UNIVERSITI TEKNOLOGI MARA

MODIFIED POLYDIMETHYLSILOXANE SURFACE AS SELECTIVE SORBENT FOR THE ISOLATION OF (S)-ISOMER IN CYPERMETHRIN

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PhD

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AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the result of my own work unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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ABSTRACT

In this study, a novel selective sorbent was prepared by immobilization of NBoc-Phenylalanine-NBoc-Histidine (BCPA) chiral selector with modified polydimethylsiloxane (PDMS) surface for the isolation of the (S)-isomer in cypermethrin (CPM) compound prior to its determination by gas chromatography-micro electron captured detector (GC-μECD). Characterization of the newly synthesized sorbent material was performed using attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR), X-ray photoelectron spectroscopy (XPS), nuclear magnetic resonance (NMR), emission scanning electron microscopy (FESEM), atomic force microscopy (AFM), and Tensiometer (TM) to correlate sorbent characteristics with their performance. ATR-FTIR and NMR studies indicate strong hydrogen bonding interactions between polydimethylsiloxane and NBoc-Phenylalanine-NBoc-Histidine chiral selector (BCPA) immobilization. XPS indicates the elemental composition and chemical state of the chiral selector that exists within the immobilized structure. The applicability of the new sorbent for the isolation of (S)-isomer from alpha cypermethrin (ACPM) standard compound was examined by direct solid-liquid interface adsorption method followed by GC-μECD analysis. Response surface methodology (RSM) was applied to assist the optimization of both adsorption and GC methods. The enantioseparation of the CPM compound was performed on a CycloSil B capillary column. The calibration curve was linear in the range of 0.020 - 1.200 μgmL^{-1} with correlation coefficient (R^2) of 0.9612. Limit of detection and limit of quantification were 0.467 and 1.41 μgmL^{-1}, respectively, and acceptable recoveries were achieved in the range of 66.44 to 118.82 %. Results show that the incorporation of NBoc-Phenylalanine-NBoc-Histidine chiral selector with modified PDMS surface polymer had provided an enantiomer discriminating capability to the sorbent. It indicates that NBoc-Phenylalanine-NBoc-Histidine chiral selector had specifically exhibited enantio-selectivity for (S)-isomer. An analytical Eco-Scale was then measured as an approach to evaluate the greenness of this analytical methodology. The Eco-Scale score of 77 results from the minimal reagent/solvents used in this analysis proves that this straightforward isolation method can be considered as an excellent green analysis that is highly selective towards (S)-isomer.

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