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JAHORINA
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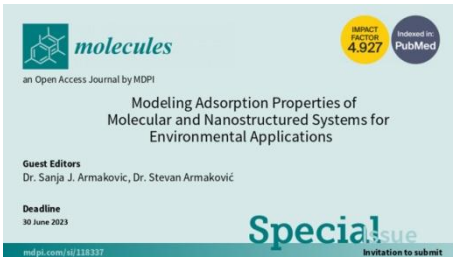

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OPTIMIZATION OF LINDANE SORPTION FROM AQUEOUS SOLUTION BY MACROPOROUS COPOLYMER USING EXPERIMENTAL DESIGN

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Abstract

In the present study, synthesized macroporous copolymer was used for the sorption of lindane from an aqueous solution. This organochlorine pesticide is classified, according to US EPA, as mutagenic and teratogenic. Residues of lindane can persist in the environment, migrate long distances, and cause widespread contamination. In this way, lindane residues can reach the human body through the food chain. The usage of this pesticide is banned in most countries. However, lindane remains a serious toxicological problem at industrial sites where lindane was used coupled with improper wastewater disposal and has led to serious contamination. In addition, some countries still allow the production and use of lindane, and despite localized restrictions, lindane contamination remains a global problem. Taking into account these reasons, its removal from the environment is of a great significance. Macroporous copolymer was syntesized via suspension copolymerization and characterized by Fourier transform infrared spectroscopy (FT-IR), and scanning electron microscopy (SEM). The parameters which affect sorption efficiency of the lindane were: pH, sorption time (t_{sorp}), ion strength (ion), rpm, and dose of sorbent. These variables were optimized by the experimental design which include Plackett-Burman Design (PBD) and Central Composite Design (CCD). After screening step by the PBD, the optimum conditions were obtained by the CCD where the values were investigated in two levels. The design of experiment showed that the ion strength and dose of sorbent were the most significant parameters, while the other variables do not have such an effect on sorption efficiency. Accordingly, the optimum conditions to reach the maximum recovery were: pH 8, 180 min sorption time, 300 rpm, 2 w/v % ion strength and 8 g/L dose of sorbent. The results showed that the studied copolymer could be an efficient sorbent for trace lindane in water with recoveries above 80 %.

Key words: *organochlorine pesticide, design of experiment, DoE, macroporous copolymer, optimization*

Introduction

Lindane (γ-hexachlorocyclohexane), organochlorine pesticide, is used to protect crops, but it is also an ingredient in some cosmetic products such as shampoos and lotions (Pelin Böke et al., 2020). US EPA classified lindane as mutagenic, genotoxic, and teratogenic (Khan et al., 2021). Lindane is officially banned in many countries, but it is still in use in developing countries (Jain et al., 2022). Due to its persistence, as well as long-distance migration, it

represents a potential danger to the environment and human health. Lindane is also neurotoxic, and chronic lindane exposure results in multiple adverse effects (Xu et al., 2020). Lindane removal is important because of its ability to contaminate the environment. Due to the growing awareness of the toxic effects of lindane, a large number of methods such as chromatography (McManus et al., 2013), capillary electrophoresis (Regan et al., 2003), spectrophotometry (Marzuki et al., 2017), and adsorption (Nguyen et al., 2020) have been developed for its removal from soil and water. Adsorption, as one of them, shows advantages such as high efficiency, simplicity, as well as cost-effectiveness (Suručić et al., 2023). Due to their easy modification and versatility, porous polymer materials can be observed for lindane removal.

In this work, copolymer based on glycidyl methacrylate (GMA) was syntesized and characterized by various methods while the parameters which affect sorption efficiency were optimized by the experimental design. In this way, an effective sorbent for removal of lindane from aqueous solution was obtained.

Materials and Methods

Glycidyl methacrylate (GMA), ethylene glycol dimethacrylate (EGDMA), 2,2'-azobisiso-butyronitrile (AIBN), cyclohexanol and 1-tetradecanol were obtained from Merck (Darmstadt, Germany). Poly(N-vinyl pyrrolidone) (PVP) was purchased from BASF (Ludwigshafen, Germany). An analytical standard of lindane with a purity of 98.2% ± 0.1% (CPAchem Ltd, Bulgaria) was used for testing lindane sorption.

