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Degradation of Bentazone, Bispyribac-Sodium, Dinotefuran, and Imidacloprid in Different Types of Agricultural Soils under Laboratory Conditions by High Performance Liquid Chromatography

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Abstract: The degradation and persistence of two herbicides (bentazone-BTZ and bispyribac-sodium-BPS), and two insecticides (dinotefuran-DNF and imidacloprid-IMI) in alluvial soil, calcareous soil, and sandy soil were investigated following application at 100 µg/g soil at various time intervals, 0, 3, 7, 15, 30, and 60 days by HPLC. After 60 days of incubation, the percentages of BTZ, BPS, DNF, and IMI residues decreased to 7.13, 4.85, 53.71, and 7.51%, respectively, in alluvial soil; 8.58, 5.28, 17.68, and 12.56%, respectively in calcareous soil; and 10.53, 8.94, 53.09 and 13.84%, respectively in sandy soil of the initial residue. Compared with BTZ and BPS, DNF and IMI dissipated more slowly into the three soils. The dissipation of the tested pesticides was faster in the alluvial soil, followed by the calcareous soil, and then the sandy soil, except for DNF. The different orders of kinetics showed that the first-order model fit the experimental persistence data quite well with high R2 values and low Δqe% and SSE values. The half-lives were 15.27, 16.39, and 17.82 days for BTZ; 11.73, 13.10, and 16.20 days for BPS; 63.01, 23.42, and 75.34 days for DNF; and 14.87, 20.39, and 14.87 days for IMI in the alluvial soil, calcareous soil, and sandy soil, respectively.

Keywords: bentazone; bispyribac-sodium; dinotefuran; imidacloprid; half-life; HPLC; soil.

1. Introduction

Herbicide use is highly important in modern agriculture because it can kill or suppress the growth of vegetation and unwanted plants, thereby increasing the quantity and quality of crops [1-3]. Numerous mechanisms, including degradation, sorption, leaching, and plant uptake, as well as the impact of variables like crop type, herbicide application rate, climate, and agricultural methods, determine the destiny of herbicides sprayed to soil [4-8].

Bentazone (BTZ) is a selective contact (foliar) herbicide for the post-emergence control of annual broadleaf weeds and yellow nutsedges in a variety of crops, including alfalfa, soybeans, beans, peas, corn, sorghum, and peppers [9-13]. It also has activity on some perennial broadleaf weeds, such as Canada thistle and field bindweed [11,14,15]. It is considered one of

the most widely used herbicides in agriculture worldwide [13,15]. It is generally applied at rates of 1.0 to 2.2 kg/ha [9]. It is a weak acid that primarily exists in anionic form and functions as an inhibitor of photosynthetic electron transport [10,13]. Due to its widespread use, persistence, and propensity to leach from soil into groundwater, the herbicide BTZ raises environmental issues [16]. Weakly adsorbed in soil, it is primarily broken down by microbes [12]. In topsoils, this compound degrades relatively quickly [15]. According to reports on BTZ degradation, seven kinds of degradation products can be generated: N-methyl bentazone, 6-OH bentazone, 8-OH bentazone, 3-isopropenyl-2,2-dioxo-1H-2,1,3-benzothiadiazin-4-one, 2,2-dioxo-1H-2,1,3-benzothiadiazin-4-one, 3-(2-hydroxy-1-methyl-1-ethyl)-2,2-dioxo-1H-2,1,3-benzothiadiazin-4-one, and 2-amino-N-isopropylbenzamide [13,17]. N-methyl-bentazone is the most stable metabolite of BTZ in soil [18].

Bispyribac-sodium (BPS) is a highly effective and wide-spectrum selective pyrimidinyl carboxy herbicide. It was developed by Kumiai Chemical, Japan, for post-emergence control of cyperaceous and dicotyledonous grasses in rice [1,3,8,19-24]. It moves throughout the plant after being absorbed through the leaf surface [1,3,22]. Its primary method of action is to prevent acetolactate synthase (also known as acetohydroxy acid synthase) from doing its job, which impacts the production of branched-chain amino acids, an enzyme essential to plant growth [1,3,8,19,22]. Due to its high solubility in water and short half-life in soil, it is widely used in agriculture. Even at low quantities, it is a hazardous and enduring herbicide [19].

Worldwide, one of the most popular groups of pesticides, neonicotinoids, now account for more than one-quarter of the global pesticide market, and their annual production is approximately 600,000 tons [25-27]. Neonicotinoids have been developed since the early 1990s for nicotine structure research with the goal of boosting the effectiveness of these pesticides at low application rates while also enhancing their bioavailability and hastening their uptake by plants to replace older chemicals such as organochlorine, chlorinated hydrocarbons, organophosphorus, carbamate, pyrethroid, and several other chemical categories, which are more harmful to humans and the environment [26,28-32]. There are more than 290 crops on which neonicotinoids are approved for use in more than 120 countries [33] Because of their distinctive qualities, such as broad spectrum activity, low application rate, systemic and translaminar activity, significant residual activity, and distinct mode of action, these neonicotinoids have become widely accepted as a crucial part of integrated pest control strategies [34]. Although neonicotinoids better control soil-borne insects and piercing-sucking pests, information on their environmental persistence and potential ecological impacts is still inadequate [20,26,32]. Despite being effective insecticides with low toxicity, neonicotinoids have caused serious environmental concerns due to their long-term use [26].

The most recent generation of neonicotinoids, dinotefuran (DNF), has been widely employed to combat biting and sucking insects because of its strong insecticidal action, quick absorption, plant translocation, and environmental and human safety [27,35-37]. Currently, DNF is marketed and used in a variety of ways throughout the world [37]. Due to its extensive use, DNF will eventually leak into the environment. With a half-life of 50–100 days in the soil environment, DNF is relatively stable despite being safe for both people and the environment [37-40]. However, because DNF is a relatively new insecticide, a small amount of risk information is available. The primary processes via which DNF degrades in the environment are photolysis and microbiological degradation. 1-Methyl-3-(tetrahydro-3-furylmethyl) guanidium dihydrogen and 1-methyl-3-(tetrahydro-3-furylmethyl) urea are the main degradation products of DNF [37].

Approximately 120 nations have registered products containing imidacloprid (IMI) for use on more than 140 agricultural crops [41]. IMI is thought to be a somewhat polar substance with strong xylem mobility, making it appropriate for soil application and seed treatment [41-43]. It can be applied through tree injection, soil injection, topical application, foliar, broadcast, ground application as a liquid formulation, or granular, or as a seed treatment [42]. Depending on the soil type, pH, application of organic fertilizers, and presence or lack of ground cover, IMI may persist in the soil [41]. The primary IMI breakdown products in soil are 6-hydroxynicotinic acid, 6-chloronicotinic acid, and imidacloprid urea, and the final product is CO_2 [41].

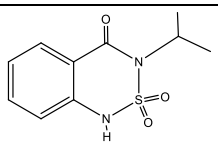
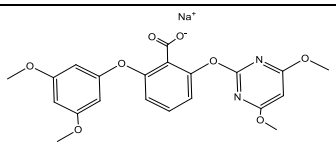
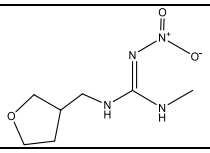
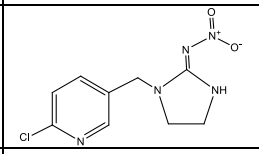
Few studies have been conducted on the environmental persistence of BTZ, BPS, DNF, and IMI in post-application settings, according to a review of the literature [32,44]. Therefore, the main objective of this research was to investigate the degradation kinetics of the tested pesticides in alluvial soil, calcareous soil, and sandy soil to provide more data for understanding the pest-controlling (weeds and insects) effect in soil and evaluating their half-lives in different soils.

