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CONTENTS

Original scientific article

REMOVAL OF LINDANE FROM AQUEOUS SOLUTION BY GLYCIDYL METHACRYLATE BASED CHELATING MACROPOROUS COPOLYMER: KINETICS AND MECHANISM

Tamara T. Tadić[1], Bojana M. Marković[1], Mila V. Ilić[1], Aleksandra B. Nastasović[1], Antonije E. Onjia[2]

[1]University of Belgrade - Institute of Chemistry, Technology and Metallurgy, Njegoševa 12, 11000 Belgrade, Serbia
[2]Faculty of Technology and Metallurgy, University of Belgrade, Karnegijeva 4, 11000 Belgrade, Serbia

Abstract

Lindane belongs to the persistent organic pollutants (POPs) group, which can cause carcinogenic, endocrine, and neurological problems in an organism. The widespread presence of lindane in the environment demands efficient methods of its removal. In the present work, we investigated the removal of lindane from an aqueous solution using a synthesized macroporous copolymer surface modified with different functional groups through two-step post-functionalization. The modified copolymer was characterized by Fourier transform infrared spectroscopy (FTIR-ATR), scanning electron microscopy (SEM), and mercury intrusion porosimetry. For the kinetics of lindane sorption, pseudo-first-order, pseudo-second-order, Elovich, Avrami, and fractional power models were used via linear and non-linear regression analyses. The quality of the fitting of each model to experimental data was assessed based on seven error functions: coefficient of determination (R^2), Marquardt's percent standard deviation (MPSD), Chi-square statistic test (χ^2), hybrid fractional error function (HYBRID), the sum of the errors squared (SSE), sum of the absolute errors (EABS), and average relative error (ARE). The investigation of error estimation methods showed that the pseudo-second-order model best fits the experimental kinetics data for both regression analyses. The mechanism of lindane sorption was investigated by subjecting the data to the liquid film diffusion model, intraparticle diffusion model, Bangham, and Boyd models. The results showed that intraparticle diffusion was not the sole rate-controlling step; film-diffusion also affected the sorption process.

Keywords: chelating polymer, glycidyl methacrylate, lindane, kinetics, mechanism.

Introduction

Lindane (γ-hexachlorocyclohexane, γ-HCH) is an organochlorine pesticide. It is commonly used in agriculture as an insecticide and in pharmaceutical products as an ingredient in lotions and shampoos to control scabies and lice (Zhang et al., 2020). Due to its chemical stability, high lipid solubility, and migration over long distances, lindane can persist in the environment and cause widespread and long-term contamination (Benimeli et al., 2008). According to US EPA, lindane has been classified as mutagenic, genotoxic, and teratogenic (Khan et al., 2021). During the production of hexachlorocyclohexane (HCH) four isomers (α, β, γ, δ) are formed, but only the γ-HCH isomer has insecticidal activity. During the creation of lindane, a large amount of other waste isomers (α- and β-hexachlorocyclohexane) is created, so the production of γ-HCH isomer is non-effective (Vijgen et al., 2011). In 2009, the Stockholm Convention listed lindane and its α and β isomers as persistent organic pollutants (POP). After that, lindane, along with its α and β isomers, was banned or restricted in many countries (Zhang et al., 2020). Broad lindane applications represent severe threats to the environment and human health. Due to its slow degradation, high lipid solubility, and chemical

stability, lindane residues have been leaching underground for years and contaminating surface and groundwater (Kumar & Pannu, 2018). However, in some developing countries (India), lindane is still used due to its versatility in controlling pests and low cost (Jain et al., 2022).
Lindane can affect the endocrine system and central nervous system. In addition, due to lipophilicity, lindane can accumulate in human breast milk and in organs that are rich in fat. Many studies reported that exposure to lindane could be associated with various types of cancer (prostate, lung, breast, etc.) (Jayaraj et al., 2016; Mortazavi et al., 2019).
Since lindane contaminates water through household and agriculture, its removal is important. Modification in various ways allows porous polymer materials to be applied for the sorption of different types of materials. Macroporous copolymer based on glycidyl methacrylate and ethylene glycol dimethacrylate (PGME) is considered as an excellent starting point for surface modification and post-functionalization via reaction of the epoxy ring with nucleophilic groups. PGMA is one of the most adaptable reactive polymers with excellent adsorption properties and the ability to reuse and regenerate, which makes it a promising adsorbent.

