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DEVELOPMENT AND VALIDATION OF A GC-MS/MS METHOD FOR THE DETECTION OF ORGANOCHLORINE AND ORGANOPHOSPHATE PESTICIDES IN COMPOST AND SOIL

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Abstract

Many legacy and emerging chemicals pose significant environmental concerns due to their persistence, bioaccumulation potential, toxicity, and widespread presence in environment. Among those, are organochlorine (OCs) and organophosphate (OPs) pesticides. This study aimed to develop a GC-MS/MS method for the targeted analysis of selected OCs (1,2,3,4,5,6-hexachlorocyclohexane (HCH) isomers, aldrin, and chlordecone) and OPs (dichlorvos, chlorpyrifos, and chlorpyrifos-methyl). The linearity of the developed method was assessed for selected organochlorine and organophosphate pesticides, yielding good R^2 values (≥ 0.98) for all target compounds except chlordecone. Furthermore, a spiking procedure in sol and compost was developed for OCs and OPs. To quantify OCs and OPs in soil and compost, Soxhlet extraction with toluene was used, followed by clean-up with silica-based chromatographic columns. Good recoveries were obtained for aldrin and chlorpyrifos-methyl. Lower correlation with spiked concentrations for some compounds was likely due to matrix effects. The results showed that GC-MS/MS is an efficient method for the quantification of OCs, and OPs, in complex matrices as soil and compost, highlighting their applicability for environmental monitoring and contamination assessment.

Key words*: GC-MS/MS, compost, soil, clean-up.*

INTRODUCTION

Spiking organic chemicals into soil enables controlled and reproducible experiments to study their interactions with the soil matrix and to validate analytical methods. In the same time, spiking can be used to validate analytical methods for detecting chemicals in soil (Brinco, Guedes, Gomes da Silva, Mateus, & Ribeiro, 2023). High-volume spiking facilitates homogenous distribution of compounds in samples, enhancing reproducibility and analytical method validation (Keighley, Ramwell, Werner & Sinclair, 2021). Furthermore, it is a technique commonly used in environmental science, soil chemistry, and ecotoxicology that is introducing relatively large concentrations of specific organic compounds (van Hall & van Gestel, 2025). In the same time, high-volume spiking ensures a uniform and known concentration across samples, improving reproducibility of lab-based experiments (Brinch, Ekelund & Jacobsen, 2002; Northcott & Jones, 2000). The ageing process of organic chemicals in soil reduces their bioavailability and extractability, influencing their environmental fate and toxicity (Alexander, 2000; Hatzinger & Alexander, 1995; Wang, Schlenk & Gan, 2019). This phenomenon is particularly important for understanding the environmental fate, persistence, and toxicity of pollutants such as pesticides, polycyclic aromatic hydrocarbons (PAHs) (Preda, Mocanu & Tănase, 2024), and other hydrophobic organic compounds (Duan et al., 2015). Many organic chemicals may diffuse into micropores or become tightly associated with soil organic matter, making them harder to extract or degrade (Barriuso, Benoit & Dubus, 2008; Jespersen, Trapp & Kästner, 2024; Ukalska-Jaruga et al., 2023). Furthermore, some organic contaminants can form non-extractable residues through covalent or strong non-covalent interactions with humic substances in soil (Loeffler et al., 2020). For this reason, in order to avoid the formation of non-extractable residues it is recommended that for some organic contaminants, the tests or the analysis with the

spiked soils to be performed no longer than several weeks after the spiking procedures (Northcott & Jones, 2000).

