Health and Safety Executive

Lessons learned: A regulators experience of applying the OECD 106 evaluators checklist

HSE

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Introduction

Background

The OECD 106 checklist[1] was developed by EU Member State experts in collaboration with ECPA and EFSA to provide supplementary information to aid consistency in the conduct, evaluation and reporting of batch sorption/desorption studies.

The checklist is a tool for applicants and regulators to help identify where deviations from the OECD 106 guideline[2] could lead to significant and systematic errors in fitted parameters. It is not intended to replace the quality checks outlined in the OECD 106 guideline.

K_{foc} and 1/n are critical input parameters to FOCUS modelling for groundwater and surface water. The models are sensitive to these input parameters. For example, a 10 % change in 1/n can lead to a 100 % change in PEC_{GW}. Therefore, robust studies performed to standardised guidelines are needed to have confidence in the modelled predictions.

Aim

HSE's Chemicals Regulation Division has been applying the OECD 106 checklist to pesticide active substance evaluations for several years. Presented here are the key lessons learned from a regulatory perspective.

OECD 106

Direct Method:

- Both mass concentration in the liquid phase (C_{aq}) and mass sorbed per mass of dry soil (Cs) at equilibrium are measured analytically.

Indirect Method:

- Mass sorbed per mass of dry soil (C_s) at equilibrium determined by calculation.

$$C_s = \frac{(\text{Initial concentration} - C_{aq}) \times \text{volume of solution}}{\text{Mass of soil}}$$

Active substance evaluations:

- The soil adsorption coefficient (K_f) is normally derived using the 'indirect method' based on solution depletion.
- However, decisions made regarding the study set-up can result in the study not fulfilling key parts of the checklist and therefore not being relied upon during the active substance evaluation.
- The checklist highlights some of the most important quality checks and helps regulators confirm the acceptability of the fitted sorption parameters (K_f and 1/n) .

Methods

Sorption parameters

Data for eight soils were tested according to the indirect and the direct method outlined in OECD 106. The key quality criteria, outlined in the OECD 106 checklist, were used to confirm the acceptability of the fitted sorption parameters (K_f and 1/n) and the appropriateness of their use in regulatory models.

Direct vs Indirect: PECgw

The fitted sorption parameters determined via the direct and indirect method were compared. Focus PEARL v4.4.4 was used to estimate PECgw concentrations. The simulations were performed on winter cereals with 1000 g a.s./ha applied in autumn.

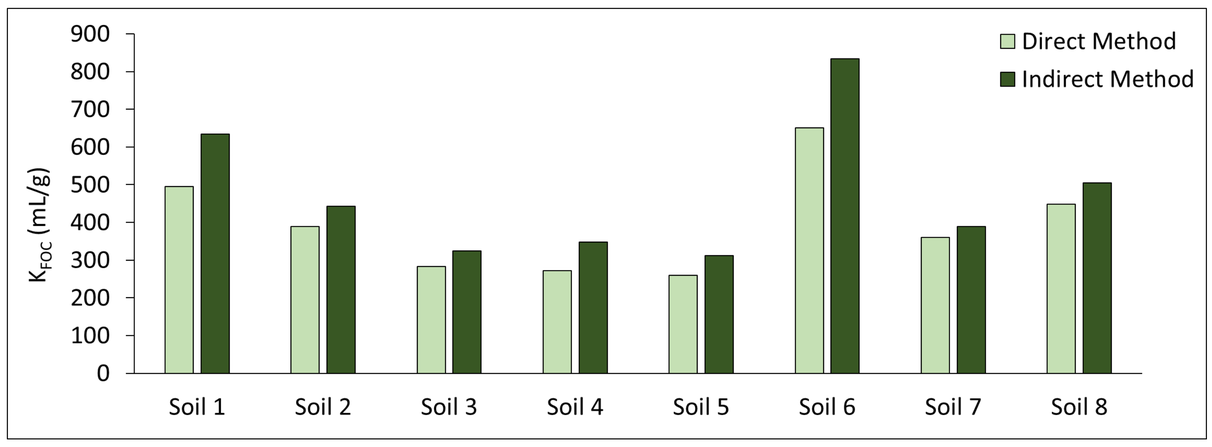
Quality Checks

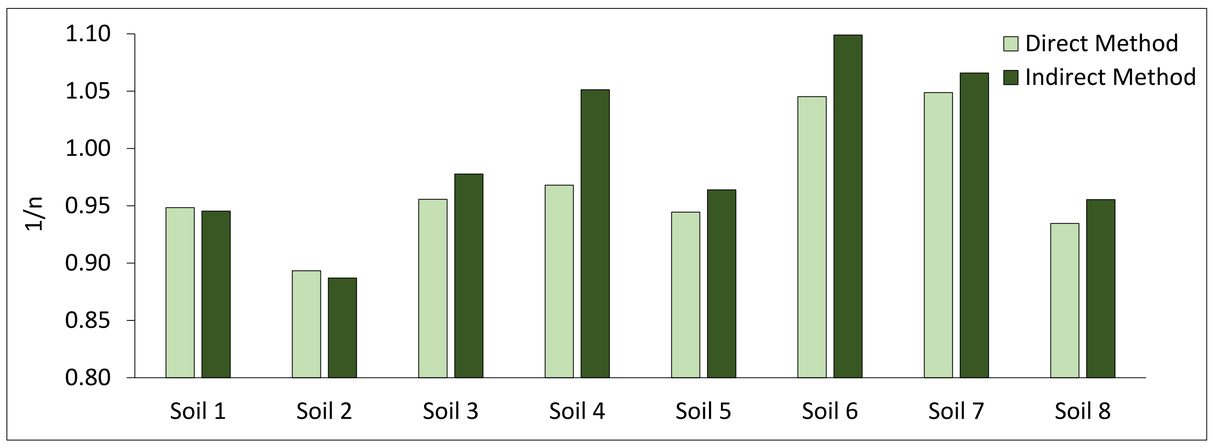
	KEY QUALITY CRITERIA				
	Indirect method only		Used for both direct and indirect method		
Soil	K_{fE}/K_f	Kd x soil/solution ratio	Mass balance	% absorbed	Visual fit
Soil 1	1.33	2.02-3.00	81.2	58.0	Good
Soil 2	1.26	0.79-3.00	85.3	54.6	Poor
Soil 3	1.22	0.83-1.25	91.0	46.1	Good
Soil 4	1.39	1.47-3.10	81.5	58.6	Good
Soil 5	1.16	1.95-3.97	90.9	64.4	Acceptable
Soil 6	1.34	1.00-4.06	86.6	61.0	Poor
Soil 7	1.00	4.00-9.25	100.5	81.8	Good
Soil 8	1.11	1.04-3.50	95.0	56.3	Poor

