacloprid in early 1990s, imidacloprid has been the most widely used neonicotinoid on the insecticide market and has been registered globally on more than 140 crops in 120 countries (Jeschke et al. 2011; Simon-Delso et al. 2015). It provides excellent protection against a wide range of soil-borne insects and early season sucking/chewing insects (Bonmatin et al. 2015; Liu et al. 2015). Following the success of imidacloprid, other neonicotinoid insecticides with similar structure were launched into the market, including clothianidin, thiamethoxam, dinotefuran, acetamiprid, thiacloprid, and nitenpyram (Li et al. 2018a). The annual uses of neonicotinoids increased from approximately 17% of the total insecticide market in 2006 to 25% in 2014 (Goulson 2013; Jeschke et al. 2011; Thompson et al. 2020). As of 2016, the usages of neonicotinoid insecticides increased to 24% of the total market, with an average sale of nearly $1.6 billion (Kurwadkar et al. 2016).

The large-scale use of neonicotinoids in urban and rural areas raises concerns on the environmental health and other non-target organisms. Residues of neonicotinoids have been found in some water systems, and a correlated reduction of aquatic insect populations was observed (Van Dijk et al. 2013; Huseth and Groves 2014). In Japan, a study was conducted to monitor the presence of neonicotinoids in rivers in Osaka City (Yamamoto et al. 2012). Acetamiprid,

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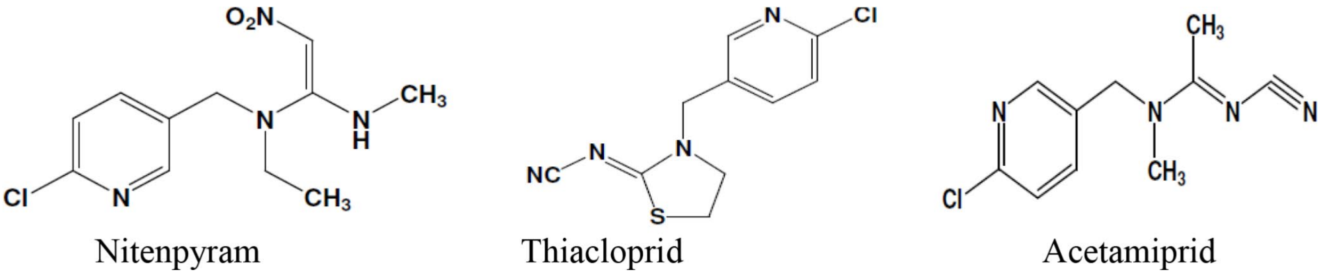

Fig. 1 Chemical structures of three insecticides

clothianidin, dinotefuran, imidacloprid, nitenpyram, and thiamethoxam were analyzed and dinotefuran was most often found with the highest concentrations (maximum 0.2 µg/L) in most of the samples. Radolinski, et al. (2018) conducted a column test with thiamethoxam-coated corn seeds in soil columns and found that in the leachate the thiamethoxam concentrations were high enough to be acutely toxic to aquatic life.

Environmental persistence of neonicotinoids is contingent on their mechanisms of resistance to both biotic and abiotic degradations. Photodegradation is one of the major abiotic degradation mechanisms. The photodegradation of imidacloprid in water solutions have been reported by many researchers (Moza et al. 1998; Wamhoff and Schneider 1999; El-Hamady et al. 2008). Their results indicated that imidacloprid is quick to degrade under sunlight, with an average half-life of 1.3 h. Photodegradation of other neonicotinoids with addition of catalyst such as TiO_2, Fe_3N, and Ag_3PO_4 was also reported, and the results confirmed the unstable status of neonicotinoids under the exposure of sunlight (Zabar et al. 2012; Lee et al. 2020; Padervand et al. 2021; Zelić et al. 2022). Very few studies have been done for the photodegradation of neonicotinoids in soils, as compared to those in water. An apparent reason is that light does not penetrate into soils. However, the neonicotinoids falling on the soil surface through foliar spray are subjected to photodegraduation. It is necessary to study the photodegration of the neonicotinoids in thin layers of soils from the fields where the insectidies are applied. Gupta et al. (2008) studied the photodegradations of acetamiprid and thiacloprid in silty caly loam in India. Li et al. (2018a) studied the photodegradation of clothianidin and thiamethoxam in agriculural soils in Mississippi. Data concerning photodegradations of other neonicotinoids in both water and soils are scarce.

