MICROSOF

Recherche de microplastiques dans 33 sols français

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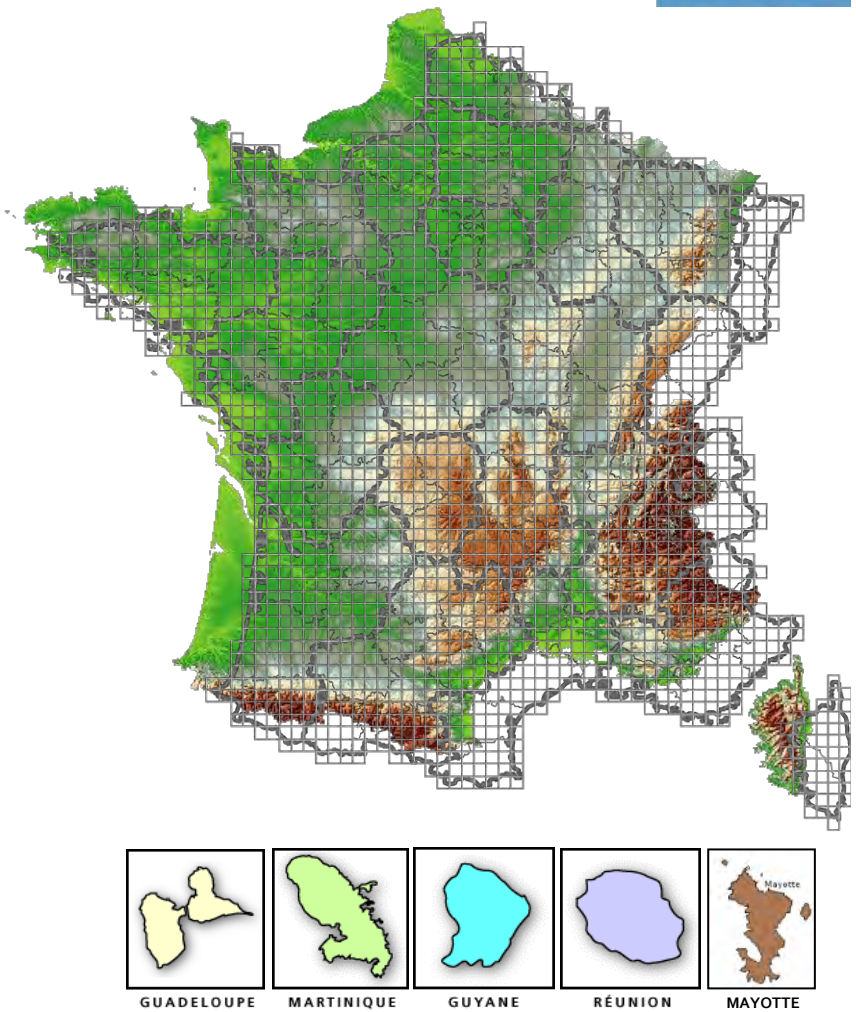
Contexte

Réseau de Mesure de la Qualité des Sols (RMQS) :

- 2240 sites
- Répartis selon un maillage de 16 km x 16 km
- Représentatifs des sols français et de leurs usages
- Rééchantillonnés tous les quinze ans

→ **Suivre l'évolution de la qualité des sols français**



Projet MICROSOF (2020-2022) :

33 sols différents issus d'une collecte organisée par l'INRAe d'Orléans

Froger, C. (2020, septembre 11). *Présentation du réseau de mesures de la qualité des sols (RMQS) et du projet Phytosol.* [PowerPoint presentation]

Le projet MICROSOF

Etablir les premières références nationales sur la contamination des sols français par les microplastiques

