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Low-Cost Sorbent for Removing Pesticides during Water

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1. Introduction

Pesticides are toxic chemicals for fighting against various diseases and pests. These compounds are carcinogenic, teratogenic, embryotoxic, and mutagenic. Expired or not used, they become very dangerous wastes, that when improperly stored, penetrate in uncontrolled way to the natural environment making the threat for all living forms. Therefore, the Sophia Declaration was created to underline the negative influences of durable organic pollutants, useless plant protection means, and other substances unsafe for human, environment, animals, and natural resources conditions such as ground waters or soil, and to emphasize the economic results related. The Declaration stresses the accelerating activities to remove above mentioned pollutants and appeals to governments and local organizations that removal of useless dangerous substances was a priority. It also turns to European Union and other sponsors to support domestic initiatives that introduce strategies of removal the durable organic pollutants, useless plant protection means, and other dangerous substances.

The past years remained dozens of thousands of tons of pesticide wastes that have been stored since 50's of the 20th century. There is also possibility to worsen the construction condition along with the occurrence of corroding the concrete bunkers and wells, the outdated pesticides are deposited, and in consequence, a toxic leakage. The leakage can be transported by underground water and then in a form of so-called underground inflow, it is caught by a network of surface waters. Therefore, there is a necessity to search for the solutions to reduce the pesticide migration in an environment as well as to introduce new concepts. Thus, it seems to be purposeful to undertake studies upon application of sorption process using selected adsorbents as a screen for pesticides in order to reduce their migration from other graveyards, stores and contaminated soils and concrete. The manuscript presented the study upon the possibility to removal of pesticide from aqueous solutions by using low-cost sorbent.

2. Material and methods

2.1 Sorbates and sorbent

On a basis of literature data and own studies, chloroorganic pesticides that most often occurred near the graveyards at the highest concentrations were selected as representative sorbats. (Ignatowicz, 2008; 2009) Individual pure active substances (DDT, DDE, DDD) were

applied. Technical grade DDT of 99,8±0,1% purity, DDE 99,8±0,2% purity and DDD of 98,5±0,3% purity obtained from Institute of Industrial Organic Chemistry Analytical Department in Poland were used as an adsorbates. A sample solution of pesticide has been prepared by dissolving 1 g of pesticide in 10 ml of methanol and then diluted to 1 L with doubly distilled deionised water. The concentrations of prepared solutions were applied: 5 mg pesticide per litre.

Fig. 1. Compost prisms.

Properties of compost						
Manurial (mg/kg$_{dm}$)						
Ca	Mg	Nog	N-NH$_4^+$	Pog	C	K
20.2	4.2	8.1	0.2	6.1	265.8	2.2
Metal (mg/kg$_{dm}$)						
Pb	Cu	Cd	Cr	Ni	Zn	Hg
5.4	25.1	0.3	4.3	3.5	123	0.2
Permissible standard						
500	800	10	500	100	2500	5
Other (%)						
pH	Hydration		Dry mass		Organic matter	
5.88	73.7		26.3		70.3	

Table 1. The characteristic of compost.

Sewage sludge achieved directly from dairy treatment plant "Mlekovita" in Wysokie Mazowieckie (Dąbrowski, 2006) with sawdust addition composted under natural conditions in Rudka Forest Inspectorate was used as natural adsorbents. (Dąbrowski, 2005) „Mlekovita" is the largest dairy plant in Podlasie region, and one of the largest in Poland.

Individual biological sewage treatment station generates almost 1 100 tons of dry sludge that can be re-used. The characteristics of the compost are given in Table 1. The used waste products comply with the requirements of the ordinance of the Minister of the Environment concerning agricultural usage of municipal sewage sludge (Dz.U. z 2002 r., nr 134, poz. 1140).

2.2 Sorption procedure

Studies under static conditions were performed in accordance to methodology applied in Belgium, Germany, France, Italy, England, USA, Poland and other. (Spadotto, 2003; Ignatowicz, 2009; Hamadi, 2004; Kumar, 2003; Witbowo, 2007; Yuh-Shan, 2006; Mashayekhi, 2006; Tsui, 2007) They were aimed at plotting the adsorption isotherms due to which it is possible to compare the sorption capacities of different adsorbats on different adsorbents. Selected adsorbent, previously degassed, washed with distilled water and dried, was ground in spherical mortar and dried in electric drier at 150 °C for 3 hours till constant weight. Such prepared sorbent served for weighing following samples: 0.001, 0.002; 0.005; 0.01; 0.025 g per 100 ml solution. Representative samples of adsorbent were added into the conical flasks with glass stopper and containing working solution of the pesticide (5 mg L^{-1}). Flasks were shaken in electric oven at constant oscillation amplitude (9) for 24 hours, and then remained for 48 hrs to reach a complete adsorption equilibrium. After that, samples were subjected to double filtration on soft filter paper. Then, pesticide concentration in a filtrate according to obligatory methods was determined using gas chromatograph AGILENT6890. (Balinova, 1996; Siepak, 2001, 2009; Hussen, 2007; Munoz, 2009) Freundlich's, Langmuir's, Temkin, Jovanovic, BET's and Huttig's isotherms (Atkins, 2006) were plotted on a base of achieved results applying Statistica software in order to analyze the processes.

2.3 Analytical procedure

Pesticide concentrations were determined in collected samples in accordance to obligatory methodology using gas chromatograph AGILENT6890 equipped with ECD and NPD detector. The injector temperature was 210 °C and the flow rate of helium was 1.0 mL min^{-1} . The column DB (35m length 0.32 mm i.d. 0.5 µm film thickness) temperature was set at 120 °C for 2 min and increased at a rate of 13 °C min^{-1} to 190 °C. The temperature was finally increased to 295 °C and maintained isothermally for 20 min.

Moreover, after sample digestion according to EPA 3015 procedure using microwave digester Mars 5, also metals concentrations were determined by means of ICP-AES technique, except of mercury determined by means of CV-AAS technique. (Balinova, 1996; Siepak, 2001, 2009; Hussen, 2007; Munoz, 2009) Chromatogram of chloroorganics pesticide present Figure 2.