Materials and Methods

Starting material, PGME was modified in two-step post-functionalization. In the first step, the reaction with diethylene triamine (PGME-deta) was performed (Malović et al., 2007), and in the second step, with chelating agent (ethylenediaminetetraacetic dianhydride) according to the procedure described in the literature (Su et al., 2017). PGME-deta and chelating agent (1:1) were added to the flask with N,N-dimethylformamide (DMF) and the mixture was kneaded for 4 hours at 80 ± 0.2 °C. The obtained chelating copolymer was subsequently washed, dried, and characterized by Fourier transform infrared spectroscopy (FTIR-ATR), scanning electron microscopy (SEM), and mercury intrusion porosimetry.
The mechanism and kinetics of lindane sorption were determined in a non-competitive batch process at unadjusted pH (pH=6.3) and room temperature (25 ± 0.2 °C). The sorbent (0.90 g) was contacted with 90 cm^3 of lindane aqueous solution with a concentration of 150 μg/dm^3 and shaken on an orbital shaker for 180 min, at a speed of 450 °/min. At appropriate time intervals, 5 cm^3 of the solution was sampled with a micropipette and prepared for measurement by GC-ECD. All measurements were performed in duplicate, and the results were expressed as the mean value.
Sorption capacity (Q_t, μg/g) was calculated from:

$$Q_t = \frac{(C_0 - C_t)V}{m} \quad (1)$$

where: C_0 (μg/dm^3) and C_t (μg/dm^3) stand for the initial and final lindane concentration at time t, respectively, V (dm^3) is the solution volume, and m is the mass of the chelating copolymer.

FTIR spectra were taken in ATR mode in the range 4000–400 cm^{-1} using a Nicolet SUMMIT FT-IR Spectrometer (Thermo Fisher Scientific, Waltham, MA, USA). SEM micrographs were obtained on JEOL JSM-6610LV instrument (JEOL Ltd., Tokyo, Japan). Pore size distributions were determined by a high-pressure mercury intrusion porosimeter Carlo Erba Porosimeter 2000 (Washington, DC, USA, software Milestone 200). The specific surface area, S_{Hg} was calculated from the cumulative pore volume distribution curve as described in the literature (Nastasović et al., 2022). Lindane detection was determined on a gas chromatograph, Agilent 7890A GC coupled with an electron capture detector (ECD). TG-5MT capillary column (30 m × 0.25 mm × 0.25 μm) was used. The injection mode was splitless, while the temperature regime was set in the following way: 50 °C for 3 min, then increased at a rate of 30 °C/min to 210 °C and held at this temperature for 20 min. Hydrogen was used as the carrier gas with a 60 mL/min flow rate.

Results and discussion

Characterization of the sorbent

The introduction of reactive groups improves adsorption capacities, selectivity, and the pH range for sorption (Galhoum, 2019). Adsorption ability of GME-based polymer can be enhanced by two-step post-functionalization, firstly by ring-opening of the epoxy ring with diethylene triamine (deta), and secondly, by the introduction of a chelating agent through reaction with amine groups of deta. The post-functionalization of the chelating polymer was confirmed using FTIR-ATR spectrometry (Figure 1).