This study was aimed at examining the effects of sunlight on the degradation of three commonly used neonicotinoids nitenpyram, thiacloprid, and acetamiprid in agricultural soils and water bodies. Natural sunlight, ultraviolet A (UVA) light and ultraviolet B (UVB) light were used as light sources to investigate the degradation rates, patterns, and effects of sunlight wavelength ranges on the photodegradation processes of these neonicotinoids. The degradation pathways and products of these neonicotinoids under sunlight were also scrutinized.

Table 1 Physical-chemical properties of neonicotinoid insecticides

Neonicotinoid	Water solubility (g/L) [a]	Octanol/water partition coefficient [a]	Dissociation coefficient [b]
Acetamiprid	4.2	0.8	0.7
Thiacloprid	0.184	1.26	ND [c]
Nitenpyram	840	-0.64	3.1

a. Data from Jeschke et al. (2011)

b. Data from Bonmatin et al. (2015)

c. No data available

Materials and Methods

Neonicotinoids

Analytical grade nitenpyram ($C_{11}H_{15}ClN_4O_2$; assay 99.9%), thiacloprid ($C_{10}H_9ClN_4S$; assay 99.0%), and acetamiprid ($C_{10}H_{11}ClN_4$; assay 99.5%) were purchased from Chem Service, Inc. The chemical structures and physical-chemical properties of these insecticides are presented in Fig. 1; Table 1, respectively. Stock solutions of 100 mg/L these neonicotinoids were prepared in high-performance liquid chromatography (HPLC) grade methanol (Fisher Scientific) and stored in refrigerator at 4°C.

Soil

Soil samples from Mississippi State University's Truck Crops Branch Experiment Station located in Crystal Springs (TCB) were used for the photodegradation test. The TCB soil was defined as Providence silt loam. The soil samples were taken from top 0–60 mm at the TCB field. They were air-dried and ground to pass a sieve with 2-mm openings. The moisture content of the air-dried soil samples was measured to be 1–2%. The cation exchange capacity, organic carbon content; clay content, silt content, and pH were measured to be 10.9 cmol/kg, 0.31%, 19.3%, 65.0% and 5.0 respectively.

Water Solutions

Deionized (DI) water, tap water, and DI water containing 100 mg/L humic acids were used as the solutions in which the neonicotinoids were dissolved and tested for photodegradation. The DI water was generated from Barnstead E-Pure Water System (Thermo Scientific) and had a resistivity 15.0 MΩ·cm and a pH 6.1. The tap water was from the City of Jackson, Mississippi drinking water plant and had a hardness 164 mg/L and pH 7.3. The humic acid-containing water was made by dissolving humic acid (Technical grade, Thermo Scientific) into DI water. The water containing 100 mg/L humic acids was used because the concentration of humic substances in surface waters typically ranges from 0.1 mg/L to 20 mg/L and in some stagnant waters, it is possible to reach 500 mg/L (Barlokova et al. 2023).

Light Sources

The light sources included the natural sunlight during 9:00 AM to 3:00 PM in June in Jackson, Mississippi, UVA light (315–400 nm), and UVB light (280–315 nm). UVC (200–280 nm) light was not used because when the natural sunlight passes through the atmosphere, the ozone layer adsorbs vast majority of the lights with wavelength less than 290 nm. The UVA and UVB lights were produced from UV lamps (Spectroline E-series ENB-280 C, Spectronics Cor.). The intensities of UVA light, UVB light and visible light (400–700 nm) in these three light sources were measured using a data logging radiometer (PMA2100, SOLAR Light Co.), and the results are summarized in Table 2. The ambient temperature during the sunlight exposure test period typically ranged from 24 °C to 32 °C, while the temperature in the laboratory where the exposures to UVA and UVB lights were conducted was 20 ± 2 °C.