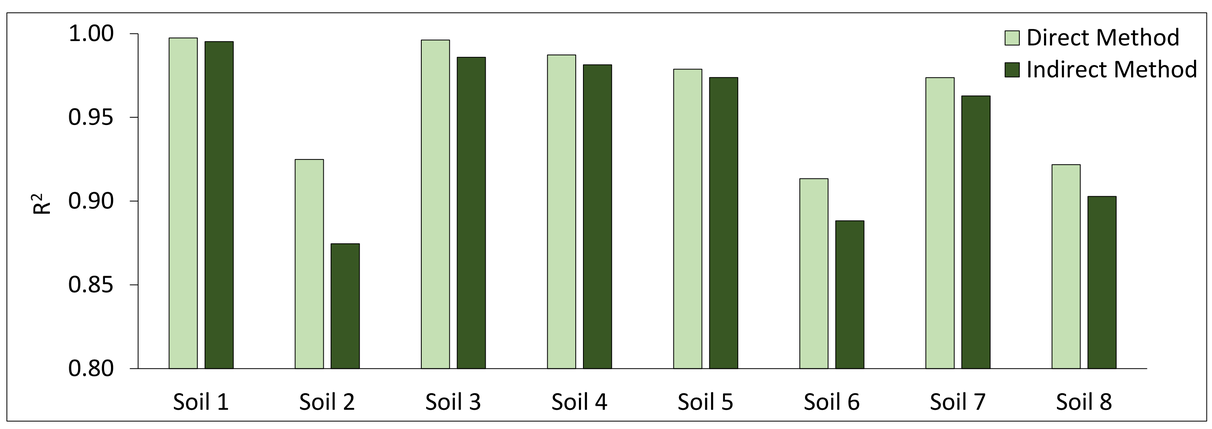
Soils excluded for indirect method; Soils excluded for indirect and direct method; Soils included for indirect and direct method

K_{fE}/K_f: Check of the effect of systematic errors as a result of loss of test substance. Only used for indirect method.

Direct v Indirect







Groundwater Modelling

	PEC_{GW} (µg/L)			
	All soils included		Soils failing quality criteria excluded	
Scenario	Direct	Indirect	Direct	Indirect
Sorption parameters	K_{OM} = 218.5 1/n = 0.967	K_{OM} = 259.8 1/n = 0.993	K_{OM} = 198.3 1/n = 0.967	K_{OM} = 217.8 1/n = 0.991
Châteaudun	0.000	0.001	0.005	0.004
Hamburg	0.055	0.051	0.166	0.131
Kremsmünster	0.033	0.032	0.101	0.083
Okehampton	0.074	0.068	0.216	0.170

Conclusions

Using the direct or indirect method can have significant effects on the fitted sorption parameters (Kf and 1/n). K_{foc} is systematically higher using the indirect method. Interestingly, 1/n also tends to be systematically higher.

The higher 1/n seems to compensate for the higher K_{foc} in the modelling - so even though the parameters values are very different, the results of the groundwater modelling are much more similar when soils failing quality criteria are not excluded.

The groundwater modelling results demonstrate the importance of using high quality data. The PECs are much higher and would result in a different regulatory decision when soils failing the quality criteria are removed.

Key Lessons Learned: Regulatory Perspective

- Altering the study design (*i.e.* 1:1 soil:solution ratio, shorter equilibrium time) could result in the more cost-effective indirect method study being accepted.
- In cases where % adsorption is low, use a 1:1 soil:solution ratio to maximise sorption. This helps with improving other quality control checks.
- Use a short equilibrium time for degrading substances to maximise mass balance measurements.
- Ensure mass balance is calculated correctly by measuring the test item and not % radioactivity and do not include NERs in mass balance calculations.
- When using the direct method, ensure the liquid entrained in the soil pellet is accounted for.
- For the indirect method, the soil/solution ratio is calculated from the soil mass and the total liquid volume present in the original soil suspension.
- For the direct method, the ratio is derived after centrifugation, and is therefore calculated as the ratio between the soil mass divided by the residual moisture volume in the soil pellet.

References

1. EFSA, 2017. Technical report on the outcome of the pesticides peer review meeting on the OECD 106 evaluators checklist. EFSA supporting publication 2017:EN-1326. 18 pp.doi:10.2903/sp.efsa.2017.EN-1326.
2. OECD guideline 106: Adsorption - desorption using a batch equilibrium method. 21 January 2000.