3. Sorption isotherms

Among the several existing isotherms, the sorption datas were subjected to four commonly used isotherms models (Tab. 2), namely Langmuir (1918), Freundlich (1894), BET (1938), Huttig (1948), Jovanovic (1969) and Temkin (1963), to evaluate the maximum saturation capacity of adsorbent.(Atkins, 2006)
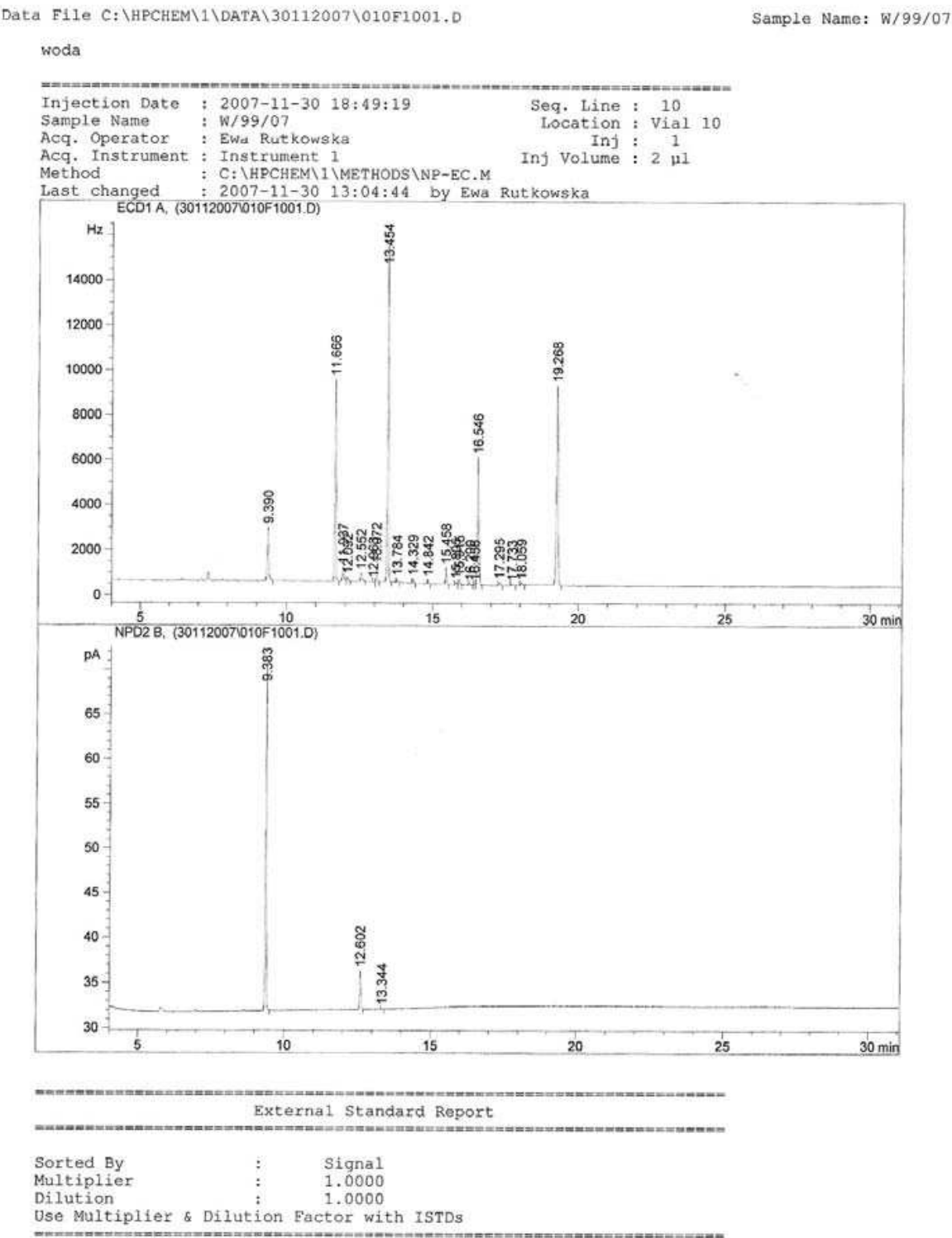
Data File C:\HPCHEM\1\DATA\30112007\010F1001.D   Sample Name: W/99/07

woda

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==============================================================================
Injection Date   : 2007-11-30 18:49:19           Seq. Line :   10
Sample Name      : W/99/07                        Location : Vial 10
Acq. Operator    : Ewa Rutkowska                       Inj :    1
Acq. Instrument  : Instrument 1                 Inj Volume : 2 µl
Method           : C:\HPCHEM\1\METHODS\NP-EC.M
Last changed     : 2007-11-30 13:04:44  by Ewa Rutkowska
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==============================================================================
                         External Standard Report
==============================================================================

Sorted By           :      Signal
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs
==============================================================================
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Instrument 1 2007-12-03 11:14:18 Ewa Rutkowska   Page 1 of 2

Fig. 2. The chromatogram of chloroorganic pesticide.

3.1 Freundlich model

The first mathematical fit to an isotherm was published by Freundlich and is a purely empirical formula for microporous and heterogeneous adsorbates:

$$q_e = K_F c^{1/n} \tag{1}$$

where q_e (mg.g^{-1}) is the amount of chloro - pesticide adsorbed on the adsorbent surface at equilibrium), c (mg.L^{-1}) the pesticide concentration in aqueous solutions at equilibrium, K_F (mg.g^{-1}) – constant - is the maximum multilayer adsorption capacity and $1/n$ is a characteristic constant which measures the adsorption intensity (k and n are empirical constants for each adsorbent-adsorbate pair at a given temperature). As the temperature increases, the constants k and n change to reflect the empirical observation that the quantity adsorbed rises more slowly and higher pressures are required to saturate the surface. The linear form of the Freundlich isotherm is shown in Table 2.