Organochlorine pesticides (OCs), such as HCH isomers, aldrin, and chlordecone, are persistent synthetic compounds previously used in agriculture and now recognized for their environmental persistence and toxicity (Micuți et al., 2018; Pang et al., 2022). Among them, the 1,2,3,4,5,6-hexachlorocyclohexanes (HCHs). HCHs can be considered as model compounds since they are persistent, global pollutants that were banned by the Stockholm Convention (Liu et al., 2017). Other priority organochlorine pesticides are: aldrin, and the chlordecone a synthetic organochlorine pesticide, extensively used in banana plantations of the French West Indies from 1972 to 1993 and which therefore was detected in pumpkin and zucchini in the French Region Aquitaine (Chevallier et al., 2018). Comparing with the HCHs, the persistence of chlordecone in the environment is higher due to its bis-homocubane structure (Momtaz & Khan, 2024) numerous chlorine atoms. Organophosphate pesticides (OPs) are a group of synthetic chemicals used to control insects on crops, animals, and in homes. Among them, dichlorvos is an is an insecticide and acaricide, while chlorpyrifos and chlorpyrifos-methyl are acetylcholinesterase inhibitors and they were extensively used to control insect pests on various crops and in stored grain (Kunwar et al., 2021; Mazari et al., 2020). Reliable quantification of OCs and OPs in complex matrices such as soil and compost requires advanced analytical methods with high sensitivity and specificity. In the case of contaminants suitable for gas chromatography-mass spectrometry methods, the used of tandem mass spectrometry using triple quadrupole analysers (GC-QQQ-MS) is employing the development of multiple reaction monitoring (MRM) methods. MRM method development involves three critical steps: (1) selection of precursor ions, (2) optimization of collision energy, and (3) adjustment of dwell time to balance sensitivity. A higher dwell time increases the sensitivity but also decreases the number of points available for each target peak (Badea, Geana, Niculescu & Ionete, 2020). GC-QQQ-MS is considered a highly sensitive technique for targeted and trace-level analysis (pg to fg range on-column). GC-QQQ-MS allows for the selection of a specific precursor ion, its fragmentation in the collision cell, and the subsequent selection of a fragment ion for detection, significantly reducing chemical noise and enhancing the signal-to-noise ratio. In spite of its sensitivity, the linearity domain of the MRM methods is somewhat limited and depends on the sensitivity of the mass spectrometer, as well on the ionization efficiency of the compound and on matrix effects (Vijaykumar et al., 2025). Usually, the linearity domain of the MRM methods is ranged from sub-ng/mL to µg/mL, depending on the compound and method sensitivity.

This study aims to develop and validate a GC-MS/MS method for quantifying selected organochlorine and organophosphate pesticides in soil and compost. The method includes assessment of linearity, recovery rates, and matrix effects in both sample types.

MATERIALS AND METHODS

Chemicals. Most of the target chemicals (α-HCH (analytical purity: 99%, β-HCH (99 %), γ-HCH (97 %), δ-HCH (99 %), hexachlorobenzene (HCB) (99 %), dichlorvos (99 %) and chlordecone (99 %) were obtained from Sigma-Aldrich (Darmstadt, Germany), while aldrin (99%), chlorpyrifos (99%) and chlorpyrifos-methyl (99%) and triphenyl phosphate (TPP) (99%) were purchased from Dr. Ehrenstorfer (Germany).

Linearity domain test. To evaluate the linearity, two calibration mixtures - one for OCs and one for Ops - were prepared in dichloromethane (DCM).

Table 1. The concentration domains of OCs and OPs tested in the linearity test.

Compound	Tested concentration range (ng/mL)
Aldrin	54-8130
α-HCH	45-6750
β-HCH	57-8625
γ-HCH	58-8700
δ-HCH	45-6825
Chlordecone	50-8812
Dichlorvos	49-7481
Chlorpyrifos	59-8850
Chlorpyrifos-methyl	50-7620

HCB (505 ng/mL) and TPP (505 ng/mL) were used as internal standards for OCs and OPs, respectively. The range of concentration range for OCs and OPs is summarized in Table 1.

Spiking of soil and compost. Approximately 60 g of soil and 60 g of compost were spiked with 25 mL of an acetone solution containing the target OCs and OPs (excluding dichlorvos and chlordecone). Samples were homogenized manually, to accelerate acetone evaporation. The theoretical concentration for the target compound in soil were: 0.09 µg/g for aldrin, 0.08 µg/g for α-HCH 0.04 µg/g for β-HCH, 0.05 µg/g for γ-HCH, 0.05 µg/g for δ-HCH, 0.10 µg/g for chlorpyrifos and 0.09 µg/g for chlorpyrifos-methyl while the theoretical concentration for the target compounds in compost were 0.09 µg/g for aldrin, 0.08 µg/g for α-HCH 0.05 µg/g for β-HCH, 0.05 µg/g for γ-HCH, 0.06 µg/g for δ-HCH, 0.11 µg/g for chlorpyrifos and 0.09 µg/g for chlorpyrifos-methyl.