2. Materials and Methods

2.1. Pesticides.

Two herbicides, BTZ and BPS, and two insecticides, DNF and IMI, were tested. The physicochemical characteristics of the tested pesticides are summarized in Table 1.

Table 1. Physicochemical characteristics of the tested pesticides.

Pesticides	Bentazone	Bispyribac-sodium	Dinotefuran	Imidacloprid
Structure				
Type	Herbicide	Herbicide	Insecticide	Insecticide
Chemical family	Thiadiazine	Pyrimidinylthiobenzoate	Neonicotinoid	Neonicotinoid
IUPAC name	2,2-dioxo-3-propan-2-yl-1*H*-2λ^6,1,3-benzothiadiazin-4-one	sodium 2,6-bis[(4,6-dimethoxypyrimidin-2-yl)oxy]benzoate	2-methyl-1-nitro-3-[(tetrahydro-3-furanyl) methyl] guanidine	*N*-{1-[(6-Chloro-3-pyridyl)methyl]-4,5-dihydroimidazol-2-yl}nitramide
CAS Number	25057-89-0	125401-75-4	165252-70-0	138261-41-3
Formula	$C_{10}H_{12}N_2O_3S$	$C_{19}H_{17}N_4NaO_8$	$C_7H_{14}N_4O_3$	$C_9H_{10}ClN_5O_2$
Water solubility (g/L)	0.50	733	39.83	0.51
Tested material	Technical 99.0% a.i.	Technical 96.0% a.i.	Technical 97.0% a.i.	Technical 95.0% a.i.
Supplied company	Kafr El-Zayat Pesticide Co.	AgroChem Co.	Egyptian Chemical Industries Co.	Chem Galaxy Co.

2.2. Soils.

The soils used in this investigation were collected from three different agricultural locations in Egypt. Soil samples were taken from the top 35cm. The collected soils were air-dried in a laboratory at 23°C for seven days, after which they were crushed and passed through a sieve (2mm) [45-46]. The physicochemical properties of the selected soils are summarized in Table 2.

Table 2. Physicochemical properties of the soils used in this study.

Properties	Alluvial soil	Calcareous soil	Sandy soil
Texture class	Clay loam	Sandy loam	Sand
Coordinates (N/E)	311006/295804	304835/294536	173085/3367732
Water holding capacity (mL)	45	34	32
pH	8.3	8.2	8.5
EC (ds/m)	1.3	2.4	1.2
Organic matter content (%)	3.5	1.3	0.2
Total carbonate (%)	7.9	40.1	39.0
Total soluble cations (meq/L)	18.8	33.5	17.4
Total soluble anions (meq/L)	13.3	23.3	15.5

2.3. Chemicals and solvents.

Sodium chloride, sodium acetate, anhydrous magnesium sulfate, formic acid, acetone, acetonitrile, methanol, and dimethylformamide were purchased from Algomhoria Chemical Co., Alexandria, Egypt. Water (HPLC grade), methanol (HPLC grade), and acetonitrile (HPLC grade) were purchased from Sigma Aldrich Co. (Spruce Street, Louis, MO, USA) [47].

2.4. Stock solution.

Stock solutions of BTZ, BPS, DNF, and IMI (µg/mL) were prepared in acetonitrile of HPLC grade. The working solution was diluted in HPLC-grade acetonitrile and HPLC-grade methanol at a ratio of 1:9 to reach the required final concentration (µg/mL). Prior to use, the standard solutions were kept at 4°C.

2.5. Application of the tested pesticides to soils.

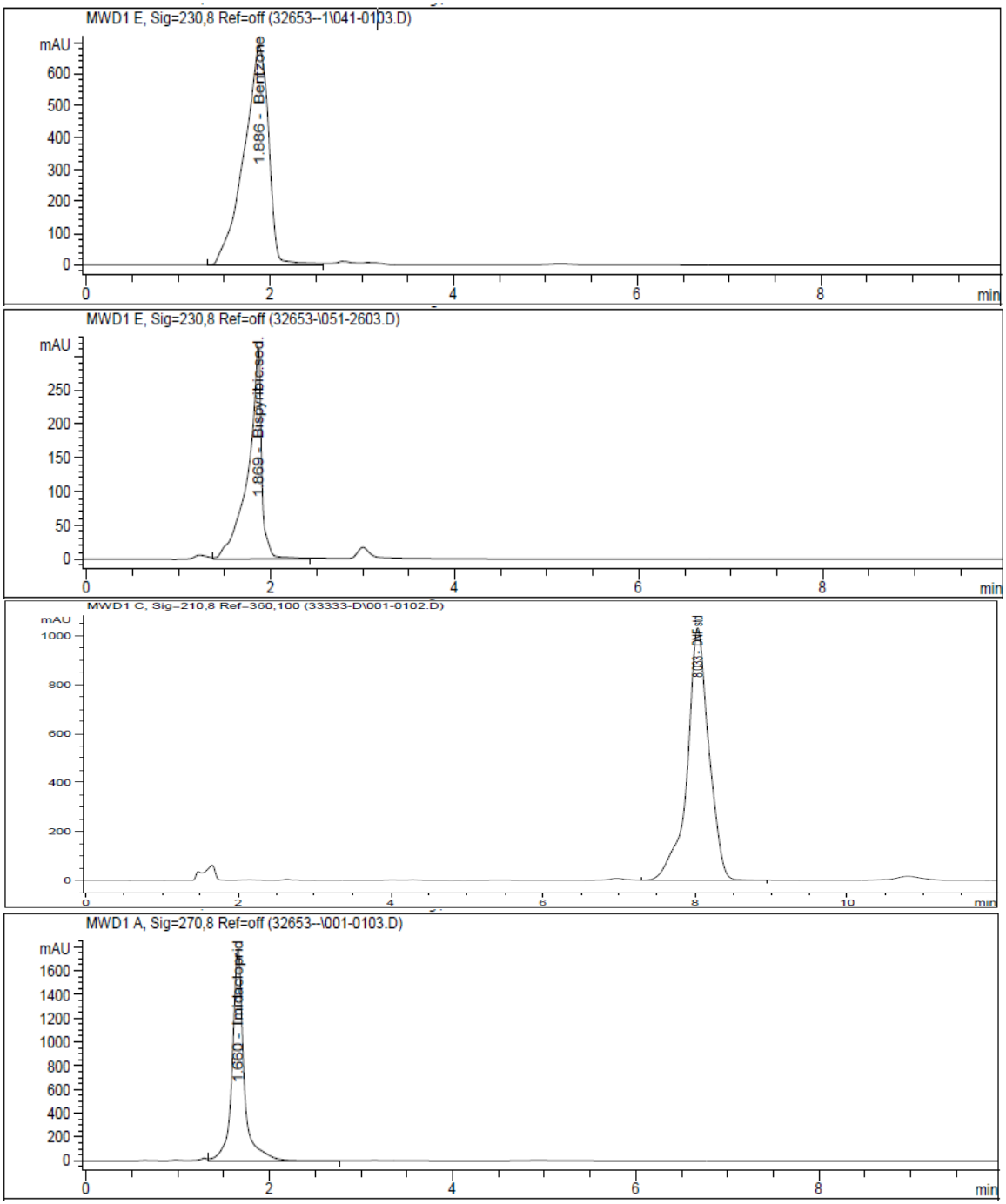
BTZ, BPS, DNF, and IMI were applied at a concentration of 100 µg a.i./g soil. Three replicates were made for each soil type, and the volume of each replicate was 300 g. The stock of pesticide was mixed with distilled water equal to 60% of the water-holding capacity of the soil. The solution was added to the soil to a final concentration of 100 µg/g soil. All treatments were incubated throughout the experimental period at room temperature.