Photodegradation Test in Water Solutions

The photodegradations in three different water solutions were conducted under natural sunlight. Samples containing nitenpyram, thiacloprid, and acetamiprid were prepared by dissolving these three neonicotinoids in DI water, tap water, and humic acid-containing ID water, respectively. The concentrations of the neonicotinoids in the samples were all 100 µg/L. For each exposure period, triplicate samples were put under natural sunlight and triplicate control samples were wrapped by aluminum foil and placed in a dark place. Glass beakers containing 50 mL sample solutions were placed outside with the sunlight directly shining into the solutions through the openings of the beakers. The samples were collected at the end of the exposure periods of 1, 2, 4, 6, 8, 12, 16, and 24 h and were processed as following: each sample was transferred to a separatory funnel, then 20 mL of dichloromethane was added into the funnel to mix with the extractant. The organic fraction was separated in a round bottom flask and evaporated using a rotary evaporator (Heidolph G1, Gemany). The remaining residues were then dissolved using HPLC grade methanol and transferred to a 2 mL amber vial for HPLC analysis. The average recovery was 90.3 ± 2.1 for nitenpyram, 92.5 ± 2.3% for thiacloprid, and 95.1 ± 0.9% % for acetamiprid, respectively.

Table 2 Intensities of visible, UVA, and UVB lights of three light sources

Light source	Visible (W/m^2)	UVA (W/m^2)	UVB (W/m^2)
Sunlight	113–141	17.6–18.1	0.019–0.036
UVA	0	14.5	0
UVB	0	0	0.058

Photodegradation Test in Soil

Each of the stock solutions of nitenpyram, thiacloprid, and acetamiprid was diluted with extra HPLC grade methanol and then mixed with the soil to achieve a neonicotinoid concentration of 100 mg/kg on dry soil basis. The spiked soil samples were then put in a fume hood for 2 h for the evaporation of the extra solvent. To make an exposure test sample, 10 g of the soil spiked with neonicotinoid was evenly spread into an approximately 5 mm thick layer in a petri plate. This thickness was used because previous studies reported that light rarely penetrates more than 4–5 mm through soil (Tester and Morris 1987). Triplicate samples were used for each exposure period and each light source. The samples were exposed to three different light sources (sunlight, UVA, UVB) for 1, 2, 4, 6, 8, 12, 16, 24, 48, 96, and 168 h. After each exposure period, the soil samples were transferred into centrifuge tubes and extraction solution (20% acetonitrile and 20%water) was used to extract the neonicotinoids from the soil samples. The extraction was performed by fixing the centrifuge tubes on a shaker and agitating them at 200 rpm for 30 min. After agitation, the tubes were placed in a centrifuge and spun at 4500 rpm for 20 min. The supernatant in each of the tubes was collected and passed through a 0.45 µm filter paper. For the same sample, the extraction process was repeated three times to obtain a necessary recovery. The extraction recoveries of nitenpyram, thiacloprid, and acetamiprid were found to be 89.3 ± 3.5%, 94.1 ± 1.3%, and 93.3 ± 1.8% respectively. The combined supernatants from the three extractions were concentrated using the rotary evaporator. The evaporation residue was dissolved in methanol and transferred into 2 mL amber glass vials, then stored in a refrigerator for further analysis of the neonicotinoids and degradation products.

Sample Analysis

The liquid samples from all photodegradation tests were analyzed using a HPLC system equipped with a Phenomenex-Diamonsil 5 µm, C_{18} column. The insecticide was separated at a flow rate 0.6 mL/min with a mobile phase of 35% water with 0.1% formic acid and 65% methanol. The absorbance wavelength was set at 254 nm. The sample injection volume was 10 µL, and all insecticides were detected within 10 min. The retention times were 4.2 min for nitenpyram, 8.0 min for thiacloprid, and 6.7 min for acetamiprid.