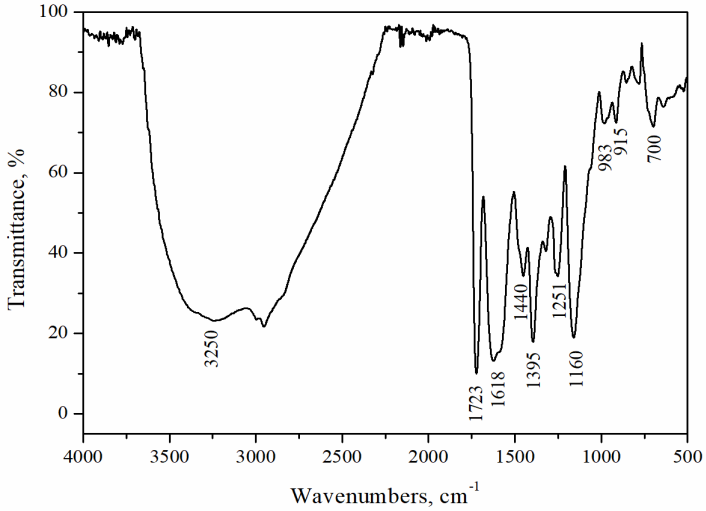

Figure 1. FTIR-ATR spectrum of the chelating copolymer

A broad band at 3800–2200 cm^{-1} in the FTIR-ATR spectrum of chelating copolymer originates from the presence of -NH_2 and -OH groups (overlapping bands for O-H and N-H amine stretching) (Peng et al., 2008; Tadić et al., 2022). A band at ~1620 cm^{-1} corresponding to -NH bending mode (Galhoum et al., 2019), while C-N stretching vibrations of the tertiary aliphatic amine were observed at 1160 cm^{-1} (Hwang et al., 2013). The peak at 1440 cm^{-1} was attributed to the vibration of the double covalent bond in –COOH (Wang et al., 2013).

As seen from SEM micrographs in Figure 2a, the particles were smooth spheres, while the micrographs of particle surface and cross-section (Figure 2b and 2c) clearly show a three-dimensional globular porous structure. The S_{Hg} value of the chelating copolymer calculated from the cumulative pore volume distribution curve was 35 m^2/g.

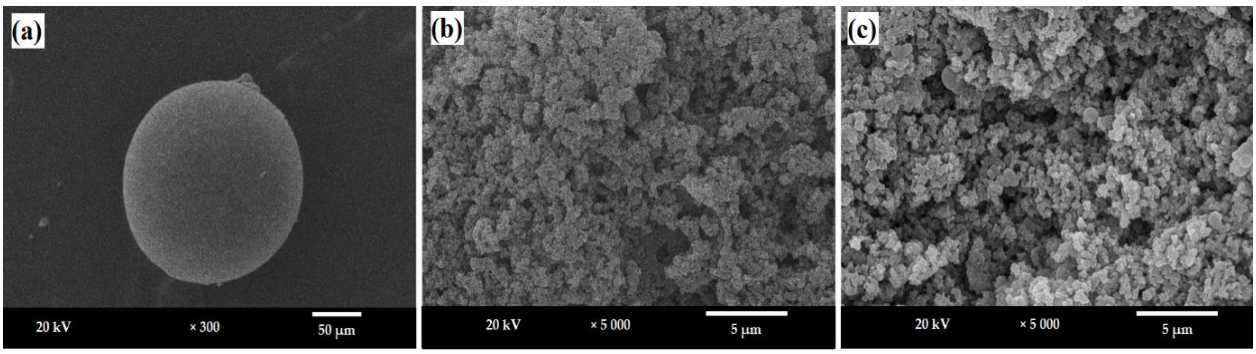

Figure 2. SEM micrographs of (a) particle beads (magnification 300×), (b) particle surface (magnification 5000×) and (c) cross-section (magnification 5000×) for chelating copolymer

Lindane sorption kinetics

The kinetic mechanism of lindane sorption on chelating copolymer was determined by linear and non-linear forms of pseudo-first-order, pseudo-second-order kinetic model, Elovich, Avrami and fractional-power model (Nastasović et al., 2022). Non-linear regression was used to fit the experimentally obtained data in the program Origin 9.0©, OriginLab Corporation. The following error functions were used to match the applied kinetic models with the experimental data: determination coefficient (R^2), sum squared error (SSE), squared angular errors (SAE), average relative error (ARE), hybrid fractional error function (HYBRID), Marquardt's percent standard deviation (MPSD), and chi-square test (χ^2) (Marković et al., 2014; Sivarajasekar & Baskar, 2019). Figure 3 shows the comparison of the fits for different linear and non-linear kinetic models, while Tables 1 and 2 summarize calculated kinetic parameters and the relevant correlation coefficients for linear and non-linear kinetic models, respectively.