2.6. Extraction and cleanup.

At various intervals of 0, 3, 7, 15, 30, and 60 days, 10 g of the soil sample was taken into 25-mL centrifuge tubes. The soil samples were ground with a mixture of salts (composed of 0.15 g of sodium chloride, 0.35 g of sodium acetate, and 0.50 g of anhydrous magnesium sulfate) in a mortar for 3 min to induce phase separation and insecticide partitioning. The samples were placed in brown bottles and extracted with 20 ml of 0.1% formic acid: methanol: dimethylformamide (5:2:2:1) in acetone: acetone: acetonitrile. The samples were placed in glass tubes and shaken for 25 min in a water bath at 36°C. The tube was centrifuged at 4,000 rpm for 5 min and filtered using Whatman filter paper. To eliminate undesirable co-extractives, the organic layer was passed through a 15-mL centrifuge tube containing 0.13 g of magnesium sulfate and 0.07 g of activated charcoal. The tube was closed, shaken by hand for 90 s, and centrifuged for 4 min at 5000 rpm. The prepared sample was filtered through a 0.22-mm nylon syringe filter and transferred to an autosampler vial prior to HPLC analysis [48].

2.7. HPLC analysis.

All the soil samples were analyzed via HPLC. Separation was performed on a C18 column (250×4.6 mm, 5 µm). The HPLC separation conditions were as follows: flow rate, 1 mL/min; injection volume, 20 µL; column temperature, 25°C; and mobile phase, water: methanol: acetonitrile (5:3:2). The retention times under these separation conditions were 1.528 for BTZ, 1.586 for BPS, 8.033 for DNF, and 2.102 min for IMI (Figure 1).



Name	RetTime (min)	Area (mAU*s)	Amt/Area	Amount (ppm)
Bentazone (BTZ)	1.886	1.28707e4	7.76961e-3	100
Bispyribac-sodium (BPS)	1.869	3233.42114	3.09270e-2	100
Dinotefuran (DNF)	8.033	2.05790e4	4.85933e-3	100
Imidacloprid (IMI)	1.660	1.59448e4	6.27165e-3	100

Figure 1. HPLC standard of the tested pesticides at 100 µg/mL.

2.8. Order kinetic models.

Six kinetic models were tested to investigate the effects of the tested pesticides on alluvial soil, calcareous soil, and sandy soil. The experimental values of the residue concentration for each pesticide in each soil were applied in the linearized equation of different models to determine the model parameters.

2.8.1. Zero order.

The zero-order equation is represented in Table 3, where $[A]_t$ = the residue amount of pesticide in the soil with time t; $[A]_0$ = the initial concentration; and k = the zero-order rate constant in units of M s^{-1}. Thus, a plot of $[A]_t$ versus t should have a linear relationship with the slope $-k$ and the intercept of $[A]_0$. The half-life of the zero-order equation is dependent on both the rate constant and the initial concentration of the reactant, as shown in the following equation: $t_{1/2} = [A]_0/2k$.

Table 3. Integrated rate equations of degradation kinetics.

Order	Differential equation	Integrated equation	Graph to be drawn	Intercept	Slope	Half-life expression
0	$-\frac{d[A]}{dt} = k[A]^0$	$[A]_t = [A]_0 - kt$	$[A]_t\ vs.t$	$[A]_0$	$-k$	$t_{1/2} = \frac{[A]_0}{2k}$
1/2	$-\frac{d[A]}{dt} = k[A]^{1/2}$	$[A]_t^{1/2} = [A]_0^{1/2} - \frac{1}{2}kt$	$[A]_t^{1/2}\ vs.t$	$[A]_0^{1/2}$	$-\frac{1}{2}k$	$t_{1/2} = \frac{(2-\sqrt{2})[A]_0^{1/2}}{k}$
1	$-\frac{d[A]}{dt} = k[A]^1$	$ln[A]_t = ln[A]_0 - kt$	$ln[A]_t\ vs.t$	$ln[A]_0$	$-k$	$t_{1/2} = \frac{ln2}{k}$
3/2	$-\frac{d[A]}{dt} = k[A]^{3/2}$	$\frac{1}{[A]_t^{1/2}} = \frac{1}{[A]_0^{1/2}} + \frac{1}{2}kt$	$\frac{1}{[A]_t^{1/2}}\ vs.t$	$\frac{1}{[A]_0^{1/2}}$	$+\frac{1}{2}k$	$t_{1/2} = \frac{2(\sqrt{2}-1)}{k[A]_0^{1/2}}$
2	$-\frac{d[A]}{dt} = k[A]^2$	$\frac{1}{[A]_t} = \frac{1}{[A]_0} + kt$	$\frac{1}{[A]_t}\ vs.t$	$\frac{1}{[A]_0}$	$+k$	$t_{1/2} = \frac{1}{k[A]_0}$
3	$-\frac{d[A]}{dt} = k[A]^3$	$\frac{1}{[A]_t^2} = \frac{1}{[A]_0^2} + 2kt$	$\frac{1}{[A]_t^2}\ vs.t$	$\frac{1}{[A]_0^2}$	$+2k$	$t_{1/2} = \frac{3}{2k[A]_0^2}$

2.8.2. Halve (1/2) order.

The differential equation, integrated equation, and half-life equation are shown in Table 3, where k is a 1/2 order rate constant with units of $M^{3/2}$ s^{-1}. Thus, a plot of $[A]_t^{1/2}$ versus t should have a linear relationship with the slope $-\frac{1}{2}k$ and the intercept of $[A]_0^{1/2}$.

2.8.3. First order.

The first-order equation is expressed in Table 3. The integral form can be found by:

$$[A]_t = [A]_0{}^{-kt} \quad (1)$$

where the pesticide concentration at time t = 0 is $[A]_0$, the pesticide concentration at time t is $[A]_t$ and k is constant with units of s^{-1}. The concentrations are expressed in terms of their natural logarithm using the method above:

$$ln[A]_t = ln[A]_0 - kt \quad (2)$$

2.8.4. Three-halves (3/2) order.

The nonlinear and linear forms of the 3/2 order equation are included in Table 3, where k is a constant with units of $M^{1/2}$ s^{-1}. Thus, a plot of $1/[A]_t^{1/2}$ versus t should have a linear relationship with the slope $+\frac{1}{2}k$ and the intercept of $1/[A]_0^{1/2}$.

2.8.5. Second order.

The second-order equation may be written in the form shown in Table 3, where k is a second-order rate constant with units of M^{-1} s^{-1}. Thus, a plot of $1/[A]_t$ versus t should have a linear relationship with the slope $+k$ and intercept of $1/[A]_0$.

