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Evaluation of metal–organic framework-based adsorbents for preconcentration of pesticides from water samples

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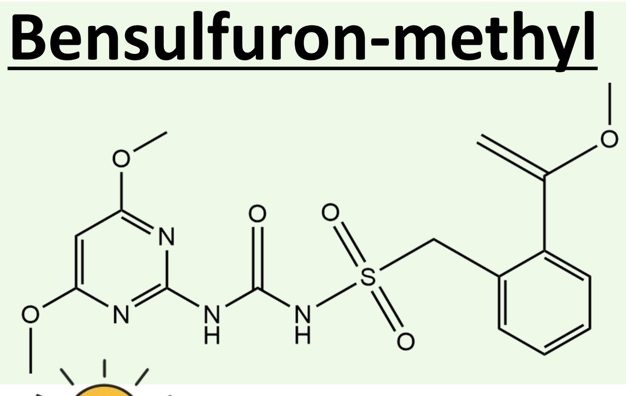
Introduction

Bensulfuron-methyl



- A sulfonylurea herbicide, widely used in paddy fields.
- Because of its high toxicity to aquatic plants, monitoring in water is necessary.

Sample pretreatment is required because analytes are present at very low concentrations and the matrix is complex.

Solid-Phase Extraction (SPE)

- An effective method for preconcentration and purification.
- Its performance depends on the **adsorbent**.

Metal-Organic Frameworks (MOFs)

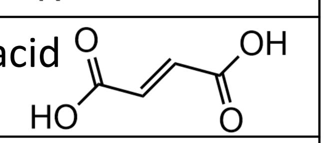
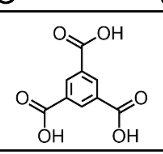
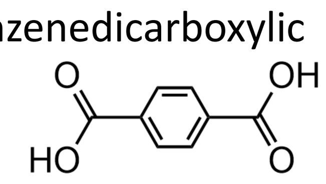
Hybrid materials constructed from metal ions and organic ligands

Versatile combinations enable adsorption property design
- Physicochemical properties, polarity, and pore size

<This study> Several MOFs were synthesized and evaluated as SPE adsorbents for bensulfuron-methyl.

Experimental

Preparation of MOFs : solvothermal synthesis

MOF	Metal salt	Organic linker
ZIF-8 (Zeolitic Imidazolate Framework-8)	$Zn(NO_3)_2 \cdot 6H_2O$	2-methylimizole 
MOF-801	$ZrOCl_2 \cdot 8H_2O$	Fumaric acid 
MIL-100(Fe) (Materials of Institute Lavoisier-100(Fe))	$FeCl_3 \cdot 6H_2O$	Terephthalic acid 
UiO-66 (University of Oslo-66)	$ZrCl_4$	1,4-Benzenedicarboxylic acid 
MIL-53(Fe) (Materials of Institute Lavoisier-53 (Fe))	$FeCl_3 \cdot 6H_2O$	
MIL-53(Al) (Materials of Institute Lavoisier-53 (Al))	$Al(NO_3)_3 \cdot 9H_2O$	

Example of **MIL-53(Al)**

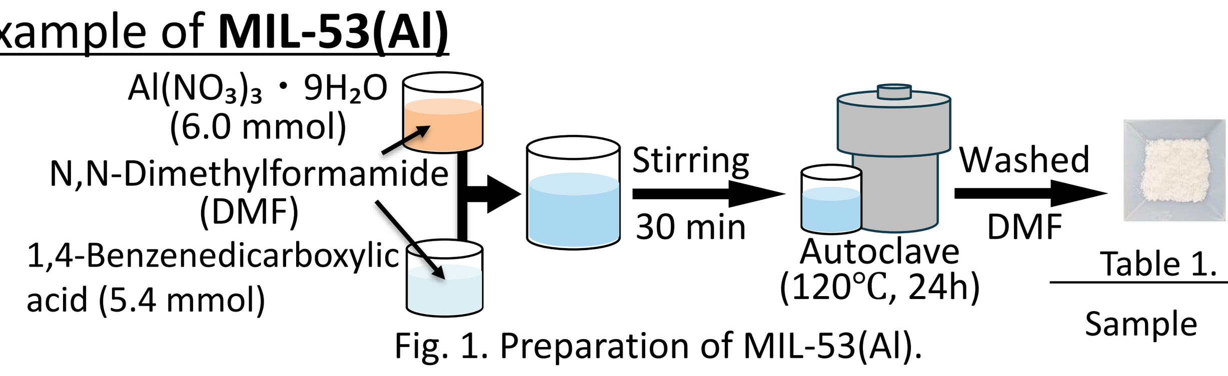


Fig. 1. Preparation of MIL-53(Al).