3.2 Langmuir model

Langmuir isotherm is a semi-empirical isotherm derived from a proposed kinetic mechanism. Langmuir isotherm is a model for monolayer localized physical adsorption on homogeneous surface; may be extended with heterogeneity effects, lateral interactions and multilayer effects. It is based on four assumptions:

- The surface of the adsorbent is uniform, that is, all the adsorption sites are equivalent,
- Adsorbed molecules do not interact,
- All adsorption occurs through the same mechanism,
- At the maximum adsorption, only a monolayer is formed: molecules of adsorbate do not deposit on other, already adsorbed, molecules of adsorbate, only on the free surface of the adsorbent.

The Langmuir equation may be written as:

$$q_e = q_m K C_e / 1 + K C_e \tag{2}$$

where q_e (mg.g^{-1}) is the amount of pesticide adsorbed on the adsorbent surface at equilibrium, C_e (mg.L^{-1}) the pesticide concentration in aqueous solutions at equilibrium, q_m (mg.g^{-1}) is the maximum monolayer adsorption capacity, K (L mg^{-1}) is the constant related to the free energy of adsorption. Eq. (1) can be linearized to five different linear forms as shown in Table 2.

3.3 BET model

Often molecules do form multilayer, that is, some are adsorbed on already adsorbed molecules and the Langmuir isotherm is not valid. In 1938 Stephan Brunauer, Paul Emmett, and Edward Teller developed a model isotherm that takes that possibility into account. The Langmuir isotherm is usually better for chemisorption and the BET isotherm works better for physisorption for non-microporous surfaces. The BET equation may be written as:

$$A = ac / (1+c)(1+Kc) \tag{3}$$

where A (mg.g^{-1}) is the amount of pesticide adsorbed on the adsorbent surface at equilibrium, c (mg.L^{-1}) the pesticide concentration in aqueous solutions at equilibrium,

a (mg.g^{-1}) is the maximum multilayer adsorption capacity, K (L mg^{-1}) is the constant related to the free energy of adsorption.

3.4 Jovanovic model

The Jovanovic model keeps the same assumptions contained in the Langmuir model, only considering, in addition the possibility of some mechanical contacts between the adsorbing and desorbing molecules. Moreover, a different extension of Jovanovic model for heterogeneous surface, the Jovanovic-Freundlich model, has been applied as a semiempirical model to the adsorption data of imprinted polymers. The Jovanovic-Freundlich model for single component adsorption was derived from a different relationship relating the surface coverage and the bulk concentration of the adsorbate. The model reduces to the Jovanovic model when the surface is homogeneous. It reduces to the monolayer isotherm at high concentration but does not obey the Henry's law concentrations. The energy distribution corresponding to this model for Jovanovic local behavior is a quasi-Gaussian function skewed in the direction of high adsorption energies. The Jovanovic equation may be written as:

$$q_e = q \, (1 - \exp(-Kc)) \tag{4}$$

where q_e (mg.g^{-1}) is the amount of pesticide adsorbed on the adsorbent surface at equilibrium, c (mg.L^{-1}) the pesticide concentration in aqueous solutions at equilibrium, q (mg.g^{-1}) is the maximum multilayer adsorption capacity, K (L mg^{-1}) is the constant related to the free energy of adsorption.

3.5 Temkin model

The Temkin [22] isotherm equation assumes that the heat of adsorption of all the molecules in the layer decreases linearly with coverage due to adsorbent – adsorbate interactions, and that the adsorption is characterized by a uniform distribution of the binding energies, up to some maximum binding energy. Temkin model is given by:

$$\theta = R \, T / \, \Delta Q \, \ln K_0 C_e \tag{5}$$

where θ is the fractional coverage, R the universal gas constant (kJ mol^{-1} K^{-1}), T the temperature (K), $\Delta Q = (-\Delta H)$ the variation of adsorption energy (kJ mol^{-1}), and K_0 is the Temkin equilibrium constant (L mg^{-1}). If the adsorption obeys Temkin equation, the variation of adsorption energy and the Temkin equilibrium constant can be calculated from the slope and the intercept of the plot θ versus $\ln C_e$ (Table 2).

3.6 Huttig model

Huttig's isother is a model for adsorption on homogeneous surface with multilayer effects. The Huttig equation may be written as:

$$A = ac \, (1+c) \, / \, 1 - Kc \tag{6}$$

where A (mg.g^{-1}) is the amount of pesticide adsorbed on the adsorbent surface at equilibrium, c (mg.L^{-1}) the pesticide concentration in aqueous solutions at equilibrium, a (mg.g^{-1}) is the maximum multilayer adsorption capacity, K (L mg^{-1}) is the constant related to the free energy of adsorption.

Isotherm	Type of relation of physicochemical quantities	Formula	Linear formula	Formula constant	Method of estimation
Monolayer					
Freundlich	Adsorption on heterogeneous surface with monolayer effect; micro-porous solids	$q_e = K_F c_e^{1/n}$	$\ln q_e = \ln K_F + \dfrac{1}{n} \ln c_e$	$A = kc^{1/n}$ n, k	Gauss-Newton
Langmuir	Adsorption on homogeneous surface with monolayer effect (lateral inter-actions and multilayer effect may be easily incorporated)	$q_e = \dfrac{qbc}{1+bc}$	$q_e = -\dfrac{1}{b}\dfrac{q}{c} + q$	$A = ac/1+kc$ a, k	Gauss-Newton
Temkin	Adsorption on heterogeneous surface with monolayer effect	$\Theta = \dfrac{RT}{\Delta Q} \ln k\, c_e$ $q_e = q_m + K \lg c$	$\Theta = \dfrac{RT}{\Delta Q} \ln k + \dfrac{RT}{\Delta Q} \ln c_e$	$A = a + k$ lgc a, k	Gauss-Newton
Jovanovic	Adsorption of organic sub-stance from di-lute solutions on heterogeneous surface with monolayer effect (vertical inter-actions and multilayer effect may be easily incorporated)	$\Theta = 1 - \exp(-kc)$		$A = a(1-\exp(-kc)$ a,k	Gauss-Newton
Multilayer					
Huttig	Adsorption on homogeneous surface with multilayer effect	$\Theta = (1+c) \dfrac{Kc}{1+Kc}$		$A = (1+c)ca/(1-kc)$ a, k	Gauss-Newton
BET	Adsorption on homogeneous surface with multilayer effect	$\Theta = \dfrac{1}{1-c}\left(\dfrac{Kc}{1+(k-1)c}\right)$	$\dfrac{c}{q_e(1-c)} = \dfrac{1}{q_m k} + \dfrac{k-1}{q_m k} C$	$A = ac/(1+c)(1+kc)$ a, k	Gauss-Newton

Table 2. The model sorption isotherm characteristic.