2.8.6. Third order.

The third-order model is given in Table 3, where k is a third-order rate constant with units of M^{-2} s^{-1}. Thus, a plot of $1/[A]_t^2$ versus t should have a linear relationship with the slope $+2k$ and the intercept of $1/[A]_0^2$.

2.9. Statistical analysis.

The experimental data of the tested pesticides in the soils were analyzed using an HPLC software program and Microsoft Excel.

3. Results and Discussion

3.1. Residues and decay of the tested pesticides in soils.

The persistence of BTZ, BPS, DNF, and IMI in alluvial soil, calcareous soil, and sandy soil treated individually with 100 µg/g soil was studied under laboratory conditions. The soil incubated at 23°C was sampled at different times (0, 3, 7, 15, 30, and 60 days) and analyzed by HPLC. The residue percentages of the tested pesticides detected 60 days after application are shown in Table 4. The initial concentrations of BTZ, BPS, DNF, and IMI in the soils were 96.81, 99.31, 92.85, 99.98, and 95.57 µg/g in the alluvial soil; 95.57, 98.99, 99.90, and 99.56 µg/g in the calcareous soil; and 92.89, 98.65, 100.00, and 99.00 µg/g in the sandy soil. After 60 days of incubation, the pesticide residue concentration decreased to 6.90, 4.82, 49.87, and 7.51 µg/g in the alluvial soil (7.13, 4.85, 53.71, and 7.51% of the initial residue remaining); 8.20, 5.23, 17.66, and 12.50 µg/g in the calcareous soil (8.58, 5.28, 17.68, and 12.56% of the initial residue remaining); and 9.78, 8.82, 53.09, and 13.70 µg/g in the sandy soil (10.53, 8.94, 53.09, and 13.84% of the initial residue remaining), respectively. As in other studies, the residues of pesticides in soils decreased with time [42,48]. However, DNF and IMI dissipated slowly compared with BTZ and BPS into the three soils, as shown in Figure 2. The dissipation of DNF was faster in calcareous soil, followed by alluvial soil and sandy soil; unlike that of other pesticides, DNF dissipation was greater in alluvial soil, calcareous soil, and sandy soil. This is due to the high percentage of calcium carbonate in calcareous soil and its effect on the adsorption and degradation of DNF [44]. Previous studies have suggested that DNF can be a possible contaminant of soil [37]. Romeh et al reported that the average initial disposition of IMI in soil under tomato was 0.640 to 1.39 µg/g soil [49], which dissipated 14 days after spraying, while that under maize was 0.65 µg/g soil 30 days after sowing [50]. Residues of IMI were nondetectable in soil samples at harvest in cotton, rice, and mustard crops [51,52].

Table 4. Residue percentages of pesticides in soils at different time intervals by HPLC.

Time (day)	Alluvial soil				Calcareous soil				Sandy soil			
	BTZ	BPS	DNF	IMI	BTZ	BPS	DNF	IMI	BTZ	BPS	DNF	IMI
0	96.81	99.31	92.85	99.98	95.57	98.99	99.90	99.56	92.89	98.65	100.00	99.00
3	90.32	96.32	85.83	98.43	93.06	95.75	98.88	87.80	90.59	95.62	92.04	97.42
7	57.17	82.35	85.28	87.94	59.04	69.75	96.13	78.45	62.30	76.54	79.68	91.37
15	27.61	45.10	75.52	79.57	30.81	24.40	90.32	59.97	31.42	32.50	74.20	76.65
30	12.81	3.96	53.07	20.43	15.40	8.12	52.84	40.03	17.21	15.48	73.64	53.30
60	6.90	4.82	49.87	7.51	8.20	5.23	17.66	12.50	9.78	8.82	53.09	13.70
Mean	48.60	55.31	73.74	65.64	50.35	50.37	75.95	63.05	50.70	54.60	78.77	71.91

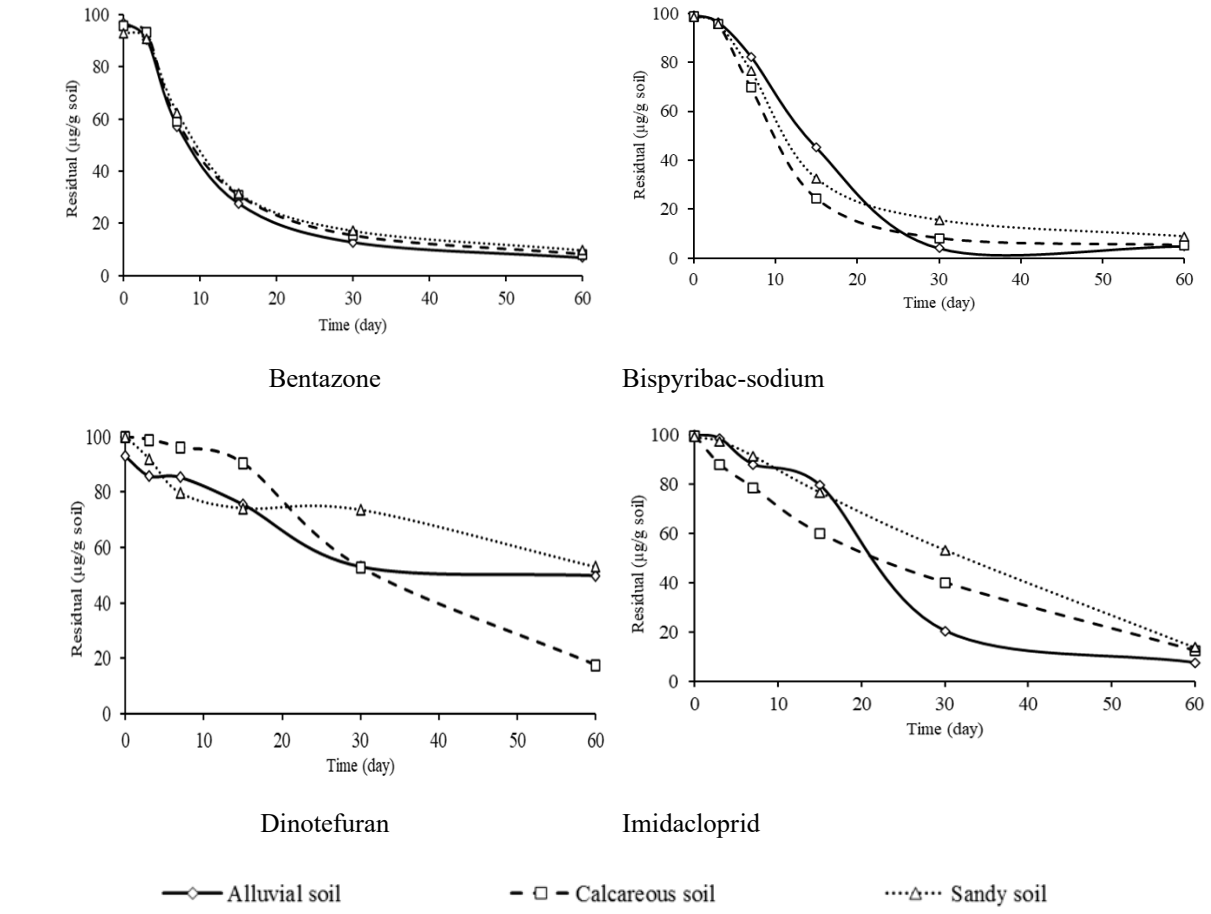
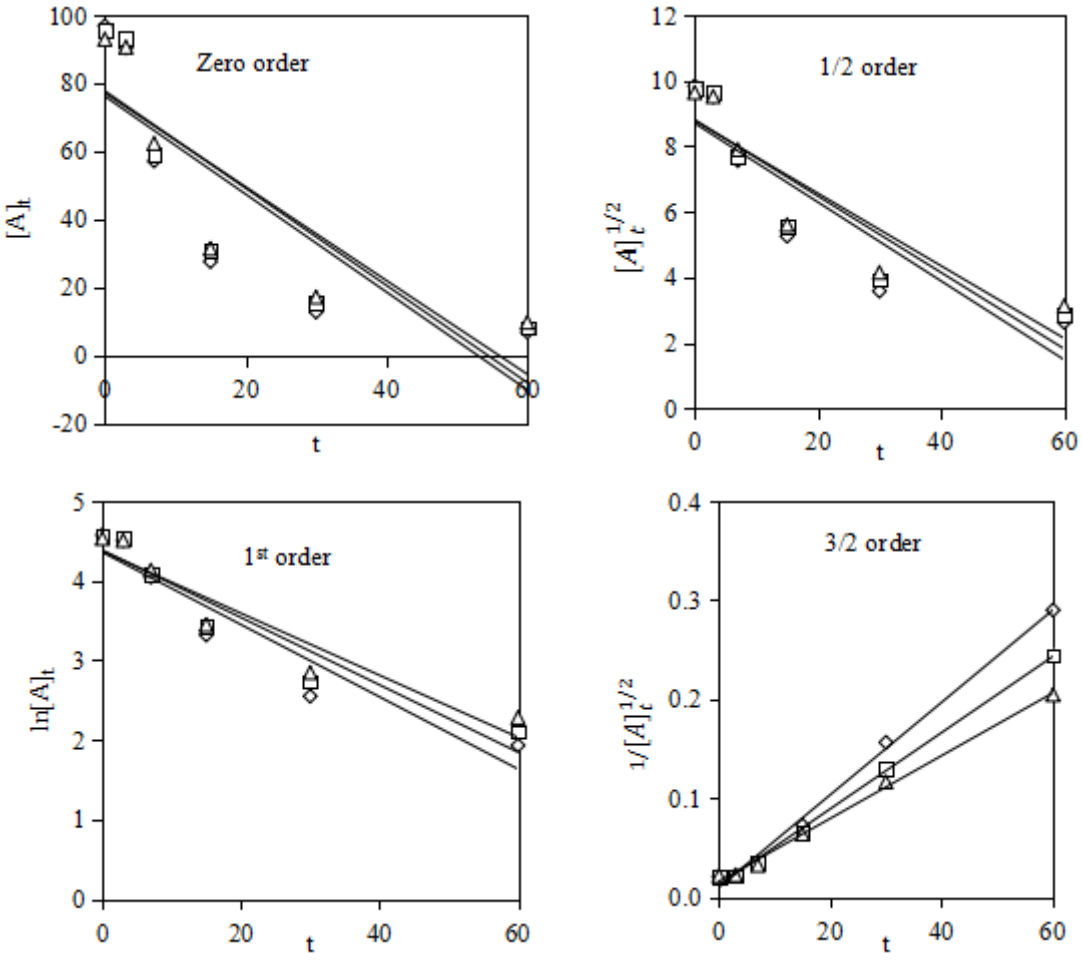


