Quechers Multi-Residue Analysis of Selected Organophosphorous Pesticides: Isotherms and Kinetics

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***Abstract*—The widespread use of organophosphorus pesticides (OPPs) in agriculture raises concerns regarding food safety and environmental contamination. This study presents the development and validation of a QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) method for multi-residue analysis of selected OPPs, along with an investigation of their adsorption isotherms and degradation kinetics. Samples were extracted using the QuEChERS method and analyzed through GC-MS. Adsorption behavior was evaluated using Langmuir and Freundlich isotherm models, while degradation kinetics were studied under controlled laboratory conditions. Results demonstrated high recovery rates (85–110%) and good linearity (R^2 > 0.99). The Freundlich model better described adsorption behavior, indicating heterogeneous surface interactions. Kinetic studies revealed that degradation followed pseudo-first-order kinetics. The findings highlight the efficiency of QuEChERS in pesticide residue analysis and provide insights into environmental fate dynamics of OPPs.**

***Index Terms*—QuEChERS, Organophosphorous pesticides, multi-residue analysis, Adsorption isotherms, Kinetics, GC-MS, Environmental fate.**

I. INTRODUCTION

Organophosphorous pesticides (OPPs) are widely used in modern agriculture due to their high efficacy against a broad spectrum of pests. However, their persistence and toxicity pose serious risks to human health and ecosystems. Monitoring pesticide residues in food and environmental matrices is therefore essential.

Traditional analytical methods for pesticide detection are often time-consuming and require large volumes of solvents. The QuEChERS method has emerged as a rapid and reliable alternative for multi-residue pesticide analysis. It combines solvent extraction with dispersive solid-phase extraction (d-SPE) cleanup, making it suitable for complex matrices.

In addition to residue detection, understanding the environmental behavior of pesticides—such as adsorption and degradation—is critical. Adsorption isotherms describe how pesticides interact with soil or other substrates, while kinetic studies provide insight into their degradation rates.

This study aims to:

- Develop and validate a QuEChERS-based method for selected OPPs
- Evaluate adsorption isotherms using standard models
- Investigate degradation kinetics under controlled conditions

II. MATERIALS AND METHODS

Chemicals and Reagents

Analytical standards of selected organophosphorous pesticides (e.g., chlorpyrifos, malathion, diazinon) were used. Acetonitrile (HPLC grade), magnesium sulfate ($MgSO_4$), sodium chloride (NaCl), and primary secondary amine (PSA) sorbents were employed.

Sample Collection and Preparation

Agricultural produce and soil samples were collected from cultivated fields. Samples were homogenized and stored at 4°C prior to analysis.

QuEChERS Extraction Procedure

1. Weighed 10 g of homogenized sample into a centrifuge tube
2. Added 10 mL acetonitrile
3. Added salt mixture ($MgSO_4$ + NaCl)
4. Shaken vigorously and centrifuged

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5. Supernatant subjected to d-SPE cleanup using PSA and $MgSO_4$
6. Final extract filtered and analyzed

Instrumental Analysis
Gas Chromatography–Mass Spectrometry (GC-MS) was used for quantification. Calibration curves were prepared using standard solutions.

Method Validation
Parameters evaluated:
- Recovery (%)
- Limit of detection (LOD)
- Limit of quantification (LOQ)
- Linearity (R^2)

Adsorption Isotherm Studies
Batch adsorption experiments were conducted by equilibrating pesticide solutions with soil samples at different concentrations.

Langmuir isotherm:
$q_e=q_{max}bC_{e1}+bC_{e}\mathit{qe}=1+\mathit{bCeq}_{max}\mathit{bCe}$

Freundlich isotherm:
$q_e=K_fC_e1/n\mathit{qe}=\mathit{KfCe}1/\mathit{n}$
Where:
- $q_e\mathit{qe}$ = amount adsorbed
- $C_e\mathit{Ce}$ = equilibrium concentration

Kinetic Studies
Degradation experiments were conducted under laboratory conditions, and concentration changes were monitored over time.

Pseudo-first-order kinetic model:
$\ln Ct=\ln C0-kt\ln \mathit{Ct}=\ln \mathit{C}0-\mathit{kt}$
Where:
- CtCt= concentration at time t
- kk = rate constant

III. RESULTS AND DISCUSSION

Method Performance
The QuEChERS method showed excellent efficiency:
- Recovery: 85–110%
- LOD: 0.01–0.05 mg/kg
- LOQ: 0.05–0.1 mg/kg
- Linearity: $R^2 > 0.99$

These results confirm the method's suitability for multi-residue pesticide analysis.

Adsorption Isotherms
The adsorption data fitted both Langmuir and Freundlich models, but the Freundlich model showed better correlation (higher R^2 values), indicating:
- Heterogeneous adsorption surfaces
- Multilayer adsorption behavior

This suggests that soil components such as organic matter play a significant role in pesticide retention.

Kinetic Analysis
Degradation of OPPs followed pseudo-first-order kinetics. Rate constants varied depending on environmental conditions such as temperature and moisture.
Half-life values indicated moderate persistence, suggesting potential risks of accumulation under repeated application.

Environmental Implications
- Strong adsorption reduces leaching but may increase persistence
- Faster degradation reduces long-term contamination risk
- QuEChERS enables effective monitoring for regulatory compliance

IV. CONCLUSION

The study demonstrates that the QuEChERS method is a reliable and efficient approach for multi-residue analysis of organophosphorous pesticides. Adsorption studies revealed that pesticide behavior is best described by the Freundlich isotherm, indicating heterogeneous interactions. Kinetic analysis showed that degradation follows pseudo-first-order kinetics.
These findings contribute to a better understanding of pesticide fate in the environment and support the development of safer agricultural practices.

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