Following the procedure (Ekmeščić et al., 2019), macroporous copolymer based on glycidyl methacrylate (GMA) was synthesized by suspension copolymerization with 80 wt.% of crosslinker (EGDMA). FTIR spectra were taken in ATR (attenuated total reflection) mode using a Nicolet SUMMIT FT-IR Spectrometer (Thermo Scientific, Massachusetts, USA) over the range of 400 - 4000 cm^{-1} with a resolution of 2 cm^{-1}. SEM analysis was performed on the JEOL JSM-6610LV instrument (JEOL Ltd., Tokyo, Japan).

Different variables which effect on sorption efficiency have been studied by using Design of experiments (DoE). For screening step Plackett–Burman Design (PBD) was used, while Central Composite Design (CCD) was applied to optimize the sorption process. Each independent variable was investigated at two levels: -1 (low level) and +1 (high level): pH (3-8), sorption time (60-180 min), ion strength (0-3 w/v %), mixing speed (0-300 °/min), dose of sorbent (1-10 g/L). The variables that had the greatest influence on the sorption process were further optimized by CCD at two levels: ion strength (1-3 w/v %), dose of sorbent (2-10 g/L). Statistical software used for DoE was MINITAB.

Table 1. Variables and levels of the PBD for the screening step.

Variables	Symbol	Level	
		Low	High
Plackett-Burman Design			
pH	*pH*	3	8
Sorption time (min)	t_{sorp}	60	180
Ion strength (w/v %)	*ion*	0	3
Mixing speed (°/min)	*rpm*	0	300
Dose of sorbent (g/L)	*dose*	1	10
Central Composite Design			
Ion strength (w/v %)	*ion*	1	3
Dose of sorbent (g/L)	*dose*	2	10

Lindane samples have been analyzed on a gas chromatograph (Agilent 7890A) coupled with an electron capture detector (ECD), and a TG-5MT capillary column (30 m × 0.25 mm × 0.25 μm) was used. The initial temperature was 50 °C for 3 minutes, then

heating was reached at a rate of 30 °C/minup to 210 °C and held at this temperature for 20 min. Hydrogen was used as the carrier gas with a flow rate 60 mL/min.

Results and Discussion

On the FTIR spectrum of the obtained GMA-based copolymer (Figure 1), the bands originating from the epoxy ring at 752 and ~1256 cm^{-1}, characteristic of C-O stretching vibrations (νC-O), can be observed. A peak at ~1150 cm^{-1} is assigned to C-O-C stretching vibration (νC-O-C), while a strong band at ~1730 cm^{-1} originates from the stretching vibrations of the ester carbonyl group (νC=O). The characteristic absorption bands at 2994 cm^{-1} and 2951 cm^{-1} originate from the asymmetric (νasim C-H) and symmetric (νsim C-H) stretching vibrations of the C-H bond of methyl and methylene group(Marković et al., 2017).

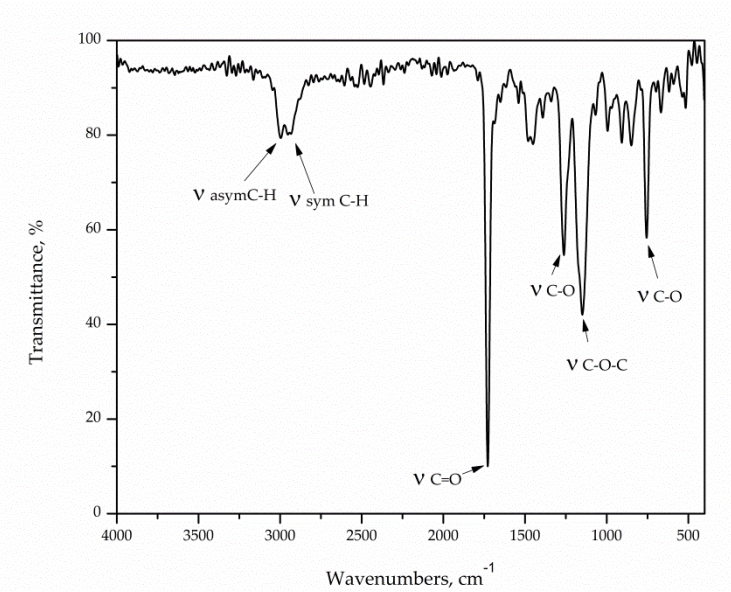

Figure 1.FTIR spectrum of GMA-based macroporous copolymer.