Extraction and clean-up of the soil and compost samples. Triplicate 10 g portions of each spiked matrix were extracted via Soxhlet using toluene for 24 hours. Internal standards (100 µL of 100 µg/mL HCB for OCs and 100 µL of 100 µg/mL TPP for OPs) were added prior to extraction. Upon evaporation, the clean-up of the extracts was performed both with 3 x 3 mL of n-hexane on SPE columns containing 500 mg silica (55 µm, 70 Å) and with 120 mL hexane on 8 g of silica gel deactivated with 10% water (w/w) packed on glass columns. About 1 ml of toluene was added to the eluates followed by the evaporation of n-hexane. Before GC-MS analysis, the recovery standard (RS), consisting of heptachlor was added.

GC-MS analysis. The target compounds were analysed using an Agilent 7890B gas chromatograph (GC) coupled to Agilent 7010 triple quadrupole mass spectrometer (Agilent Technology, Palo Alto, USA) operated in positive electron ionization (EI) mode (70 eV) and configured in multiple reaction monitoring (MRM). The samples (volume: 1 µL) were injected at 225°C into a PTV injector operated in split less mode, while the flow rate of carrier gas (helium) was 1 mL/min. The target compounds were separated on a Agilent HP-5MS (5% Phenyl Polysilphenylene-siloxane) capillary column (30 m × 0.25 mm × 0.25 µm), using the following oven program: initial temperature of 40°C (5 min isothermal), ramp with 5°C min^{-1} to 110°C (0 min), 20°C min^{-1} to 180°C (0 min), 5°C min^{-1} to 230°C (0 min) and 20°C min^{-1} to 310°C (3 min isothermal).

RESULTS AND DISCUSSIONS

Assessment of linearity domains. The linearity domains across two orders of magnitude concentrations were assessed. The respective domains were assessed as linear if the R-Square values (R^2) of the linear regressions were at least 0.98 or higher. For aldrin, linearity was observed in the concentration range of 54–1622 ng/mL ($R^2 \geq 0.98$). Higher concentrations (2710 ng/mL and 8130 ng/mL) fell outside the linear range (Figure 1).

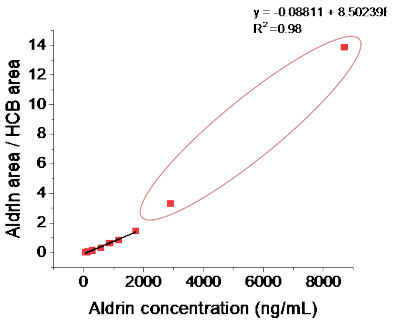

Figure 1. Linearity domain of the aldrin for developed GC-MS-MS method

Assessment of linearity domains for hexachlocylohexanes (HCHs) is presented in Figure 2. The linearity domain for α-HCH was calculated in the range 45-1347 ng/mL, while the concentrations of 2250 ng/mL and 6750 ng/mL were considered as outside of linear domain (Figure 2A). The linearity domain for β-HCH was defined in the range 57-1721 ng/mL, while the concentrations of 2875 ng/mL and 8625 ng/mL were outside of the linear domain (Figure 2B). The linearity domain for lindane (γ-HCH) was measured in the range 58-1736 ng/mL, while the concentrations of 2900 ng/mL and 8700 ng/mL were considered as outside of linear domain (Figure 2C). Also, the linearity domain for δ-HCH was defined in the range 45-1362 ng/mL, while the concentrations of 2275 ng/mL and 6825 ng/mL were outside of the

linearity domain (Figure 2D). Overall, similar linear ranges were observed for all HCH isomers, likely due to their comparable molecular structures and physicochemical properties. The assessment of linear domain for chlordecone in the range 58-2937 ng/mL gave a week R-square value (R^2 = 0.786) comparing with the other OCs, even upon exclusion of its higher concentration of 8812 ng/mL (data not shown). This indicates that hexachlorobenzene is not a good internal standard for chlordecone in GC-MS-MS determinations and Hexabromobenzene (HBB) or TPP may be preferred. The assessment of linearity domains for OPs is presented in Figure 3. Dichlorvos gave a good linearity domain in the range 49-2493 ng/mL (R^2 = 0.99) showing the highest linearity domain from all the target compounds (Figure 3A). This may be attributable to its lower molecular weight (M.W = 220.97) comparing with the rest of the target compounds. The linearity domain for chlorpyrifos was calculated in the range 59-1177 ng/mL, while the last three concentrations of 1766 ng/mL, 2950 ng/mL, 2540 ng/mL and 8850 ng/mL were considered as outside of linear domain (Figure 3B). The linearity domain for chlorpyrifos-methyl was defined in the narrow range 254-1520 ng/mL, while both the last two lowest concentrations (50 ng/mL and 101 ng/mL) and last highest concentration (7620 ng/mL) being calculated as outside linear domain (Figure 3C). Overall, the results of the linearity test shown extensively linearity domain for all the target compounds and this shown that the developed GC-MS-MS method was validated from linearity point of view.