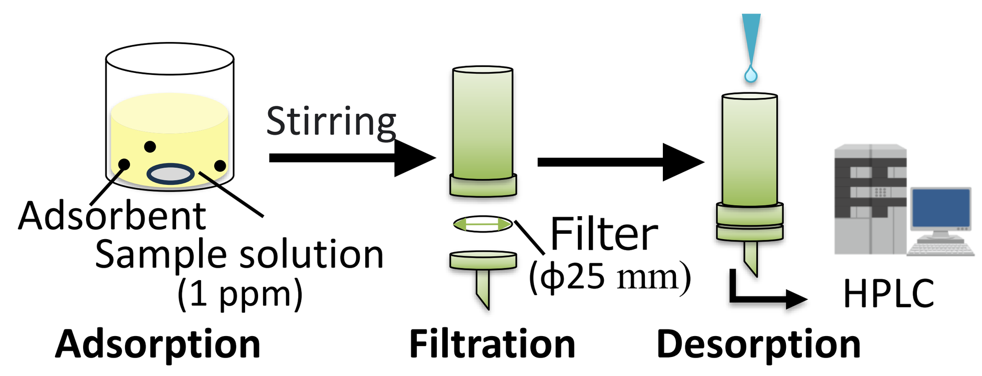


Fig. 2. Experimental procedure.

Table 1. Preconcentration conditions.

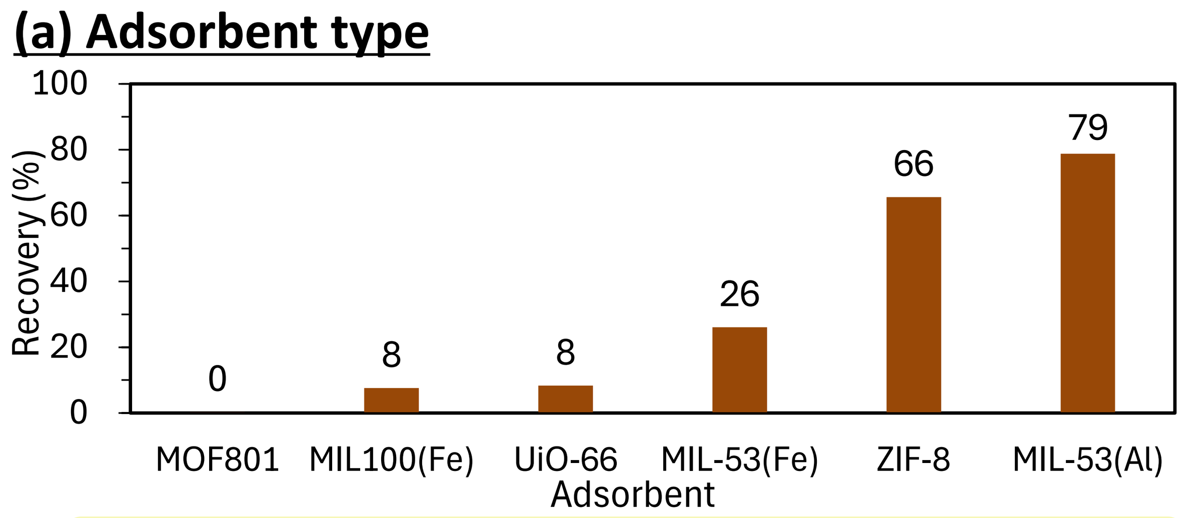
Sample	Bensulfuron-methyl (1 ppm, 100 mL)
Adsorbent	MOF preparation (5 mg)
Stirring time	20 min
Desorption	Acetonitrile with 0.1% formic acid (2 mL)

Table 2. HPLC conditions.