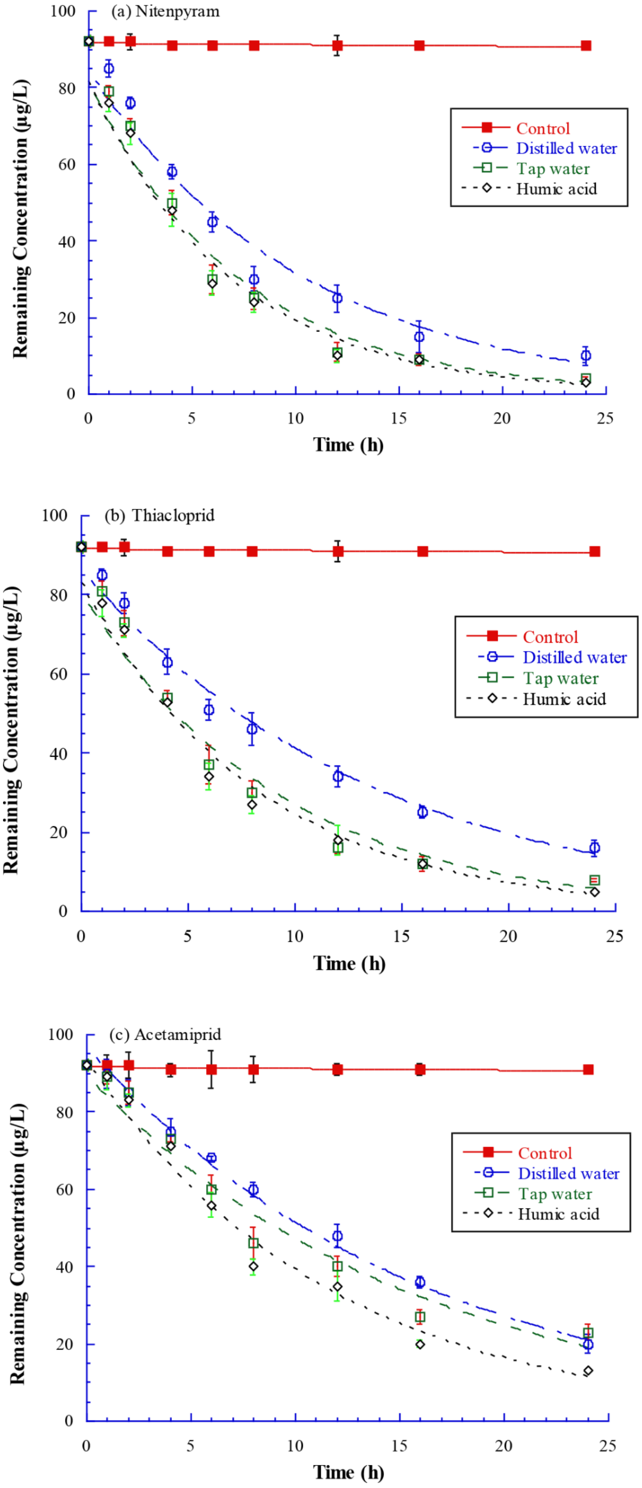

Fig. 2 Degradation of (**a**) Nitenpyram, (**b**) Thiacloprid, and (**c**) Acetamiprid in different water solutions under sunlight

The possible degradation products were identified by using a triple quad LC-MS (Triple Quad LC/MS 6420, Agilent Technology). The LC-MS system was equipped with an Eclipse Plus C_{18} column of 50×2.1 mm and particle size 1.8 µm. The mobile phase A and B used in this study were 0.1% ammonium formate and 20% methanol, respectively. The flow rate was maintained at 0.25 mL/min. The sample injection volume was 10 µL. The spectrometric scans were conducted from m/z 100 to 500 in positive mode.

Results and Discussion

Photodegradation in Waters

The photodegradation results of the three neonicotinoids in three different water solutions under the natural sunlight were presented in Fig. 2. The photodegradation of these insecticides in the water phase can be well fitted using the first-order reaction model $C_t = C_o e^{-kt}$, where C_t and C_0 represent the remaining concentration and initial concentration, respectively, and k is the constant of degradation rate. The root-mean-square deviations (RMSE) are used to indicate the quality of the curve fittings, and these values ranged from 0.2 to 0.31, indicating relatively good fits. The zero-order, first-order, and second-order reaction models were used in the regression analysis of the degradation data, and the first-order reaction kinetics was found to be the best fit.