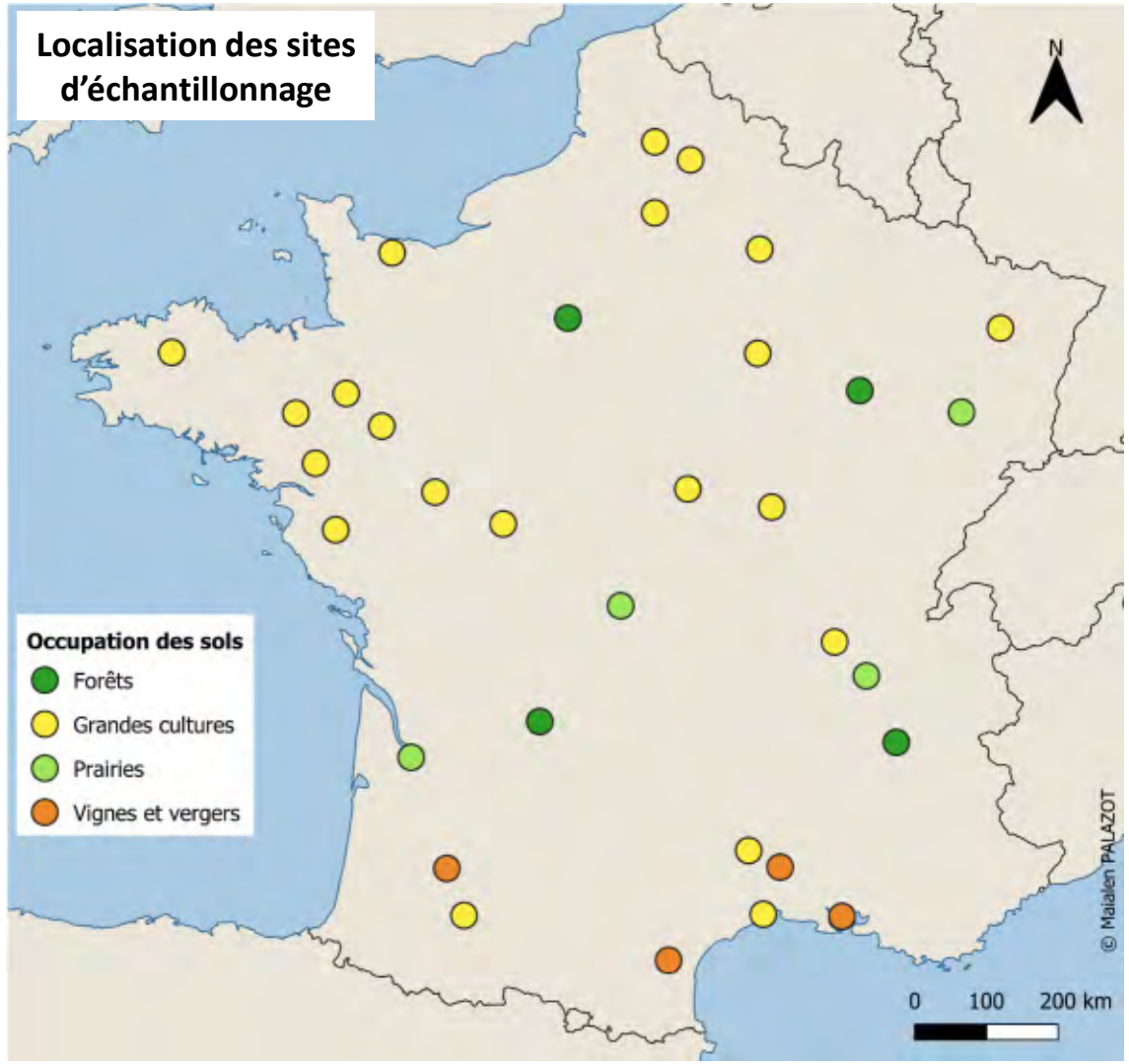
33 échantillons

- 21 grandes cultures
- 4 prairies
- 4 forêts
- 4 vignes & vergers

Objectifs

Développer un **protocole d'extraction et** de caractérisation des microplastiques piégés dans différents échantillons de sol

Quantifier (nombre) et caractériser (nature chimique, taille) les MP extraits



Matériels et méthodes

Falcons 50 mL

Densité: 1,8 g.cm^{-3}

Extraire tous les MP, même ceux de grande densité tels que le PVC et le PET

Recyclage du NaI jusqu'à 10 cycles [2]