Figure 2. Residual curves of the tested pesticides in soils.

3.2. Comparison of the tested order kinetic models.

The persistence kinetics of BTZ, BPS, DNF, and IMI in alluvial soil, calcareous soil, and sandy soil were analyzed using different order kinetic models. The validity of the order models was tested based on four values: the correlation coefficient (R^2), the normalized standard deviation (Δqe%), which were used to compare the experimental and calculated data, and the summed squared error (SSE). A relatively high correlation coefficient (R^2) close to or equal to 1, low Δqe%, and low SSE indicate that the tested model successfully describes the kinetics of pesticide persistence. The order equation plots of the persistence kinetics of the tested pesticides in soils revealed that linear relationships existed between $[A]_t$ and time of zero order, $[A]_t^{1/2}$ and time of 1/2-order, $ln[A]_t$ and time of first-order, $1/[A]_t^{1/2}$ and time

of 3/2-order, $1/[A]_t$ and the time of the second-order, and $1/[A]_t^2$ and time of the third order, each soil type is represented in Figures 3, 4, 5, and 6. The order equation parameters; $[A]_0$, $[A]_0^{1/2}$, $ln[A]_0$, $1/[A]_0^{1/2}$, $1/[A]_0$, $1/[A]_0^2$, and k were calculated from the intercept and slope of the linear plots and are reported in Table 5. When comparing the k constant in the different reaction orders, a clear decrease was observed in the values obtained with increasing reaction order (i.e., an inverse relationship between k and the reaction order). The obtained k values were 0.74 to 1.71, 1.39 to 1.59, and 0.67 to 1.54 for zero-order, and 0.000005 to 0.0009, 0.000004 to 0.0002, and 0.000004 to 0.0002 for third-order in alluvial soil, calcareous soil, and sandy soil of four tested pesticides, respectively. According to the correlation coefficient (R^2) values, the first-order model is suitable for the persistence of the tested pesticides in soils because the R^2 ranges from 0.79 to 0.96 in alluvial soil, 0.87 to 1.00 in calcareous soil, and 0.91 to 0.96 in sandy soil. In contrast, the values of Δqe (%) and SSE were lower in the ranges of 4.97-36.41 and 0.50-18.71, respectively, except for those of BPS in the alluvial and calcareous soils. Additionally, when comparing the results of persistence kinetics obtained from laboratory experiments of the tested pesticides with the results calculated from the first-order equation in three soils, the results were identical.