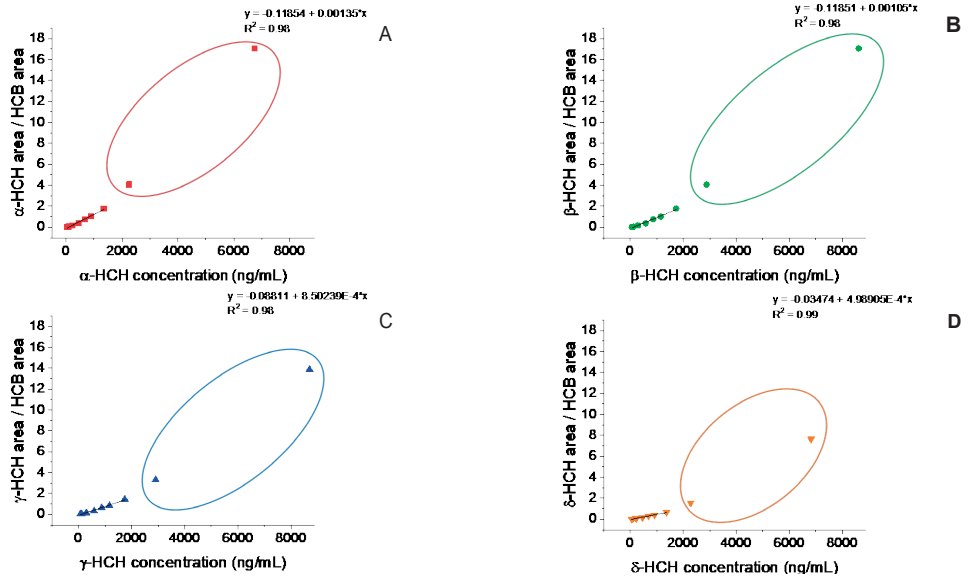

Figure 2. Linearity domain for α-HCH (■))(A), β-HCH (●) (B), γ-HCH (▲) (C) and δ-HCH (▼) (D) for the developed GC-MS-MS method

Assessment of concentrations in soil and compost. The concentrations of OCs and OPs in soil samples are presented in Table 2 and those values are comparing with those of the theoretical spiked concentrations.

Aldrin (117.20%) and chlorpyrifos-methyl (116.08%) recoveries in soil were consistent with their theoretical concentrations, indicating effective extraction and minimal matrix interference for these compounds.

For the rest of the OCs and OPs the concordance with the spiked concentration is weak (Table 2). The high recoveries may indicate a strong matrix enhancement for some compounds (e.g. γ-HCH).

