METHODICAL ASPECTS OF COLUMN EXPERIMENTS FOR ORGANIC COMPOUNDS BASED ON IMIDACLOPRID STUDIES

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PURPOSE OF THE STUDY

The transport of pollutants in groundwater takes place based on numerous phenomena and processes. It can be estimated based on data obtained from the literature or based on the results of laboratory or field studies. using, for example, batch or column experiments. Although nowadays column experiments are commonly used, there are still no detailed guidelines or procedures for their performance. As a result, the comparison of the obtained results from column experiments performed by different scientists is difficult, and sometimes even impossible. Hence, this article presents methodical aspects of column experiments, performed to investigate transport parameters of organic compounds, based on the example of studies with imidacloprid. The studies included an important stage of planning experiments, taking into account the factors affecting the uncertainty of the obtained results.

COLUMN EXPERIMENT STAND

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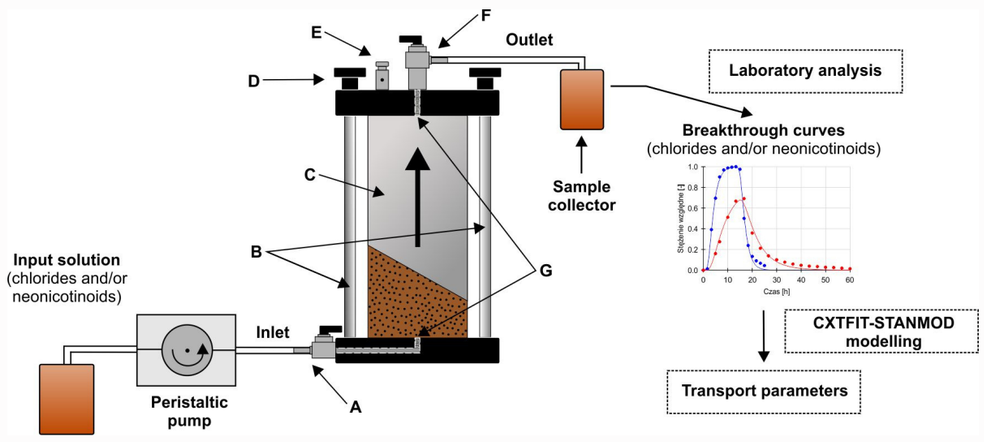

Explanation:
A — inlet valve,
B — mounting rods,
C — steel column filled with soil,
D — mounting bolts,
E — air discharge valve,
F — exhaust valve,
G — steel filter.

METHODOLOGY

The sources and shares of uncertainty arising at various stages of column experiments have not yet been clearly identified, and their impact on the obtained values of the transport parameters is still not recognized. Before starting the planning of column experiments, the main factors influencing the uncertainty of the obtained results were identified to take them into account in the experiments and minimize their influence on the results of the research.

The study consisted of a few stages. Firstly, test stands were designed. Then, pilot experiments of column studies with the use of a conservative tracer (chloride) for three different soils S-A, S-B, S-C (Tab. 1) prepared in the laboratory were conducted. The purpose of this stage was to verify whether the prepared stand meets the assumed criteria and allows for obtaining reliable test results. Finally, in the third stage of the research the reproducibility of the column experiments was assessed by conducting imidacloprid migration studies simultaneously in two identical columns at two different concentration levels, using another artificial soil S-III (Tab. 1) representing the aquifer, prepared in the laboratory.

In principle, each column experiment was carried out as follows — a solution of imidacloprid and chlorides of an appropriate known concentration was delivered by a peristaltic pump to the column filled with soil. Samples were taken at the column outlet at specified intervals and further analyzed in the laboratory to determine the concentration of the neonicotinoids tested (LC-MS/MS method) and chlorides (titration method). The results were further used to determine the breakthrough curves (BTCs) of neonicotinoids and chlorides. Finally, analysis of the BTCs in the CXTFIT-STANMOD software allowed to determine the transport parameters of the tested substances, based on the deterministic equilibrium of the convection–dispersion equation, solving the inverse problem.

Table 1. Artificial soils used in the column experiments

Soil	Chlorides			Imidacloprid
	S-A	S-B	S-C	S-III
Sand [%]	94	100	100	97
Silt and Clay [%]	6	0	0	3
Organic matter [%]	0	0	0	0.2

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FACTORS AFFECTING THE UNCERTAINY OF RESUTS

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Influencing factors to the uncertainty of the result	How the factor is included in the experiment
Preparation of the test stand	
Material from which individual elements of the station are made	Reactivity assessment — experiments with deionized water and deionized water with the addition of the test substance
Preparation of soil samples	Ensuring homogeneity — uniform procedure for the preparation of test samples, comparative homogeneity tests
Laboratory experiments	
Analytical methods	Estimate method uncertainty in the laboratory using standard procedures
Injection solution	Ensuring the homogeneity and stability of the chemical composition — a uniform methodology for the preparation of solutions, stability tests
Tracer injection method	Pulse and continuous injection
Repeatability and reproducibility of experiments	Repeated/parallel experiments
Storage and transport of samples for the laboratory	Short storage time of samples in the refrigerator, analysis of samples as soon as possible from the experiment

RESULTS

In the case of soil S–III, the phenomenon of sorption was observed, which reflects the different shapes of the breakthrough curves for chlorides and imidacloprid (Fig. 1). Soil S–III, apart from clay minerals, was also characterized by a slight organic matter content (0.2%), which confirmed the observations that the presence of clay minerals and organic matter in the soil increases the sorption of neonicotinoids.