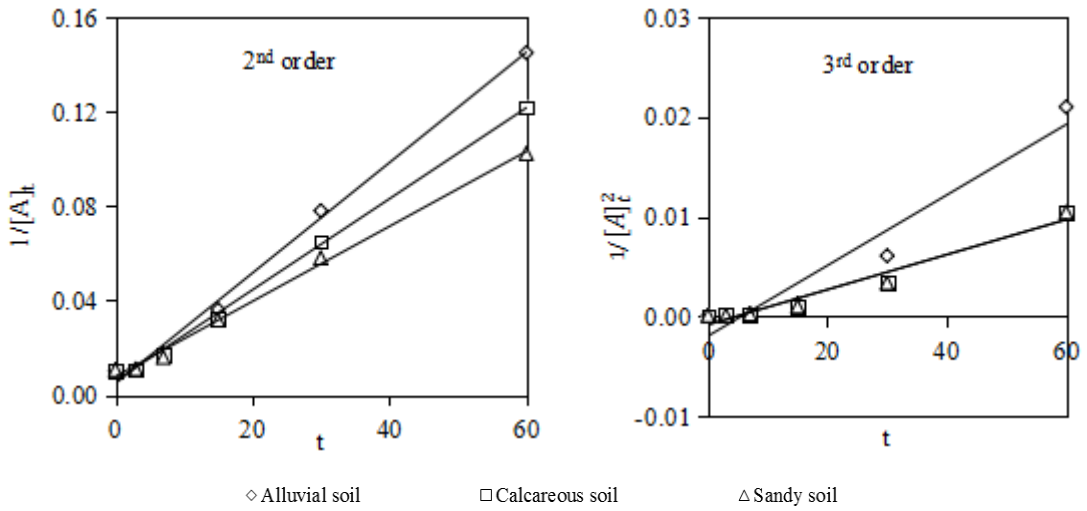
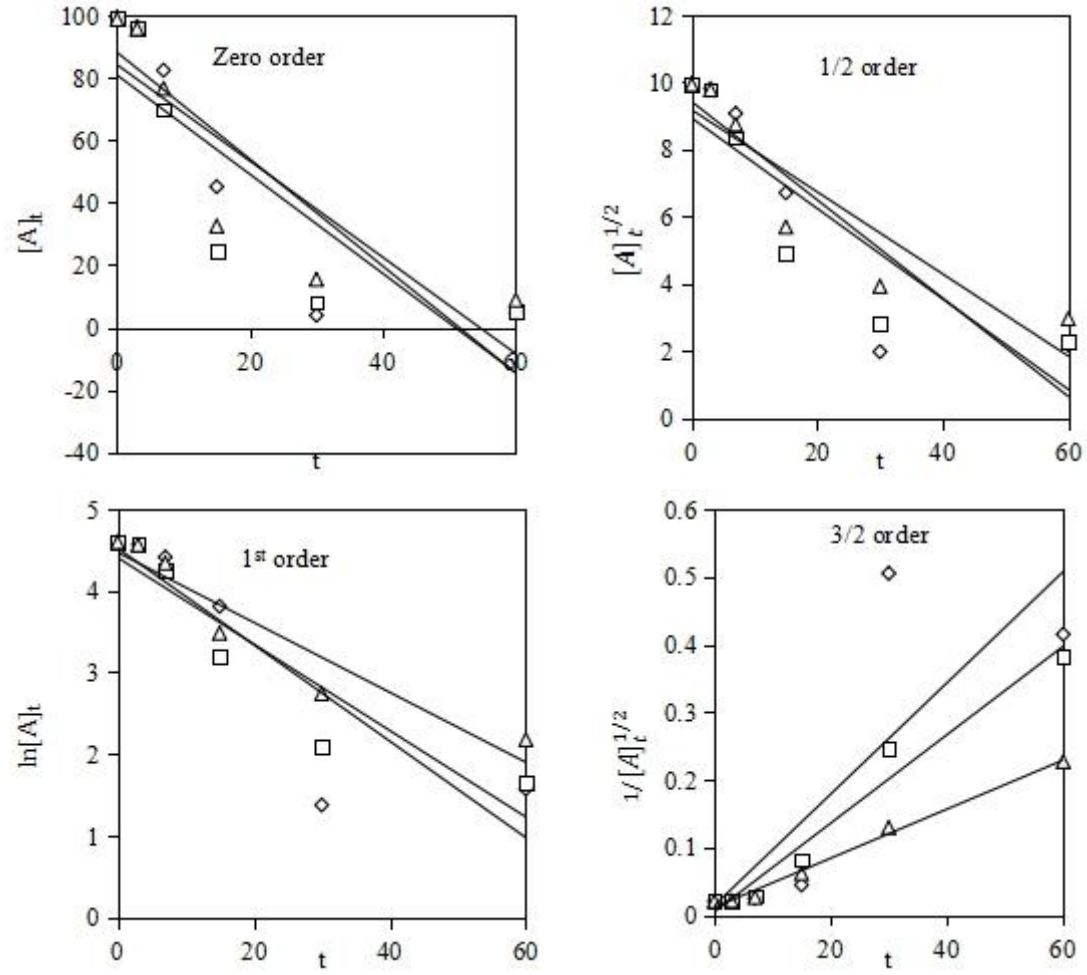


Figure 3. Plots of different orders of rate for the degradation of bentazone in soil.

In general, the results showed that the first-order model, followed by the 1/2-order kinetic model, fit the experimental persistence data quite well, with high values of R^2 and low values of Δqe% and SSE. Accordingly, the first-order equation is most suitable for describing the persistence kinetics of BTZ, BPS, DNF, and IMI in alluvial soil, calcareous soil, and sandy soil. The first-order kinetic equation is commonly used for describing the persistence kinetics of various pesticides in soil [48], and it is the best model for describing the persistence kinetics of BTZ [12,14,18], BPS [8], DNF [31,33], and IMI [31,33,41-42].



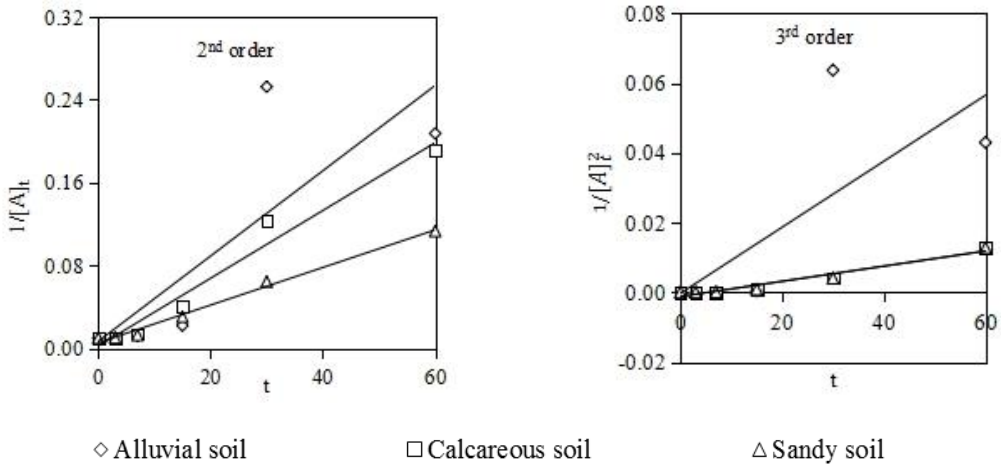


Figure 4. Plots of different orders of rate for the degradation of bispyribac-sodium in soil.