3. Results and discussion

Achieved study results are presented in Figures 3-7 and Table 3. Characteristics of applied adsorbent (Table 1) indicate that meet requirements of compost for natural applications. The parameters calculated according to the isotherm models studied are listed in Table 3. The adsorption process is described using Freundlich's, Langmuir's, Huttig's, Tiemkin, Jovanovic and BET's formulae:

$$\text{Freundlich } A = kc^{1/n}$$

$$\text{Langmuir } A = a_m kc/1 + kc$$

$$\text{BET } A = ac/(1+c)(1+kc)$$

$$\text{Huttig } A = (1+c)ca/(1-kc)$$

$$\text{Temkin } A = a + k \, lgc$$

$$\text{Jovanovic } A = a(1-\exp(-kc))$$

Following curves were achieved $A_F = 417.28 \, c^{0.273}$ for compost at correlation coefficient of R=0.91 (Fig. 3); $A_L = 3149.6 \, c/1-1056.4 \, c$ at correlation coefficient of R=0.55 (Fig. 4); and A_{BET} = 5298.18 c/(1+c)(1-4.836c) at correlation coefficient of R=0.76 (Fig. 5); and A_H =(1+c)c51159.3/(1-249.86c) at R=0.86 (Fig. 6), and A_T = 285.97 + 71.6 lgc at R=0.88 (Fig.7), and A_J = 226.07 (1-exp(-63.98c)) at R=0.82 (Fig.8).

Constants k and 1/n for Freundlich model were estimated by means of the least squares by Gauss-Newton method applying Statistica software, and then the errors for these constants were evaluated (Fig. 3). The other isotherms were calculated applying Statistica software by

Izoterm	a	b	R
Freundlich	417.283	0.273	0.91
Langmuir	3149.6	-1056.4	0.55
Temkin	285.967	71.603	0.88
Huttig	51159.3	-249.86	0.86
BET	5298.18	-4.8358	0.76
Jovanovic	226.07	63.988	0.82

Table 3. The coefficients of the adsorption isotherms (level of confidence 95%, α=0,05).

means of the least squares by Gauss-Newton method and achieved constants a and k are presented in Figure 3-9. Figures 3-9 present adsorption isotherms for studied pesticides on applied low-cost natural adsorbents as a function of adsorbate amount adsorbed by adsorbent weight unit (x/m) vs. concentration of the pesticide at the equilibrium (c_0).