Column	TOSOH（TSK-GEL）ODS-100V 5 μm
Eluent	Acetonitrile : water = 50 : 50
Flow rate	1.0 mL/min
Detection	240 nm

Results

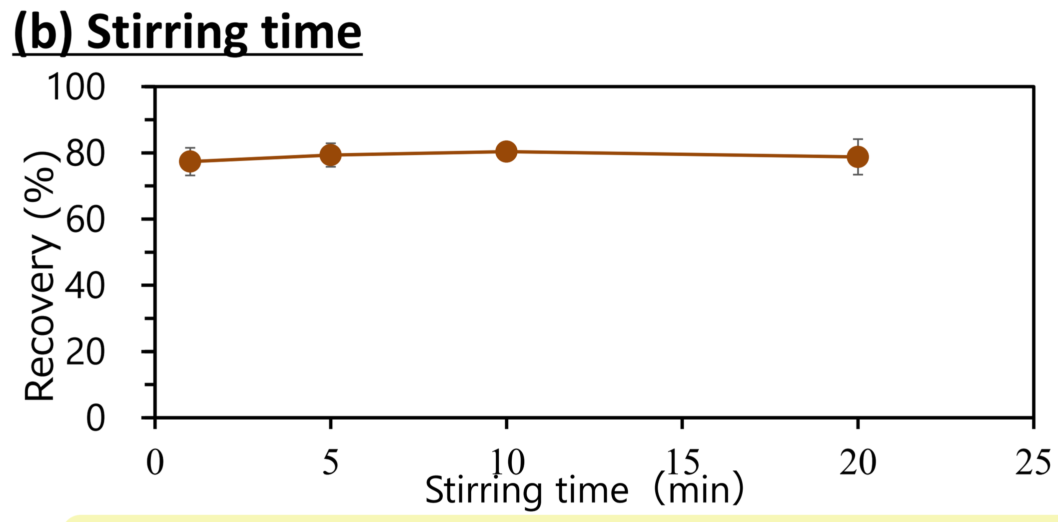
(a) Adsorbent type



- **MIL-53(Al)** achieved the highest recovery (~80%).
- The other MOFs showed lower recoveries (0–70%).

→ **MIL-53(Al)**

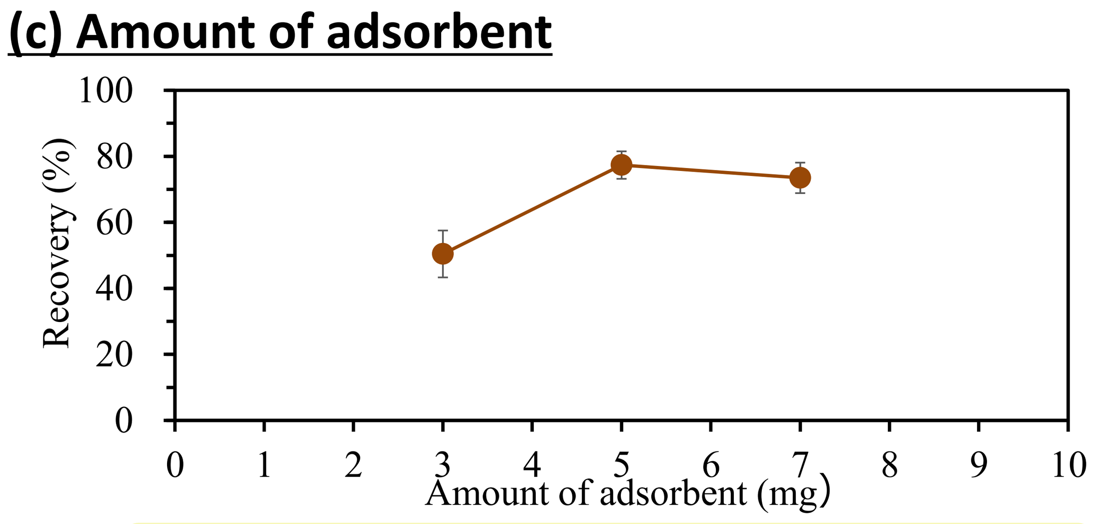
(b) Stirring time



- The extraction of bensulfuron-methyl was very fast.
- 70% adsorbed in **1 min** → Equilibrium reached.