It can be seen in Fig. 2 that approximately 32.9% of thiacloprid, 29.1% of nitenpyram, and 13.8% of acetamiprid degraded in tap water in the first 2 h under natural sunlight. The highest degradation rate was found in humic acid-containing water, the second in the tap water, and the least in the DI water. The tap water normally includes different organic and inorganic materials, which may facilitate the photodegradation rate through quenching effects. Zeng and Arnold (2013) investigated the effects of dissolved organic matters on the photodegradation of 16 different pesticides, and found that the organics can act as photosensitizers to promote an indirect photodegradation. Burrows et al. (2002) reported that under some specific short UV wavelengths the organics in water could reduce the rate of photodegradation of pesticide carbofuran, which has some similar functional groups as neonicotinoids have. Studies have concluded that the presence of humic substances in water solution will facilitate the photodegradation of herbicide atrazine and

Table 3 Photodegradation data of three insecticides in various water solutions under natural sunlight

Insecticide	Distilled water		Tap water		Humic acid-containing water	
	Half-life (h)	Degradation rate coef. (h^{-1})	Half-life (h)	Degradation rate coef. (h^{-1})	Half-life (h)	Degradation rate coef. (h^{-1})
Nitenpyram	5.4	0.13	3.7	0.19	3.6	0.19
Thiacloprid	6.3	0.11	4.7	0.15	3.7	0.19
Acetamiprid	9.1	0.076	8.9	0.078	6.5	0.11

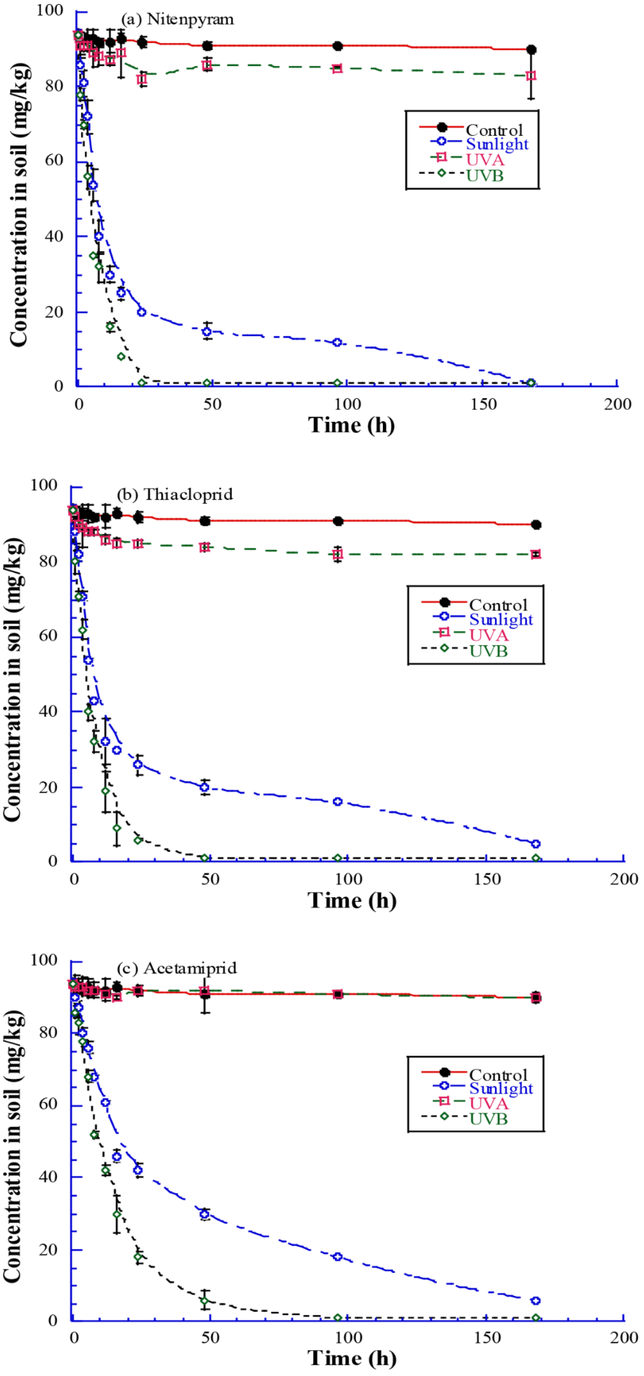

Fig. 3 Photodegradation of three neonicotinoids in TCB soil under three different light sources