The correlation coefficients (R) were employed to ascertain the fit of all isotherms with the experimental data. From Table 3, the coefficients R values were found higher for the Freundlich, Temkin, Huttig and Jovanovic models than for the Langmuir and BET models. This indicates that the Freundlich, Temkin, Huttig and Jovanovic isotherm are clearly the best fitting isotherm model for experimental data. According to the two used models (BET and Langmuir), important gaps in the adsorption capacities of the low-cost adsorbents have been noted. Based on the values fit a BET line to experimental data the parameters q and k are set. In general, the parameter of monolayer capacity adsorption q is consistent with other methods (the differences rarely exceed 20%). Adsorption equilibrium constant k determined by this method can be even negative, which is physically meaningless and can be regarded as an artifact of the method of matching. In fact, such differences may arise in the case of highly heterogeneous adsorbents or microporous. This confirms the theory of heterogeneous surfaces of compost (Atkins, 2006).
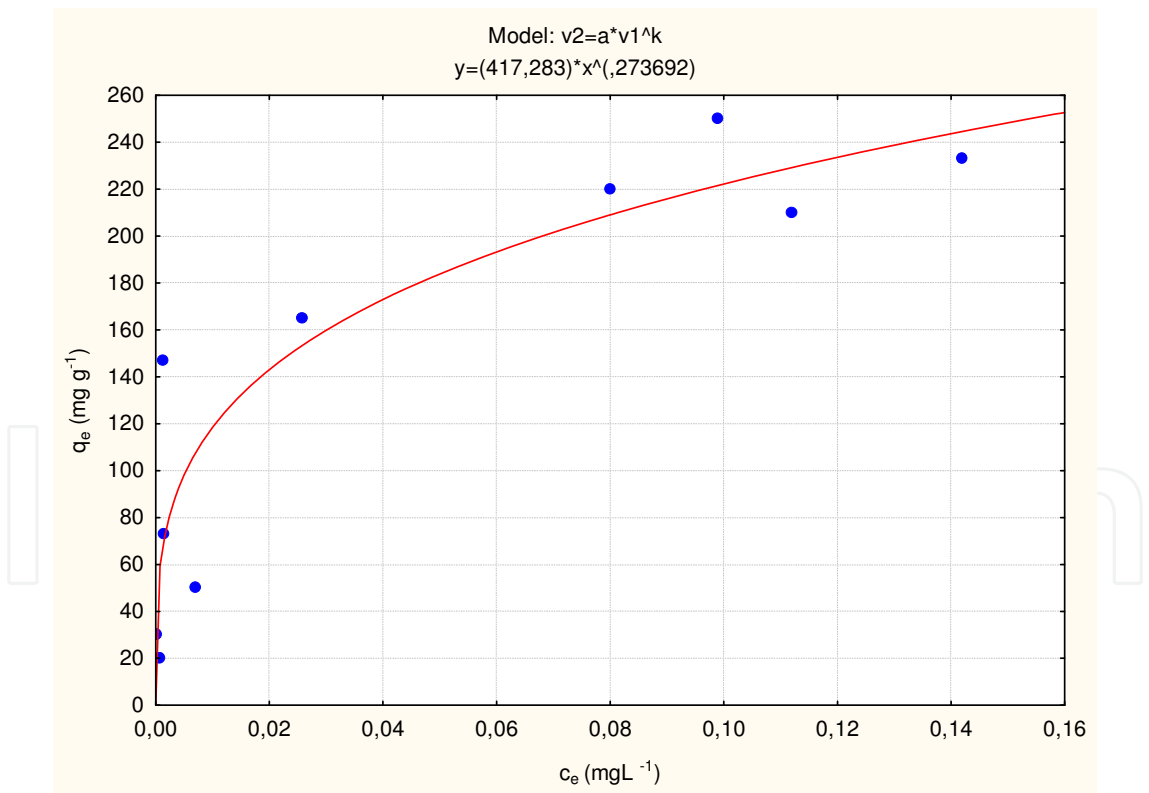
Fig. 3. Freundlich isotherm obtained using the nonlinear method for the sorption of pesticide on compost.
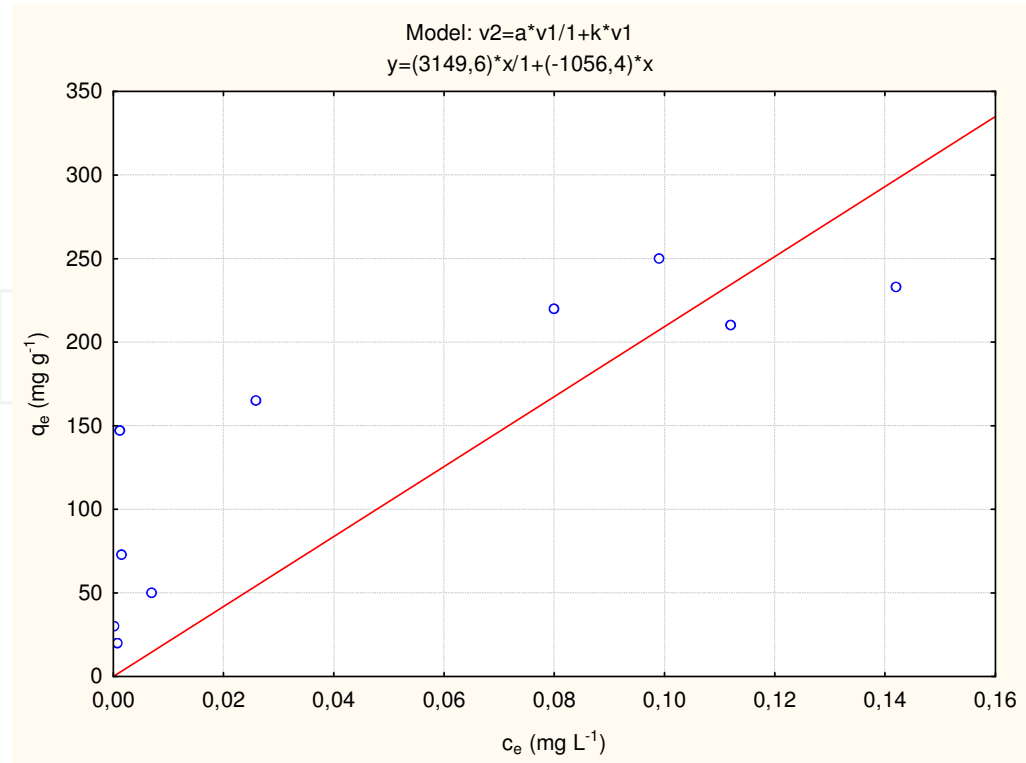
Fig. 4. Langmuir isotherm obtained using the nonlinear method for the sorption of pesticide on compost.
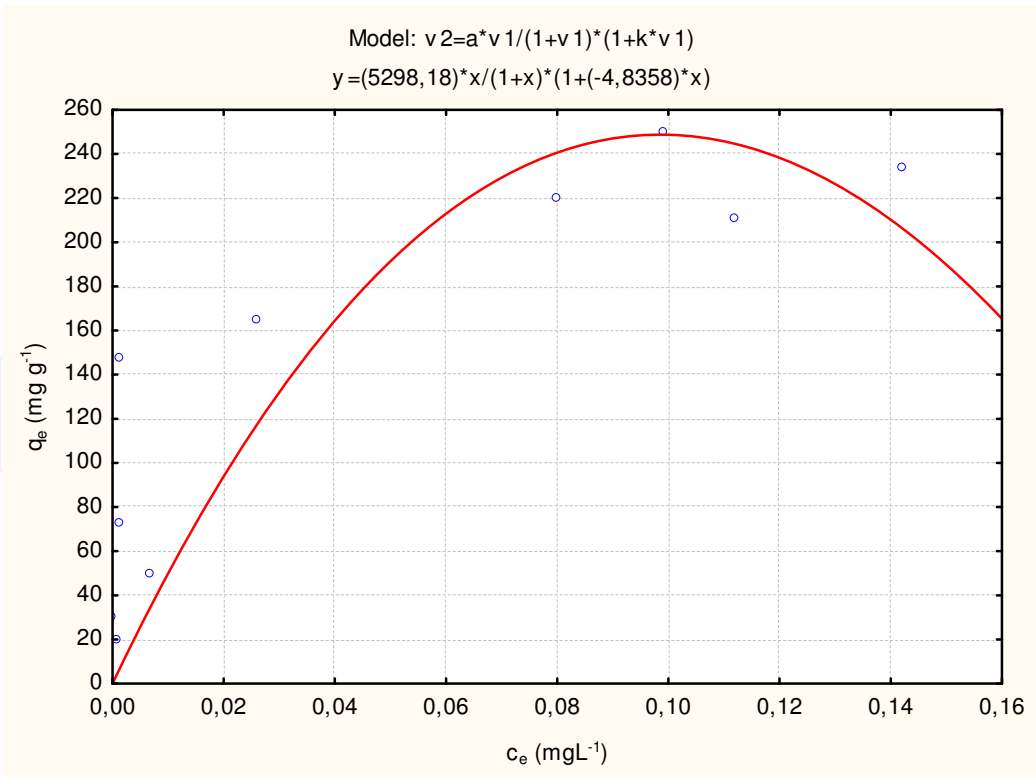