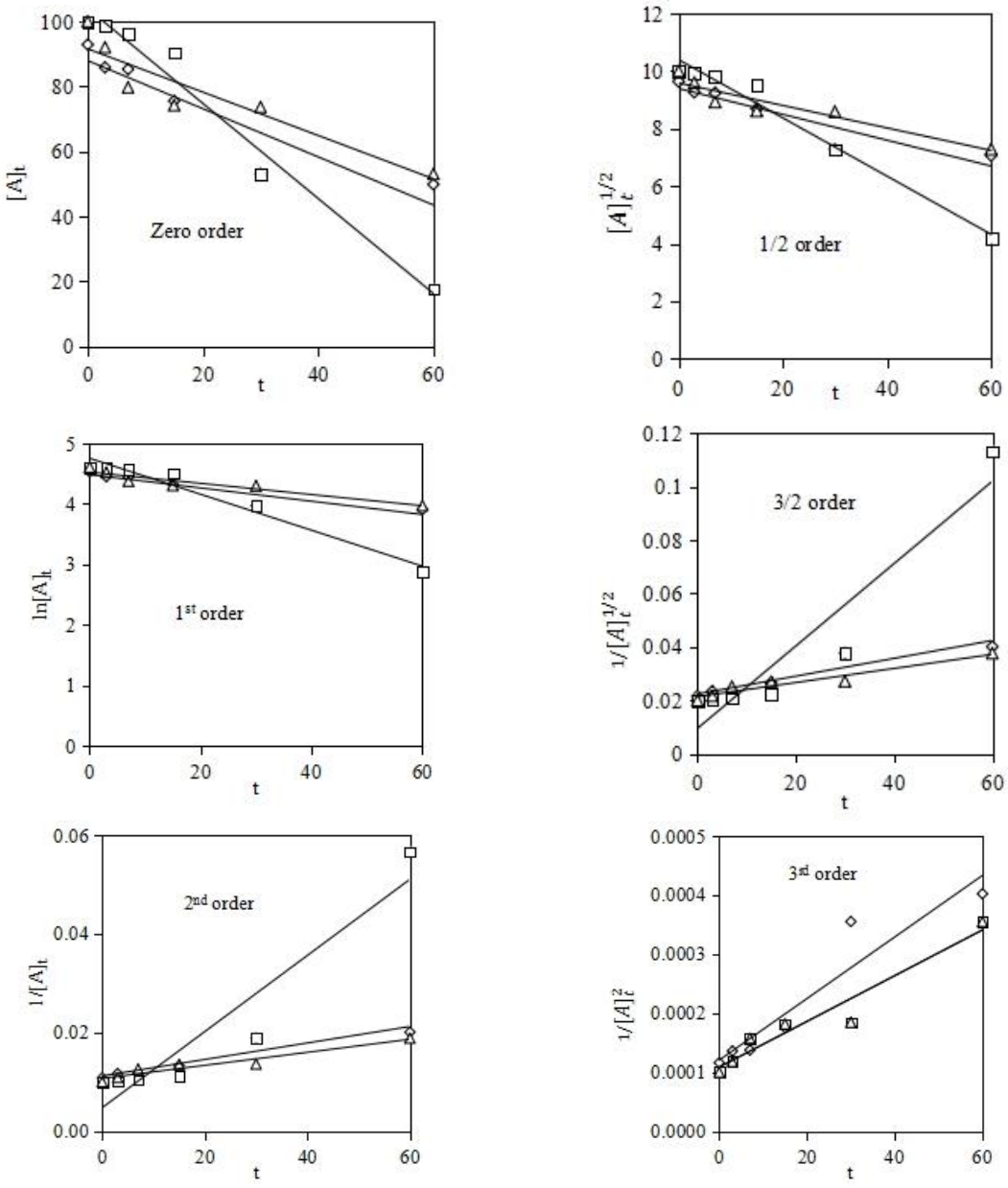


Figure 5. Plots of different orders of rate for the degradation of dinotefuran in soil.

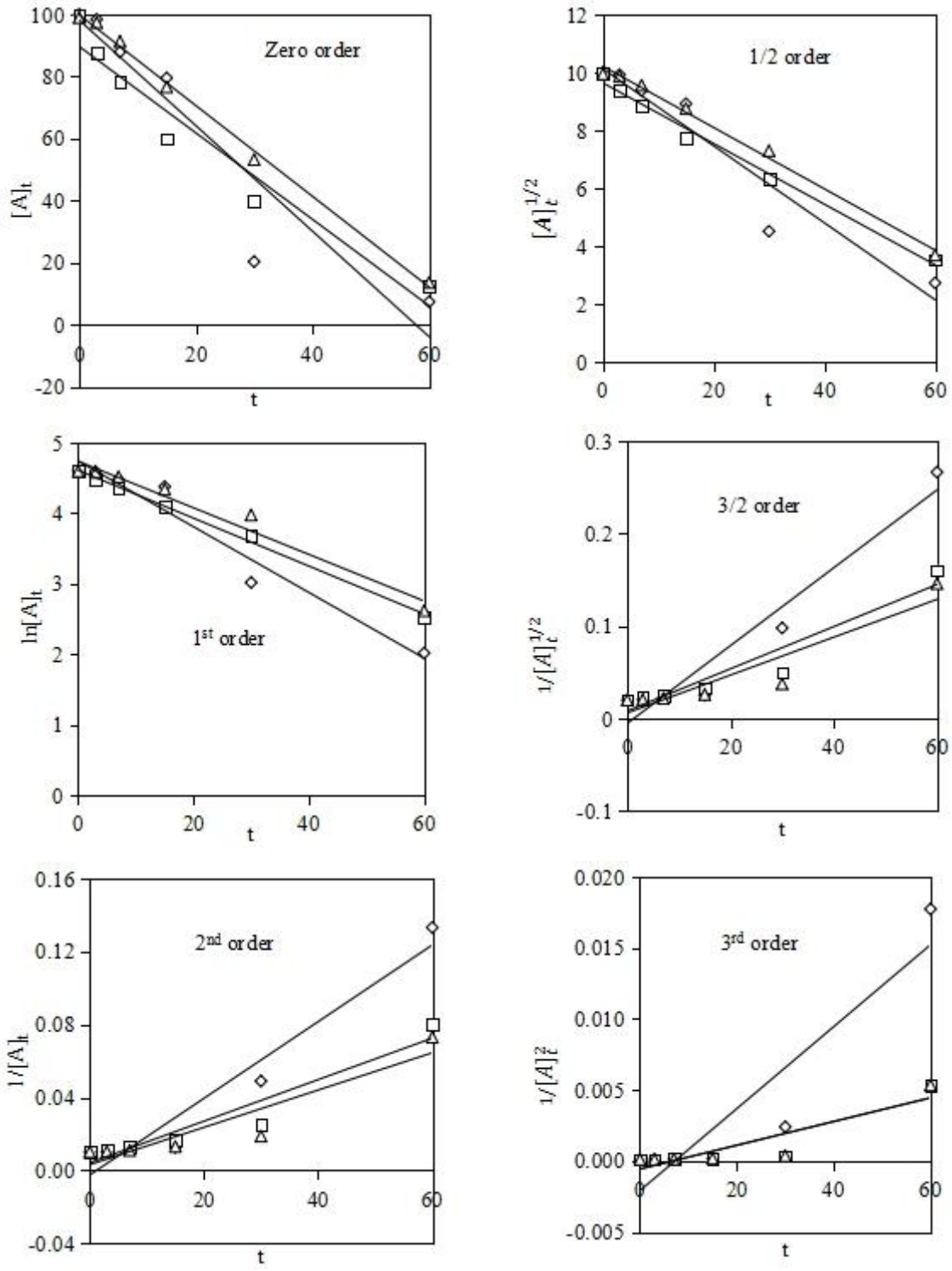


Figure 6. Plots of different order rates for the degradation of imidacloprid in soil.

Table 5. Kinetic parameters for the degradation of the tested pesticides into soils.