fungicide carboxin (Hustert et al. 1999; Prosen and Zupancic-Kralj 2005). Similar findings are confirmed in this study. The results also indicate that hydrolysis plays very little role in the degradations of these neonicotinoids because the concentrations of them in the controls decreased less than 3% over the test periods.

Table 3 summarizes the photodegradation half-lives ($t_{1/2}$) and degradation rate coefficient (k) of the three insecticides in three different water solutions under natural sunlight. The half-lives were calculated using the equation $t_{1/2} = 0.693/k$. The order of degradation rate is nitenpyram > thiacloprid > acetamiprid. For nitenpyram, the average half-lives under sunlight were 5.4, 3.7, and 3.6 h in DI water, tap water, and humic acid-containing water respectively; for thiacloprid, 6.3, 4.7, and 3.3 h respectively, and for acetamiprid, 9.1, 8.9, and 6.5 h respectively. Acero et al. (2019) investigated the photodegradation of neonicotinoids by monochromatic UV irradiation (254 nm). They found that the degradation rates of the five selected insecticides was in the order thiamethoxam > clothianidin > imidacloprid > thiacloprid > acetamiprid. This agrees with what this study found. These data in this study suggest that nitenpyram, thiacloprid, and acetamiprid are quick to degrade in water bodies which are subjected to natural sunlight.

Photodegradation in Soil

The photodegradations of nitenpyram, thiacloprid, and acetamiprid in TCB soil under natural sunlight, UVA light, and UVB light are shown in Fig. 3. Since the photodegradation mechanisms in soil are more complicated than in water, the curves cannot be fitted well with any of the basic reaction kinematics. The radiation can be absorbed directly by the insecticides and cause the direct photodegradation. It can also be absorbed by the soil substances and the energy is transferred to insecticides and initiate indirect photodegradations. The direct photodegradation mainly happens in the surface layer (0.2–0.4 mm), while the indirect photodegradation can occur deeper in the soil, up to 10 mm (Herbert and Miller 1990). Instead of fitting the curves with models, the data points were connected with smoothed curves and half-lives and DT90 were directly read by finding the times at the horizontal axis that corresponded to 50% and 90% of initial concentrations on the curves. The results are presented in Table 4.

Table 4 Half-lives and DT90[a] of three neonicotinoids in TCB soil under natural sunlight, UVA light, and UVB light

Neonicotinoid	Sunlight		UVA light		UVB light	
	Half-life (h)	DT90 (h)	Half-life (h)	DT90 (h)	Half-life (h)	DT90 (h)
Nitenpyram	7.5	101	ND[b]	ND	4.8	15.3
Thiacloprid	7.9	135	ND	ND	5.3	15.6
Acetamiprid	15.9	152	ND	ND	10.5	41.0

[a] DT90: The time required for the insecticides concentration to decline to 10% of the initial value

[b] ND: no data available

From the data in Fig. 3 and Table 4, it can be seen that the effect of UVA on the degradations of these neonicotinoids was negligible and the degradation curves under UVA are very near to the those of the controls. In contract, UVB and natural sunlight caused significant degradations of the neonicotinoids in the soil. Studies have indicated that UV lights with wavelength between 250 and 400 nm are the major contributors to the degradation of the chemical structures in pesticides (Katagi 2004). Since the natural sunlight mainly consists of visible light, UVA and UVB, the main cause of the degradation of the three neonicotinoids was UVB light.