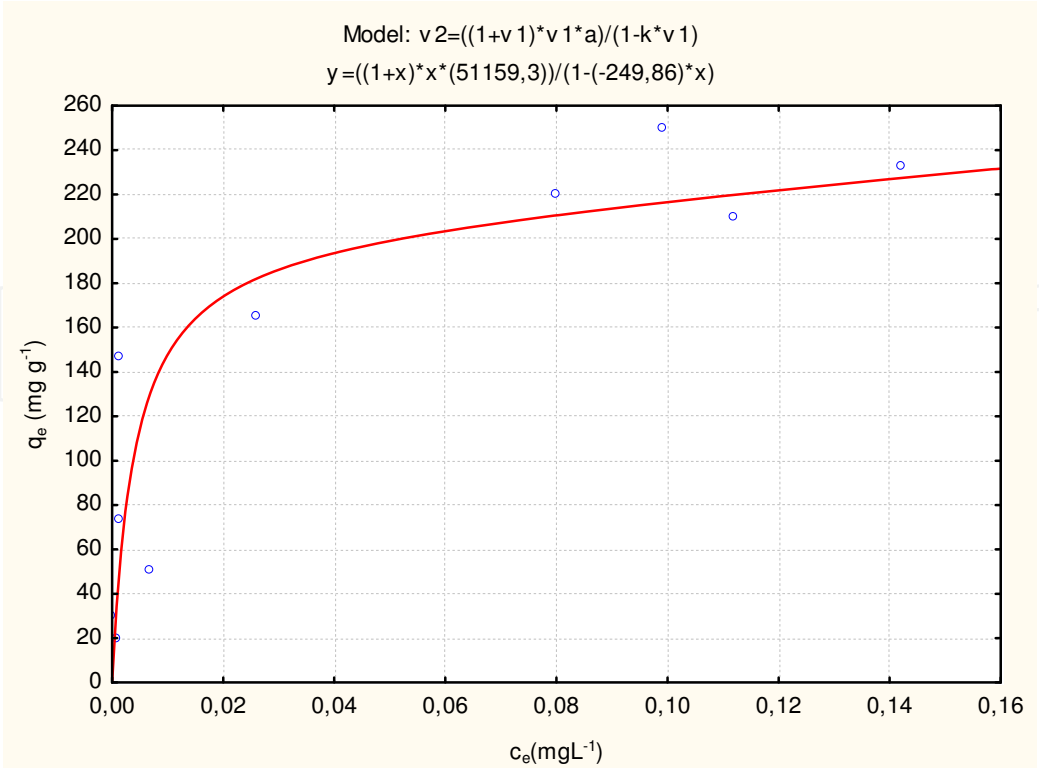Fig. 5. BET isotherm obtained using the nonlinear method for the sorption of pesticide on compost.

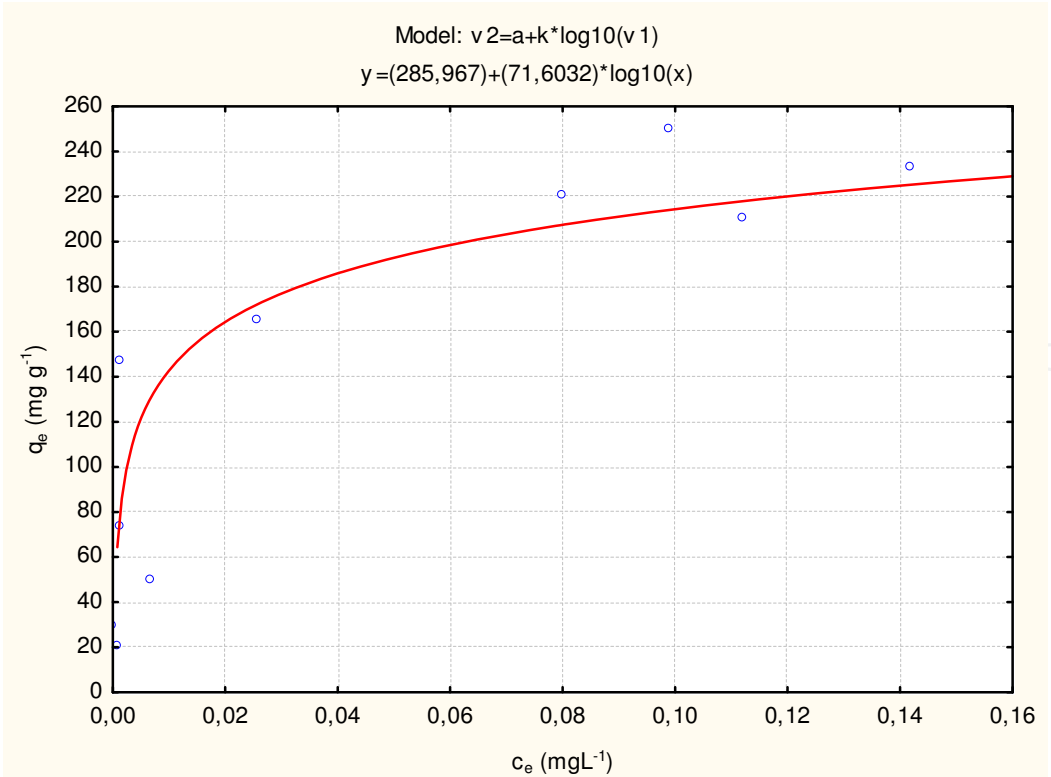Fig. 6. Huttig isotherm obtained using the nonlinear method for the sorption of pesticide on compost.