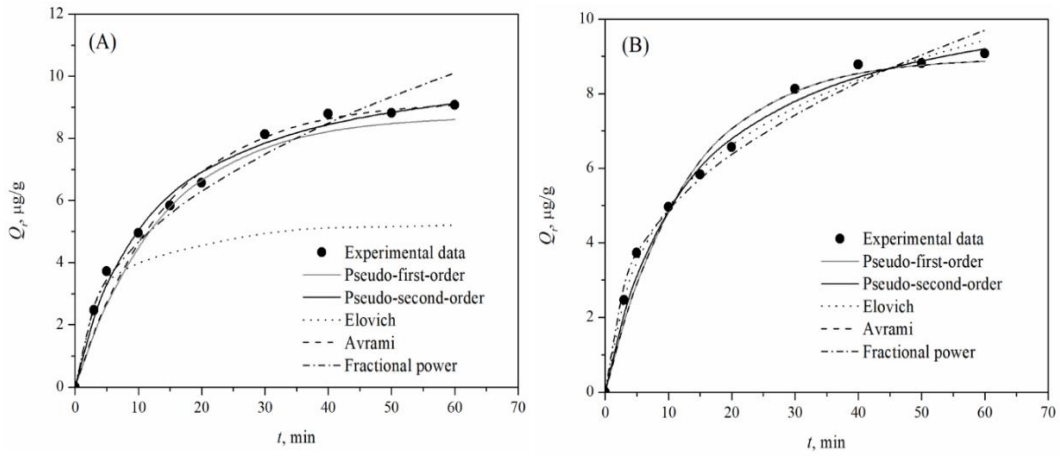

Figure 3. Kinetics model (A) linear and (B) non-linear plots for sorption of lindane onto chelating copolymer

For both linear and non-linear models, the higher R^2 values and the lowest values of other error functions suggest a better fit. According to values presented in Table 1, pseudo-second-order was the best suitable to describe the kinetic data of lindane sorption. Also, the error values indicated that the non-linear method is a better way to obtain the kinetic parameters describing the lindane sorption onto a chelating copolymer.

Table 1. Linear regression analysis for sorption kinetics of lindane onto chelating copolymer

Model	Parameter	Value	R^2	SSE	SAE	ARE	HYBRID	MPSD	χ^2
Pseudo-first-order	$k_1 \cdot 10^2$, 1/min Q_e^{cal}, μg/g	7.44 8.70	0.951	2.483	4.004	9.654	8.751	15.956	0.814
Pseudo-second-order	$k_2 \cdot 10^3$, g/ μg min Q_e^{cal}, mg/g	8.43 10.80	0.986	0.653	2.115	4.388	1.693	5.900	0.120
Elovich	α, μg/g min β, g/mg	2.17 0.43	0.592	58.928	19.352	28.807	102.069	35.740	11.739
Avrami	$k_{AV} \cdot 10^2$, 1/min Q_e^{cal}, μg/g n	8.32 9.19 0.87	0.965	1.700	2.801	7.947	6.892	14.607	0.634
Fractional-power	k_{FP}, μg/g min v, 1/min	0.43 1.76	0.956	2.160	3.757	6.790	4.285	8.691	0.286

Table 2. Non-linear regression analysis for sorption kinetics of lindane onto chelating copolymer

Model	Parameter	Value	R^2	SSE	SAE	ARE	HYBRID	MPSD	χ^2
Pseudo-first-order	$k_1 \cdot 10^2$, 1/min Q_e^{cal}, μg/g	8.04 8.94	0.969	1.501	2.800	7.178	5.336	1.521	0.443

Pseudo-second-order	$k_2 \cdot 10^3$, g/ µg min Q_e^{cal}, mg/g	7.25 11.11	0.993	0.373	1.603	3.070	0.837	3.975	0.059
Elovich	α, µg/g min β, g/mg	1.51 0.38	0.987	0.602	2.018	3.639	1.214	4.619	0.086
Avrami	$k_{AV} \cdot 10^2$, 1/min Q_e^{cal}, µg/g n	8.87 8.94 0.91	0.969	1.501	2.800	7.178	5.336	12.331	0.443
Fractional-power	k_{FP}, µg/g min ν, 1/min	2.06 0.38	0.967	1.578	3.004	6.251	4.449	11.195	0.277

The mechanism of lindane sorption was investigated by linear analysis with the liquid film diffusion model, intraparticle diffusion model, as well as Bangham, and Boyd models. The results given in Table 3 and Figure 5 showed that intraparticle diffusion was not the sole rate-controlling step; film-diffusion also affected the sorption process.