The degradation rates of nitenpyram and thiacloprid were approximately the same, but that of acetamiprid was significantly lower. The half-lives of nitenpyram and thiacloprid under UVB light were 4.8 and 5.3 h respectively, and that of acetamiprid was 10.5 h. Under the sunlight, the half-lives of nitenpyram and thiacloprid were 7.5 and 7.9 h respectively, and that of acetamiprid was 15.9 h. The intensity of UVB in sunlight was nearly 50% of that from the UVB lamps (Table 2), thus the half-lives of these neonicotinoids under sunlight were longer. This again indicates that the UVB light played a major role in the photodegradation of these insecticides.

Comparing the degradation data of the neonicotinoids in water solutions under natural sunlight (Table 3), the degradation rates of these neonicotinoids in soil were slower (half-lives longer). The lower degradation rates were very likely due to the fact that the soil blocked the light from penetrating into the samples and only the neonicotinoids on the surface layer of the soil were directed exposed to the sunlight. Studies have confirmed that more than 90% of the solar radiation is attenuated in the top 0.4 mm of soils (Herbert and Miller 1990). This indicates that the neonicotinoids in the top soils from foliar spray will degrade much faster than those beneath the surface from neonicotinoid-dressed seeds.

The DT 90 data for in Table 4 indicate that in less than 7 d, these neonicotinoids will be degraded to less than 10% of their initial concentrations in top soil layer under sunlight. Therefore, the neonicotinoids fallen on the soil surface from foliar spray application will undergo photodegradation rapidly. Comparing to the degradation of these neonicotinoids in soils without light exposure as report by Li et al. (2023), the degradation rates under sunlight are more than 100 times faster. The results also suggest that other methods of neonicotinoid application to crops, such as soil drench and seed dressing, make the neonicotinoids degrade much slower because of the lack of exposure to sunlight.

Degradation Pathways

To decipher the degradation pathways of the three insecticides, LC/MS was used to identify their possible photodegradation products. The extractants from neonicotinoid-spiked soils after being exposed to natural sunlight were analyzed for degradation products. Four possible degradation products A, B, C, and D from nitenpyram were identified as shown in Fig. 4, two degradation products D and E from thiacloprid as shown in Fig. 5, and three degradation products F, G, H from acetamiprid as shown in Fig. 6. It appears that the nitroguanidine group in these three neonicotinoids is unstable and easy to breakdown under photo radiation. This is in agreement with Davis and Rosenquist's (1937) finding that the nitroguanidine group is unstable and prone to degrade into more stable structures such as acid, urea, and amine. Elumalai et al. (2022) reported the results of photodegradation of thiacloprid and concluded that the nitroguanidine group was where the breakdown happened. In contrast, the chloropyridine ring in all the three insecticides was found to be very stable under sunlight. The degradation products A through H containing the nitroguanidine group may still have adverse effects on the soil environment. The further degradations and toxicities of these degradation products need to be further studied.