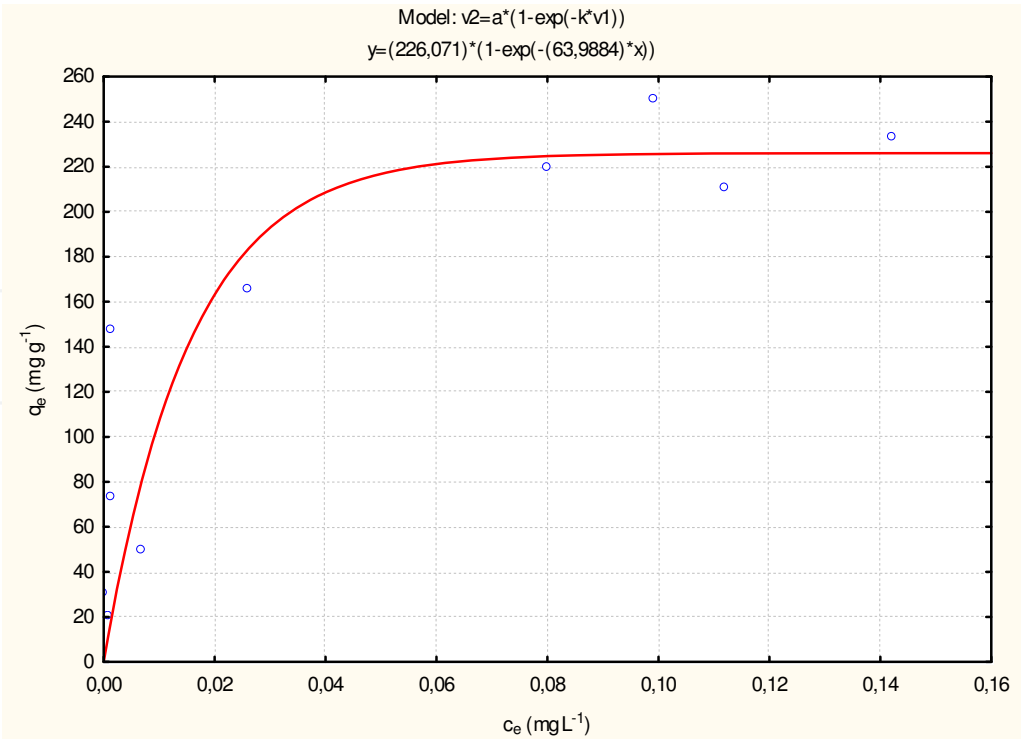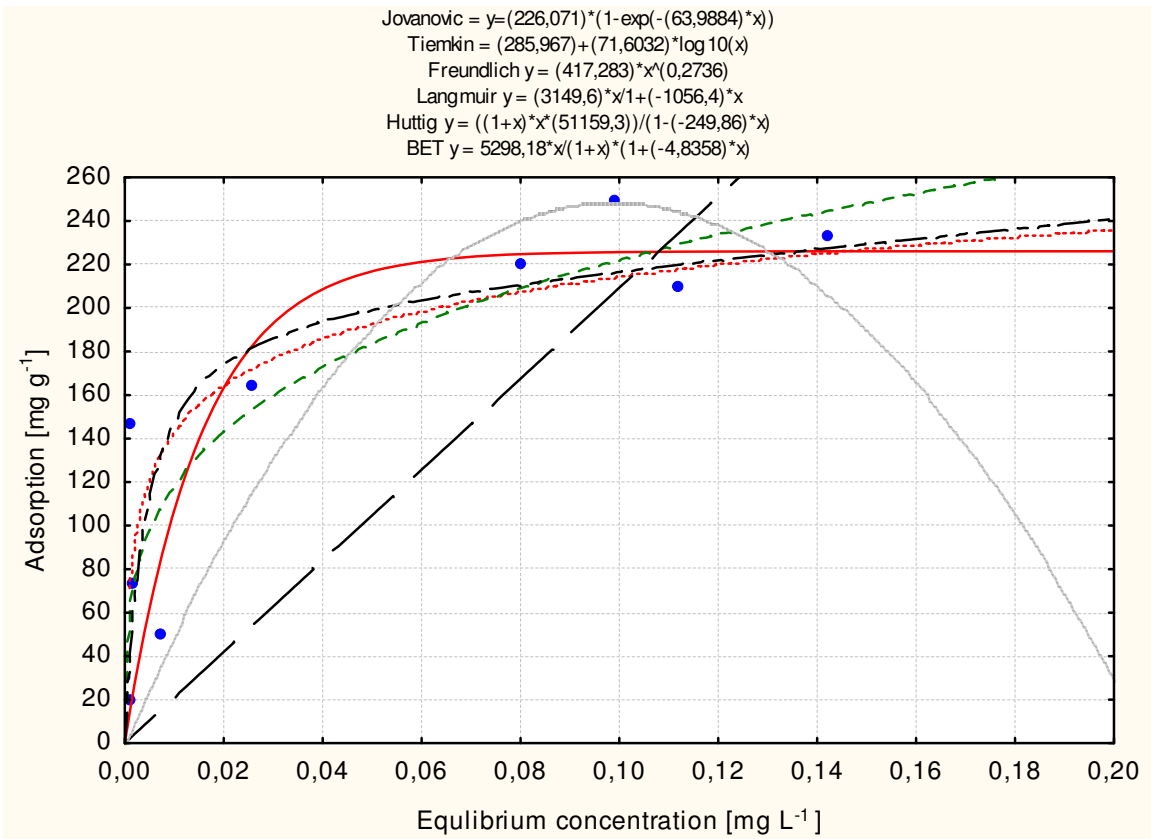Fig. 7. Temkin isotherm obtained using the nonlinear method for the sorption of pesticide on compost.

Fig. 8. Jovanovic isotherm obtained using the nonlinear method for the sorption of pesticide on compost.

Fig. 9. The comparison of model's isotherms of pesticide on compost.