→ Optimum 1 min

(c) Amount of adsorbent



Recovery increased rapidly and reached a maximum at **5 mg.** → Equilibrium reached.

→ Optimum 5 mg

Fig. 3. Effect of (a) adsorbent type, (b) Stirring time, (c) amount of adsorbent on recovery of bensulfuron-methyl.

Characterization

SEM

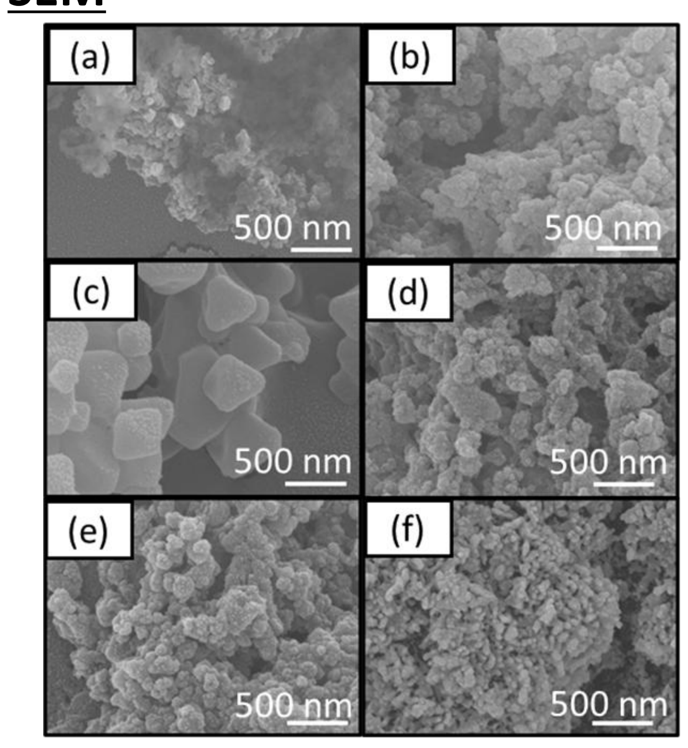


Fig. 4. SEM images of (a) ZIF-8, (b) MIL-100(Fe), (c) MOF-801, (d) UiO-66, (e) MIL-53(Fe), and (f)MIL-53(Al).