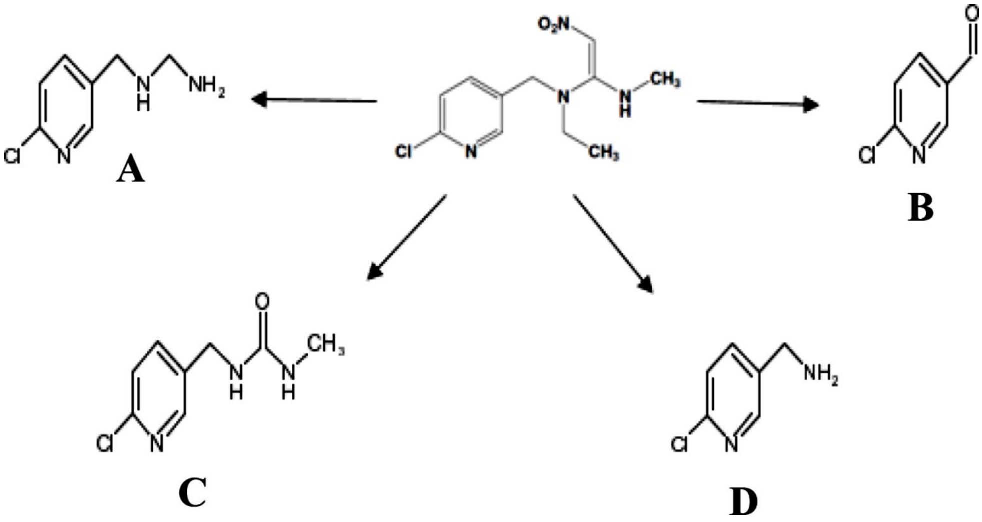

Fig. 4 Degradation pathway of nitenpyram in TCB soil under sunlight

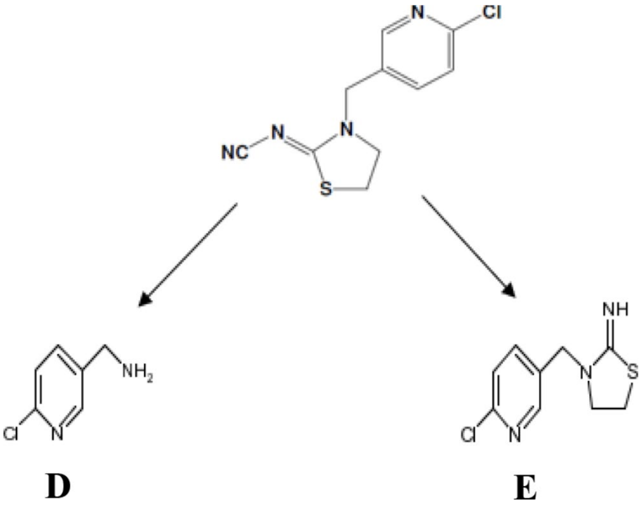

Fig. 5 Degradation pathway of thiacloprid in TCB soil under sunlight

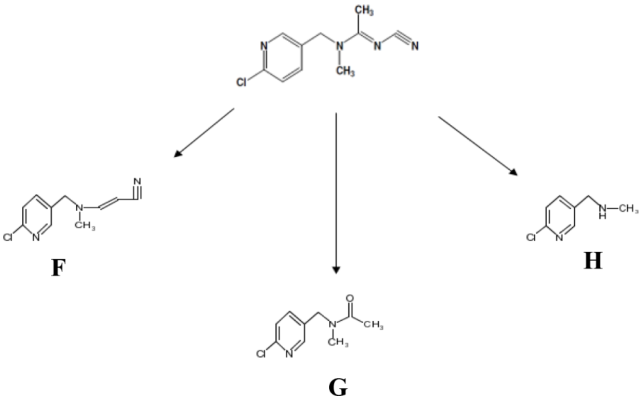

Fig. 6 Degradation pathway of acetamiprid in TCB soil under sunlight

Conclusions

The insecticides nitenpyram, thiacloprid, and acetamiprid were found to be quick to degrade in water and soil under the radiation of sunlight. UVB light in the sunlight is the main cause of the degradations and the effect of UVA light is negligible. It was also found that the degradations of these neonicotinoids degrade faster in water than in soil because of the difference in penetration capabilities of light in water and in soil. In soil, the degradations only happen on the shallow surface layer. The half-lives of nitenpyram, thiacloprid, and acetamiprid in three different water solutions under sunlight range 3.6–5.4, 3.7–6.3, and 6.5–9.1 h, respectively. The degradation rates of the neonicotinoids vary with the mediums that carry them. They degrade fastest in the humic acid-containing water, slowest in the DI water, and medium in tap water. The half-lives of nitenpyram, thiacloprid, and acetamiprid in the soil under the sunlight were found be to 7.5, 7.9 and 15.9 h, respectively. The degradation of acetamiprid is slower in both water and soil compared with the other two neonicotinoids. However, it can still degrade to less than 10% of its initial concentration in top soil layer in less than 7 d. Therefore, the neonicotinoids fallen on the soil surface from foliar spray application will be degraded under the sunlight in a short period. The analysis on the degradation pathways of these neonicotinoids suggest that the nitroguanidine group is unstable and is easy to break under photo radiation. The eight detected degradation products all contained nitroguanidine group. These products may still have adverse effects on environmental health. Their toxicities and further degradations in the environment are yet to be studied.

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Declarations

Competing Interests The authors declare that no competing interests directly or indirectly related to the work submitted here for publication.

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