Table 3. Parameters for sorption mechanism of lindane onto chelating copolymer

Model	Parameter	Value	R^2
Intra-particle diffusion	$k_{id,1}$, µg/g min $C_{id,1}$, µg/g	1.37 0.43	0.990
	$k_{id,2}$, µg/g min $C_{id,2}$, µg/g	0.20 7.49	0.989
Bangham	$k_B \cdot 10^4$, µg/g min α	9.90 0.55	0.982
Liquid film diffusion	$k_{LFD} \cdot 10^2$, 1/ min C_{LFD}	7.44 -0.05	0.972

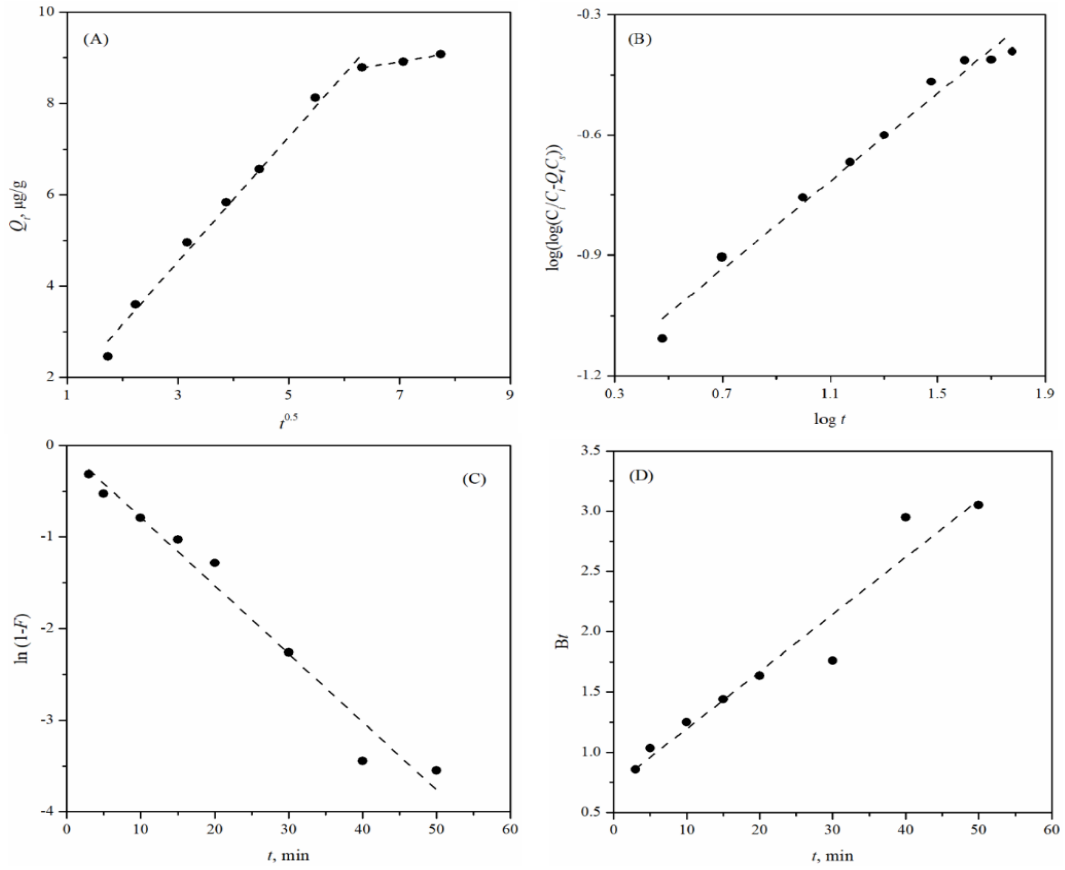

Figure 4. Linear plots for the (A) intra-particle diffusion, (B) Bangham, (C) liquid film diffusion and (D) Boyd models for the sorption of lindane onto chelating copolymer

Conclusion

In this study, PGME was chemically modified in two-step post-functionalization with diethylene triamine and chelating agent and tested as lindane sorbent. The characterization results confirmed post-functionalization and spherical porous structure of copolymer with a specific surface area of 35 m^2/g. The linear and non-linear forms of pseudo-first-order, pseudo-second-order, Elovich, Avrami, and fractional power models were used to analyze the kinetics of lindane sorption. The fitting of each model to experimental data, assessed based on seven error functions, showed that the pseudo-second-order model best fit for the experimental kinetics data for both regression analyses. Also, the error values indicated that the non-linear method is a better way to obtain the kinetic parameters of lindane sorption onto chelating copolymer. The mechanism of lindane sorption was investigated with the liquid film diffusion model, intraparticle diffusion model, Bangham, and Boyd models. It was shown that intraparticle diffusion and film-diffusion affected the lindane sorption process.

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