XRD

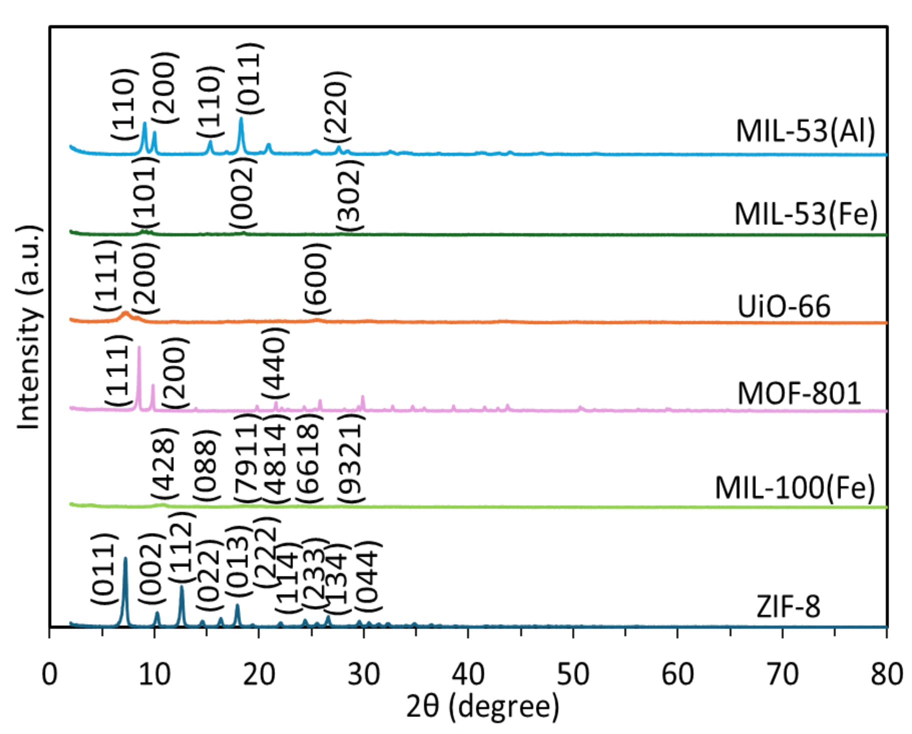


Fig. 5. The XRD patterns of ZIF-8, MOF-801, MIL-100(Fe), UiO-66, MIL-53(Fe), and MIL-53(Al).

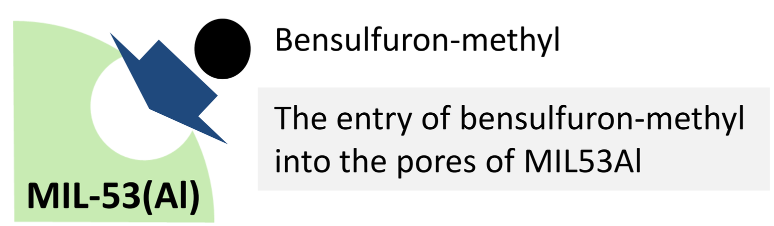
BET

Table 3. BET surface area and pore size parameters of ZIF-8, MOF-801, MIL-100(Fe), UiO-66, MIL-53(Fe), and MIL-53(Al).

	Surface area ($m^2 g^{-1}$)	Total pore volume ($cm^3 g^{-1}$)	Average pore diameter (nm)
ZIF-8	1350	0.82	2.43
MOF-801	620	0.32	2.06
MIL-100(Fe)	850	0.85	4.04
UiO-66	464	0.23	2.02
MIL-53 (Fe)	120	0.06	2.10
MIL-53 (Al)	772	1.51	7.85

Mechanism

Physisorption



Chemisorption

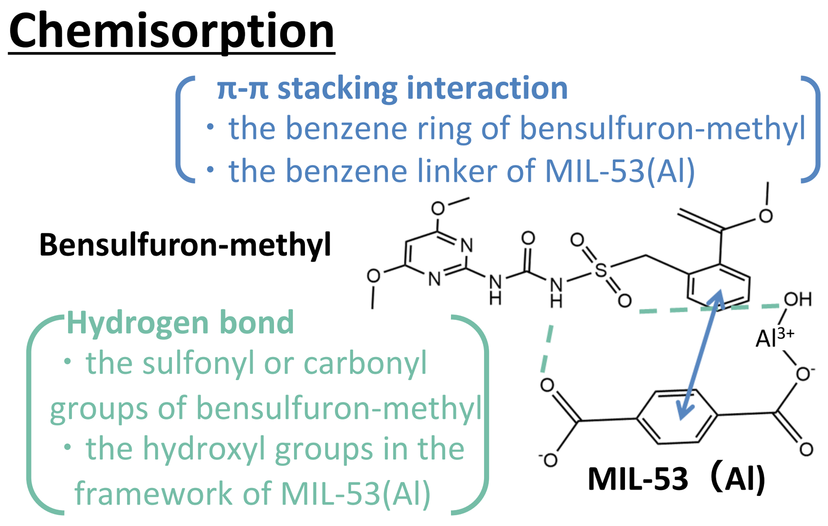


Fig. 6. Adsorption mechanisms.

Conclusion

- ZIF-8, MOF-801, MIL-100(Fe), UiO-66, MIL-53(Fe) and MIL-53(Al) were synthesized, and evaluated as SPE sorbents for the pretreatment of bensulfuron-methyl.
- **MIL-53(Al)** showed the highest recovery (80%), while the other MOFs performed poorly, suggesting adsorption performance depends on the metal–ligand combination.
- Future work will optimize pretreatment to improve recovery and assess applicability to real samples.