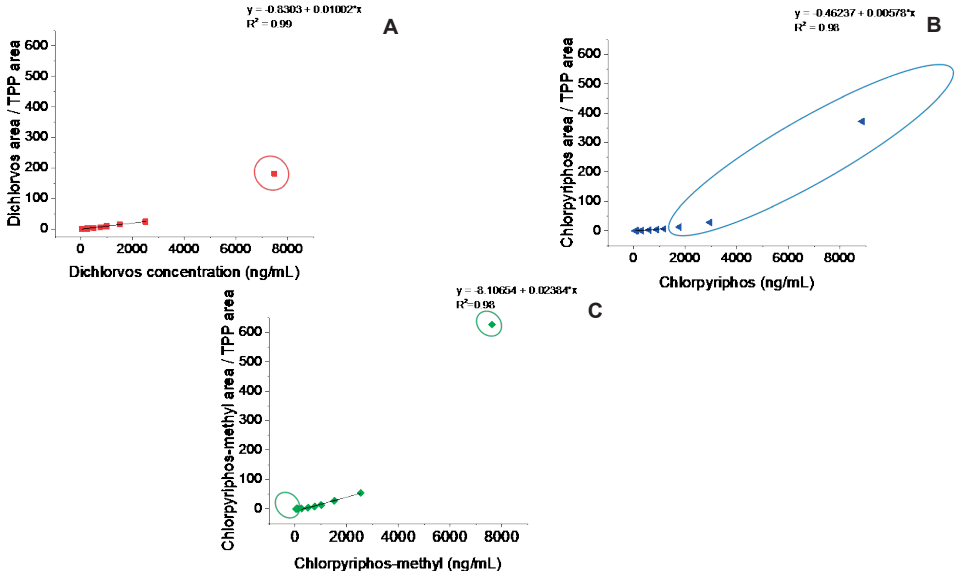

Figure. 3. Linearity domain for diclorvos (■) (A), chlorpyrifos (◀) (B), and chlorpyrifos-methyl (♦) (C) for the developed GC-MS-MS method

Table 2. Concentration of OCs and OPs pesticides determined in soil

Compound	Theoretical concentration (µg/g)	Measured concentration in sol (µg/g)	Recovery (%)
Aldrin	0.09	0.11 ± 0.002	117.20
α-HCH	0.08	0.05 ± 0.004	60.50
β-HCH	0.04	0.06 ± 0.01	134.86
γ-HCH	0.05	0.16 ± 0.01	314.24
δ-HCH	0.05	0.07 ± 0.01	127.66
Chlorpyrifos	0.10	0.15 ± 0.11	150.03
Chlorpyrifos-methyl	0.09	0.10 ± 0.05	116.08

Table 3. Concentration of OCs and OPs pesticides determined in compost

Compound	Theoretical concentration (µg/g)	Measured concentration in compost (µg/g)	Recovery (%)
Aldrin	0.10	0.08 ± 0.01	84.07
α-HCH	0.08	0.05 ± 0.002	66.50
β-HCH	0.05	0.07 ± 0.002	141.36
γ-HCH	0.05	0.08 ± 0.01	136.49
δ-HCH	0.06	0.07 ± 0.01	124.20
Chlorpyrifos	0.11	0.39 ± 0.01	363.68
Chlorpyrifos-methyl	0.09	0.11 ± 0.004	119.51

The discrepancies between measured and spiked concentrations, particularly in compost, are likely attributable to matrix effects such as co-extracted interferences causing ion suppression or enhancement in the mass spectrometer. (Wu & Ding, 2023). These effects can suppress or enhance the ionization of target OCs and OPs in the mass spectrometer detector, leading to inaccurate results. The concentrations of OCs and OPs in compost samples are presented in Table 3 and those values are comparing too with those of the theoretical spiked concentrations. Here too, the concentration of aldrin (0.08 ± 0.01 µg/g, recovery of 84.07%) and chlorpyrifos-methyl (0.11 ± 0.004 µg/g, recovery of 119.51%) are in good concordance with spiked concentrations. For the rest of the OCs and OPs, the concordance of measured concentrations with spiked concentrations is weak too, probably due to matrix effects. Non-isotopically labelled internal standards may be insufficient to fully correct for matrix effects and losses during sample preparation. Isotopically labelled analogues are recommended for improved quantification accuracy, as supported by prior studies (Badea, Lundstedt, Liljelind & Tysklind, 2013) (Peña, Sosa, Hilber, Escobar & Bucheli, 2024).

CONCLUSIONS

A GC-MS/MS method was successfully developed and applied for the quantification of selected organochlorine and organophosphate

pesticides in soil and compost. The evaluation of linearity domains gave good R^2 values for all the target compounds excepting for chlordecone. Dichlorvos exhibited the broadest linear range, likely due to its lower molecular weight and favourable ionization characteristics. Overall, the results of the linearity test shown extensively linearity domain for all the target compounds. With respect of solid matrices analyses, aldrin and chlorpyrifos-methyl showed recoveries consistent with their theoretical spiked levels in both matrices, indicating reliable quantification under the proposed method. Deviations in recovery for other analytes were attributed to matrix effects, highlighting the need for more robust internal standardization. In order to overcome those matrix effects, the use of isotopic labelled internal standards (contains atoms of ^{2}H, ^{13}C) is expecting to lead to higher accurate concentration values for a variety of OCs and OPs. Overall, the study demonstrates the feasibility of applying a GC-MS/MS method for multi-residue pesticide analysis in complex environmental matrices.

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