Using scanning electron microscopy (SEM), cross-section and the particle appearance (Figure 2) of the GMA-based copolymer were analyzed. The SEM images show that the particles of the obtained copolymer have three-dimensional globular porous structure.

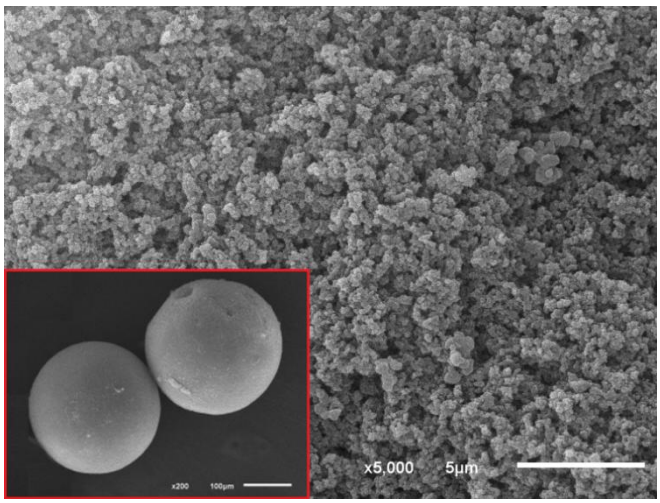

Figure 2.SEM image of cross-section for GMA-based macroporouscopolymer.
Inset: SEM image of obtained particles.

The effects of the five selected variables which effect on sorption efficiency were investigated in 12 runs. According to screening results, ion strength and dose of sorbent were the

most significant variables, while other variables had no significant effect on the sorption process.Therefore these two variables were included for the next optimization step, while the other three variables were fixed as follows: pH 8, 180 min for sorption time, and 300 rpm for mixing speed.

Contour plot (Figure 3) showed the combined influence of the significant variables on sorption efficiency. The optimum conditions to reach the maximum efficiency were: pH 8, 180 min sorption time, 300 rpm mixing speed, 2 w/v % ion strength and 8 g/L dose of sorbent. The results showed that the obtained copolymer could be an efficient sorbent for trace lindane in water with recoveries above 80 %.

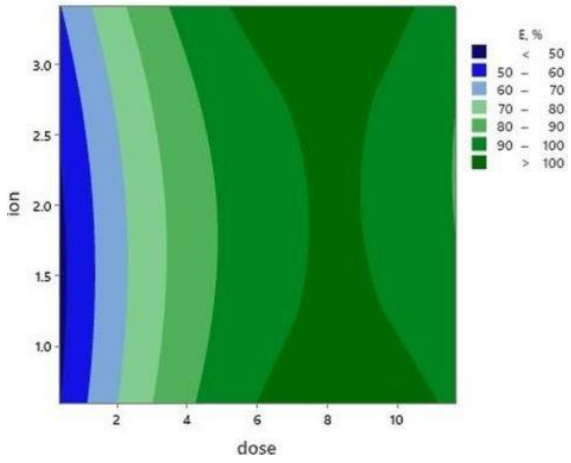

Figure 3. Contour plot ion strength – dose ofsorbent obtained from the CCD optimizationstep.

Conclusions

In this study, GMA-based copolymer was synthesized by suspension copolymerization and used as an effective sorbent forlindane removal from aqueous solution. The synthesized copolymer was characterized by FTIR and SEM. FTIR spectrum confirmed successful synthesis, while SEM showed a 3D spherical porous structure.Five affecting factors on the sorption efficiency(pH, sorption time, ion strength, rpm, and dose of sorbent) were optimized using DoE. With a combination of mathematical and statistical methods, significant process variables are identified and optimal conditions are obtained using a small number of experiments that are simultaneously investigated. PBD was used for screening step, while the optimum conditions were obtained by the CCD. According to screening step, ionic strength and dose of sorbent proved to be the most significant variables. The maximum efficiency were showed at optimum contidions: pH 8, 180 min sorption time, 300 rpm mixing speed, 2 w/v % ion strength and 8 g/L dose of sorbent. With recoveries above 80% synthesized copolymer could be an efficient sorbent for trace lindane in water.

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