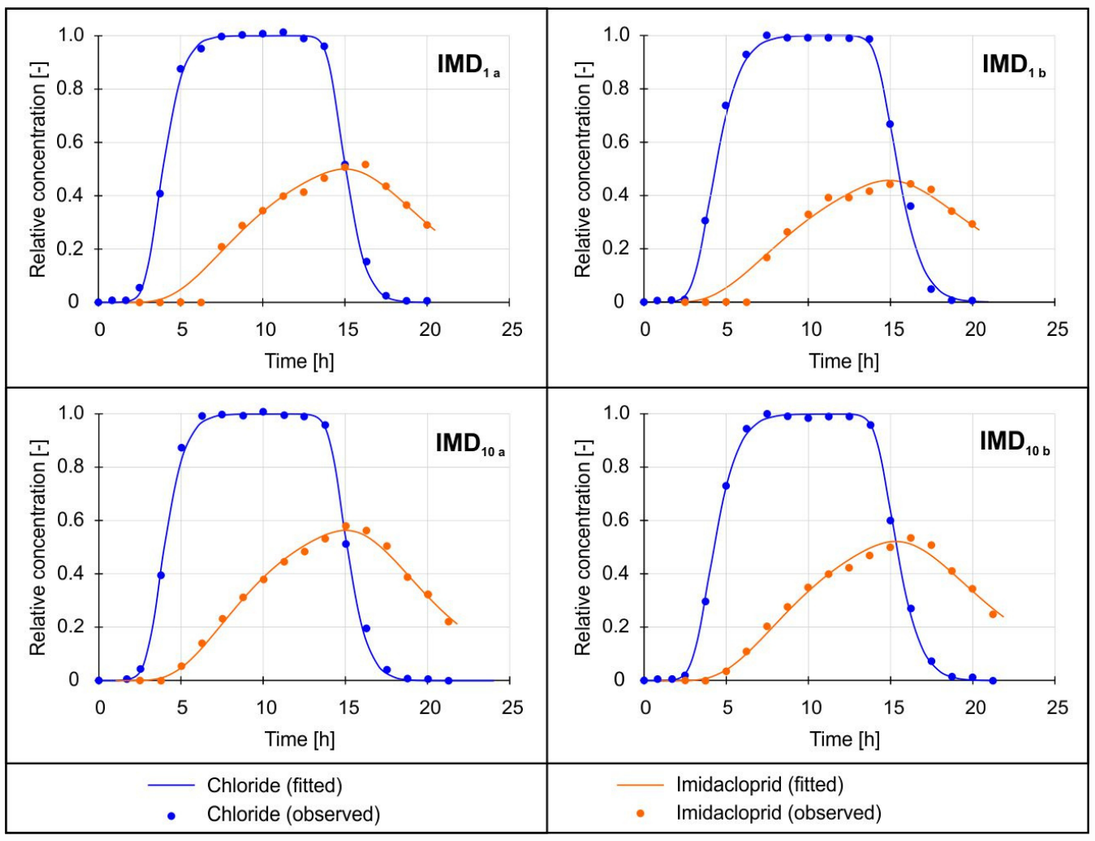
Based on the obtained results, it was found that the prepared stand meets the assumed criteria and enables obtaining reliable and comparable test results. The repeatability of the methodology of preparing and performing column experiments was also successfully confirmed. For both the lower and higher concentration levels, in both replicates very similar values of the R–factors were observed — 2.84 and 2.83 as well as 2.62 and 2.68, respectively — which means a difference of less than 3%.

Table 1. Transport parameters of chlorides and imidacloprid

Tracer[1)]	Column	Transport parameters				r^2 [–]	MSE [–]
		U [cm/min]	D [cm^2/min]	R ± SD [–]	μ [1/d]		
Cl^-_1	a	0.065	1.82	1	-	0.999	0.00022
	b	0.058	2.00			0.996	0.00076
Cl^-_{10}	a	0.065	1.88			0.998	0.00045
	b	0.060	2.02			0.998	0.00033
IMD_1	a	0.023	2.62	2.84±0.20	0.0047	0.958	0.00038
	b	0.021	3.10	2.83±0.29	0.0045	0.981	0.00038
IMD_{10}	a	0.025	2.34	2.62±0.07	0.0042	0.993	0.00028
	b	0.022	2.45	2.68±0.12	0.0040	0.985	0.00051

[1)] Indices 1 and 10 in the case of IMD indicate lower (0.858 and 0.771 μg/L) and higher (8.85 and 7.88 μg/L) concentration levels, respectively; for chlorides, the concentration was 500 mg/L.

Fig. 1. Breakthrough curves for chlorides and imidacloprid for higher and lower concentrations (labeled 1 and 10, respectively)



For more data

Considering the risk posed by organic contaminants such as imidacloprid to the environment, it is important to know the migration ability of these compounds in the soil–water system. The use of column experiments is an essential element for the correct assessment of their fate in the environment. Hence, a lot of emphases was placed on the appropriate methodology of performing column experiments, taking into account factors that can affect the uncertainty of the research results.

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