The nature of the studied pesticides (a chlorinated and hydrophobic molecule) suggests that its adsorption is of the hydrophobic type directly bound to the specific surface of the adsorbent particles. The isotherms study was performed according to the protocol described in the experimental chapter. The obtained results lead, firstly, to plot a, the amount of pesticide adsorbed on the adsorbent surface at equilibrium (mg.g⁻¹) against c_e, the pesticide concentration in the aqueous solution at equilibrium in order to classify the isotherms according to the classification of Giles et al., 1960 (Atkins, 2006). This one includes four main groups: L, H, S and C. The experimental adsorption isotherms of pesticides in aqueous solutions on the studied materials are presented in Fig. 3-9. The same group of isotherms according to Giles' classification (L) was achieved for all pesticides.(Atkins, 2006) The L shape of the adsorption isotherms means that there is no strong competition between solvent and the adsorbate to occupy the adsorbent surface sites. In this case, the longitudinal axes of the adsorbed molecules are parallel to the adsorbent surface (molecules adsorbed flat on the surface). From Fig. 3, the values of experimental maximum adsorption capacity (q_m experimental) for the three chloroorganic compounds on compost is about 260 mg g⁻¹.

According to Hamdaoui and Nafrechoux (2007) the pesticide molecules bind to the adsorbent through only one grouping and the adsorption becomes progressively easier as the absorbed quantity increases. Thus, the first fixed molecules facilitate the adsorption of the following molecules because of the lateral attraction. In the shape of these isotherms indicates that the chloroorganic pesticide is adsorbed as a monolayer and that there is no strong competition between the pesticide molecules and water to occupy the adsorption surface sites. In this case, the longitudinal axes of the adsorbed molecules are parallel to the adsorbent surface. This type of isotherm is relative to microporous adsorbents with a diameter lower than 25 Å, the adsorbent being saturated at the moment of the monolayer replenishment. (Atkins, 2006; Ignatowicz, 2009) There would be weak interactions therefore on these adsorbent surfaces because the number of layers cannot increase freely.

The equilibrium data were further analyzed using the Freundlich equation using the same set experimental data, by plotting ln qe versus Ce. The calculated Freundlich isotherm constants and the corresponding coefficient of correlation values were shown in Table 3. The coefficients of correlation are high (R≥0.91) showing a good linearity. The magnitude of the exponent n (n=3.66) gives an indication on the favorability of adsorption. It is generally stated that values of n in the range 2–10 represent good, 1–2 moderately difficult, and less than 1 poor adsorption characteristics (Hamdaoui, 2007).

Knowledge on 1/n parameter value in Freundlich's formula allows for assessing the adsorption intensity of a given substance from water phase on adsorbent; value of k constant determines the sorption capacity of an adsorbent at balance concentration in a solution. Higher k value corresponds to higher sorption capacity. In own studies, higher value of k coefficient was achieved for compost, which proves its usefulness in application as sorption screen around the pesticide graveyard. Constants 1/n in Freundlich's formula is directional coefficients of isotherms equal to the tangent of line inclination angle in logarithmic coordinates. Therefore, the higher 1/n value, the more intensive adsorption process. (Ignatowicz, 2009; Pagnanelli, 2009; Akhtar, 2009)

The parameters of Temkin model as well as the correlation coefficients are given in Table 3. The very higher values of the coefficient of correlation show a good linearity whatever the

maximum adsorption capacity used for the calculation of surface coverage. The variation of adsorption energy, $\Delta Q = (-\Delta H)$, is positive for all the studied compounds, which indicates that the adsorption reaction is exothermic. In order to seek a systematic for the observed changes between the variations of adsorption energy of the tested chloroorganic compounds, it seems that the chloro group has a negative increment (exothermic effect).

4. Conclusions

The present study indicates the suitability of the low-cost adsorbent - compost for removal of graveyard's chloroorganic pesticides from aqueous solutions. The adsorption process is described using Freundlich, Temkin, Huttig and Jovanovic formulae. The Freundlich and Temkin models were fitting better the experimental datas. According to the two used models, important gaps in the adsorption capacities of the low-cost adsorbents have been noted.

The same group of isotherms according to Giles' classification (L) was achieved for chloroorganic pesticide. The pesticide molecules bind to the adsorbent through only one grouping and the adsorption becomes progressively easier as the absorbed quantity increases. Thus, the first fixed molecules facilitate the adsorption of the following molecules because of the lateral attraction. The shape of these isotherms indicates that the chloroorganic pesticide is adsorbed as a monolayer and that there is no strong competition between the pesticide molecules and water to occupy the adsorption surface sites. In this case, the longitudinal axes of the adsorbed molecules are parallel to the adsorbent surface. This type of isotherm is relative to microporous adsorbents with a diameter lower than 25 Å, the adsorbent being saturated at the moment of the monolayer replenishment.

5. Acknowledgments

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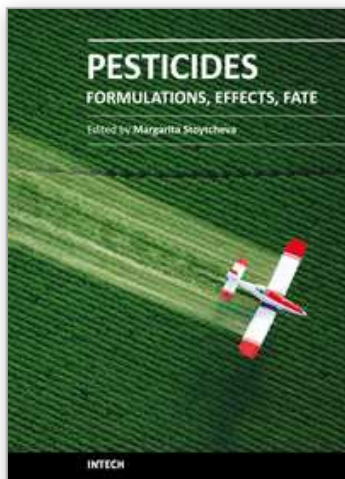
Pesticides - Formulations, Effects, Fate

Edited by Prof. Margarita Stoytcheva

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This book provides an overview on a large variety of pesticide-related topics, organized in three sections. The first part is dedicated to the "safer" pesticides derived from natural materials, the design and the optimization of pesticides formulations, and the techniques for pesticides application. The second part is intended to demonstrate the agricultural products, environmental and biota pesticides contamination and the impacts of the pesticides presence on the ecosystems. The third part presents current investigations of the naturally occurring pesticides degradation phenomena, the environmental effects of the break down products, and different approaches to pesticides residues treatment. Written by leading experts in their respective areas, the book is highly recommended to the professionals, interested in pesticides issues.

How to reference

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