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# Optimized column outflow experiments for the assessment of effective release rates of contaminants from porous media

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Identification and quantification of effective release and transport processes of reactive substances in soils can only be achieved if experimental boundary conditions are well known and if the output contains sufficient information. Column outflow experiments offer the opportunity to conduct experiments on the one hand under controlled and reproducible conditions. On the other hand, it is possible to vary the boundary conditions in a way that the result is sensitive to the wanted parameters. In standard column outflow experiments, the complete information on the release and transport process is carried by the shape, i.e. symmetry and curvature, of the breakthrough curve (BTC) and its relation to the flow of the liquid phase, indicated by the transport of a conservative substance. Quantification is frequently done by the application of inverse methods. It has been shown that inversely estimated parameters of mobile/immobile water flow models can not uniquely be identified by means of a breakthrough curve with a pulse-type input boundary condition. This is equally valid for reactive substances, if interaction- and rate parameters have to be estimated simultaneously. Uniqueness problems of this type can be avoided by the imposition of a flow interruption: If the exchange between contaminant source/sink and bulk liquid phase is limited by a slow mass transfer, the actual aqueous concentration is higher than the equilibrium concentration during an arrival wave of a contaminant and lower than the equilibrium concentration during an elution curve. Thus, an elongated residence time due to a flow interruption will result in a decrease (arrival wave) or increase (elution curve) of the effluent concentration. It is, however, not an easy task to identify the optimal flow rates and the optimal point of time and duration of the flow interruptions in order to observe kinetic limitations. Moreover, routine risk assessment and material classification may demand the obedience of additional constraints, such as limited time. We therefore performed numerical experiments in order to optimize the experimental conditions, which allow the distinction between equilibrium and non-equilibrium release. For short term column experiments we found, that the application of flow interruptions along with two different flow velocities can be applied to avoid uniqueness problems with respect to identification of partitioning coefficient and mass transfer rate. Morevoer, we found that the effect of non-equilibrium is to be observed only in a small range of the ratio of the transport time scale to the reaction time scale, given by the Damköhler-Number. We conducted column experiments to test this optimized experimental design for its suitability for the identification and quantification of rate-limited contaminant release. We use materials polluted with organic and inorganic contaminants originating from different soils, sites and materials (Coke oven sites, abandoned industrial sites, destruction debris, municipal waste incineration ash). Repacked soil columne were percolated under saturated and unsaturated conditions, which were subjected to multiple flow interruptions and different flow velocities. The breakthrough of a conservative tracer is used to characterize the advective-dispersive flow regime. Column outflow is analyzed, among others, for target contaminants, dissolved organic carbon (DOC), turbidity and UV-VIS absorptivity. Data evaluation and process identification and quantification was based on numerical inversion. With the developed experimental desing, we were able to successfully identify and quantify effective release kinetics.