Order	Parameters	Alluvial soil				Calcareous soil				Sandy soil			
		BTZ	BPS	DNF	IMI	BTZ	BPS	DNF	IMI	BTZ	BPS	DNF	IMI
0	$[A]_0$	76.15	88.09	87.92	98.21	77.71	80.80	103.94	89.72	77.07	84.15	91.54	99.90
	K	1.44	1.71	0.74	1.70	1.43	1.59	1.46	1.39	1.38	1.54	0.67	1.46
	R^2	0.70	0.78	0.86	0.89	0.72	0.70	0.97	0.95	0.73	0.75	0.86	1.00
	Δqe(%)	138.65	412.30	12.41	89.13	111.90	226.03	8.42	25.42	92.82	115.83	7.41	5.44
	SEE	109.64	359.96	4.18	52.94	86.26	202.31	2.10	7.09	70.34	95.90	2.26	0.40
1/2	$[A]_0^{1/2}$	8.70	8.70	9.39	10.13	8.78	8.78	10.39	9.64	8.81	8.81	9.58	10.22
	K	0.24	0.24	0.09	0.27	0.22	0.22	0.20	0.21	0.23	0.23	0.08	0.21
	R^2	0.81	0.81	0.87	0.93	0.82	0.82	0.98	0.99	0.82	0.82	0.88	0.99
	Δqe(%)	89.63	83.71	95.70	91.86	90.27	85.79	94.73	93.48	91.43	91.13	95.96	94.14
	SEE	263.32	304.39	408.15	362.59	272.92	273.58	421.96	346.45	275.20	298.63	437.71	398.32
1	$\ln[A]_0$	4.36	4.52	4.48	4.74	4.38	4.41	4.75	4.61	4.37	4.47	4.52	4.74
	K	0.05	0.06	0.01	0.05	0.04	0.05	0.03	0.03	0.04	0.04	0.01	0.03
	R^2	0.91	0.79	0.87	0.96	0.92	0.87	0.96	1.00	0.91	0.91	0.91	0.96
	Δqe(%)	36.41	134.27	10.01	22.72	31.42	57.56	13.01	4.97	30.59	35.20	6.82	14.13
	SEE	18.71	46.49	2.78	11.94	16.08	28.78	6.68	0.50	15.42	17.81	1.93	5.86
3/2	$1/[A]_0^{1/2}$	0.01	0.01	0.02	0.005	0.01	0.01	0.01	0.008	0.02	0.02	0.02	0.006
	K	0.01	0.009	0.001	0.008	0.01	0.008	0.003	0.005	0.01	0.006	0.001	0.004
	R^2	1.00	1.00	0.89	0.94	1.00	1.00	0.90	0.92	1.00	1.00	0.93	0.87
	Δqe(%)	85.26	78.83	91.25	88.69	86.16	80.67	90.47	89.07	86.69	86.39	91.78	89.81
	SEE	236.28	277.54	292.83	327.16	246.83	247.73	301.20	308.98	250.35	273.72	314.70	354.18
2	$1/[A]_0$	0.005	0.007	0.011	-0.003	0.006	0.002	0.005	0.004	0.008	0.006	0.011	0.003
	K	0.002	0.004	0.0002	0.002	0.002	0.003	0.0008	0.001	0.002	0.002	0.0001	0.001
	R^2	1.00	0.69	0.89	0.94	1.00	0.97	0.90	0.92	1.00	0.99	0.93	0.87
	Δqe(%)	50.39	66.62	8.78	231.97	34.05	145.64	58.17	68.93	18.76	38.69	9.17	108.29
	SEE	121.03	99.82	2.31	2675.31	54.52	1036.49	161.82	223.61	15.39	71.28	2.94	567.30
3	$1/[A]_0^2$	-0.002	0.000	0.0001	-0.0003	-0.001	-0.001	0.0001	-0.0001	-0.001	-0.001	0.0001	-0.0001
	K	0.0004	0.0009	0.000005	0.0003	0.0002	0.0002	0.000004	0.0001	0.0002	0.0002	0.000004	0.0001
	R^2	0.94	0.58	0.90	0.86	0.94	0.96	0.94	0.81	0.96	0.96	0.94	0.81
	Δqe(%)	61.29	77.74	14.07	78.24	52.24	41.86	62.08	59.50	55.57	49.78	15.87	67.02
	SEE	66.95	170.35	6.55	180.78	54.65	34.68	41.61	87.21	58.15	48.93	8.31	132.74

$$\Delta q_e(\%) = 100\sqrt{\frac{\sum[(q_{e\,(exp)} - q_{e\,(cal)})/q_{e\,(exp)}]^2}{N-1}} \quad (3)$$

$$SSE = \sum \frac{(q_{e\,(exp)} - q_{e\,(cal)})^2}{q_{e\,(exp)}} \quad (4)$$

3.3. Assessment of half-life values.

Pesticide persistence is often measured in terms of its half-life (the period of time it takes for its concentration to drop to 50%). The calculated half-lives of BTZ, BPS, DNF and IMI were 15.27, 11.73, 63.01 and 14.87 days, respectively, for the alluvial soil; 16.39, 13.10, 23.42 and 20.39 days of respectively, for the calcareous soil; and 17.82, 16.20, 75.34 and 21.00 days, respectively, for the sandy soil when they were applied at a rate of 100 µg/g soil (Table 6). The half-life values vary depending on the soil type and organic matter content [53-55]. The FOOTPRINT Pesticide Properties Database 12 reported that BTZ has a half-life in soil of 45 days in the laboratory at 20°C, 4 to 21 days in field conditions [56], 8 to 102 days in laboratory conditions according to Paszko and Muszyński [12], 8 to 35 days in the laboratory and 3 to 31 days in field studies for topsoils according to Paszko and Spadotto [15]. BPS is considered partially soluble in water and has a half-life of 60 days [23]. Neonicotinoids were previously observed to degrade more quickly in soils with cover crops than in bare soils [42]. The half-lives of DNF in soils under laboratory conditions were less than those at 72 days [57], but under field conditions, they were 9.34 to 11.4 days [58]. Shenggan et al in a study on the residue dynamics of IMI in rice, reported that the half-life of IMI in soil ranged from 12 to 24 days [59].

Table 6. Half-lives of the tested pesticides in soils fitted with a first-order kinetic model.

Pesticide	Alluvial soil	Calcareous soil	Sandy soil
BTZ	15.27	16.39	17.82
BPS	11.73	13.10	16.20
DNF	63.01	23.42	75.34
IMI	14.87	20.39	21.00

4. Conclusions

The initial pesticide concentrations ranged from 92.85 to 99.98 µg/g in the alluvial soil, 95.57 to 99.90 µg/g in the calcareous soil and 92.89 to 100.00 µg/g in the sandy soil, while after 60 days of incubation, the pesticide concentrations decreased to 4.82-49.87 µg/g in the alluvial soil, 5.23-17.66 µg/g in the calcareous soil and 8.82-53.09 µg/g in the sandy soil. BTZ and BPS dissipated faster than IMI and DNF in the three soils. The dissipation of DNF was faster in calcareous soil, followed by that in alluvial soil and sandy soil; unlike in the other tested pesticides, DNF dissipation was greater in alluvial soil, calcareous soil, and sandy soil. The persistence kinetics of BTZ, BPS, DNF, and IMI in the tested soils were analyzed using different orders of kinetic models (zero-order, 1/2-order, first-order, 3/2-order, second-order, and third-order). The results showed that the first-order model, followed by the 1/2-order kinetic model, fit the experimental persistence data quite well, with high R2 values and low Δqe% and SSE values. The half-life ranged from 15.27 to 17.82 days for BTZ, 11.73 to 16.20 days for BPS, 23.42 to 75.34 days for DNF, and 14.87 to 21.00 days for IMI in the tested soils according to the first-order equation.

Author Contributions

Conceptualization, A.E.M.; methodology, M.R.F.; data curation, M.R.F. and A.E.M.; investigation, A.E.M.; resources, M.R.F.; writing—review and editing, M.R.F.

Institutional Review Board Statement

Not applicable.

Informed Consent Statement

Not applicable.

Data Availability Statement

The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

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Conflicts of Interest

None.

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