Extraction

1. **Pré-traitement**
   Séchage 40°C
   Tamisage 5 & 2mm
   (~1kg sol)
2. **Sous-échantillonnage**
   Triplicats de 50g (poids sec)
3. **Tamisage humide**
   Eliminer les fractions les plus fines (<50µm)
4. **Elimination de la MOS***
   Réaction de Fenton
   T°C < 40°C
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5. **Séparation par densité**
   Solution saturée de **NaI**
6. **Analyse**
   Photos + logiciel de traitement d'image, FTIR

Solution de catalyseur $FeSO_4$ 7 H_2O + H_2O_2 (30%)

Ratio 1:1

Réaction exothermique
T° < 40°C, maintenue grâce à un **bain de glace**

Selon **Hurley et al. (2018)** [1]

Filtration du surnageant
Filtres 300µm et 50µm

[315 µm ; 5mm[

Blancs de labo, Spiking test

[1] Hurley, R. R., Lusher, A. L., Olsen, M., & Nizzetto, L. (2018). Validation of a method for extracting microplastics from complex, organic-rich, environmental matrices. *Environmental science & technology*, *52*(13), 7409-7417.

[2] Kedzierski, M., Le Tilly, V., César, G., Sire, O., & Bruzaud, S. (2017). Efficient microplastics extraction from sand. A cost effective methodology based on sodium iodide recycling. *Marine pollution bulletin*, *115*(1-2), 120-129.

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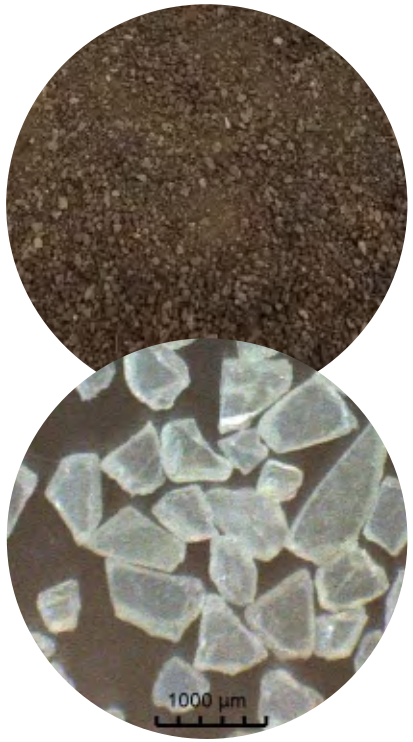
Les premiers résultats : efficacité de l'extraction



Echantillons de terre « test »

- Triplicats contrôle
- Triplicats avec ajout de MP

MP (non vieillis) obtenus par cryobroyage

- PE
- PP
- PS
- PVC
- PA

[500 ; 1000 µm[

10 MP de chaque type par réplicat

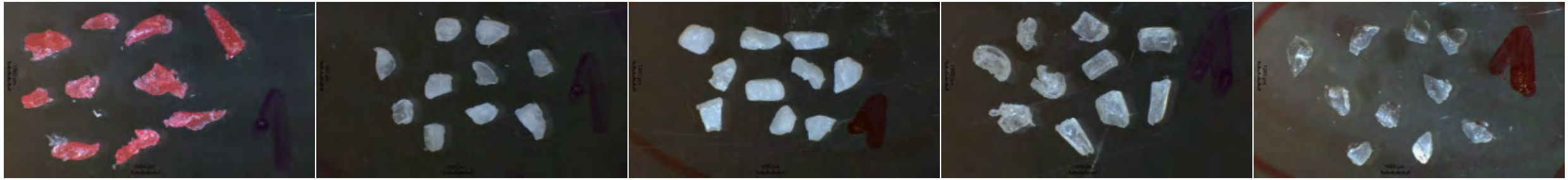
Polymère	Taux d'extraction
PE	**97%**
PP	**93%**
PS	**100%**
PA	**93%**
PVC	**97%**

Les premiers résultats : intégrité des MP

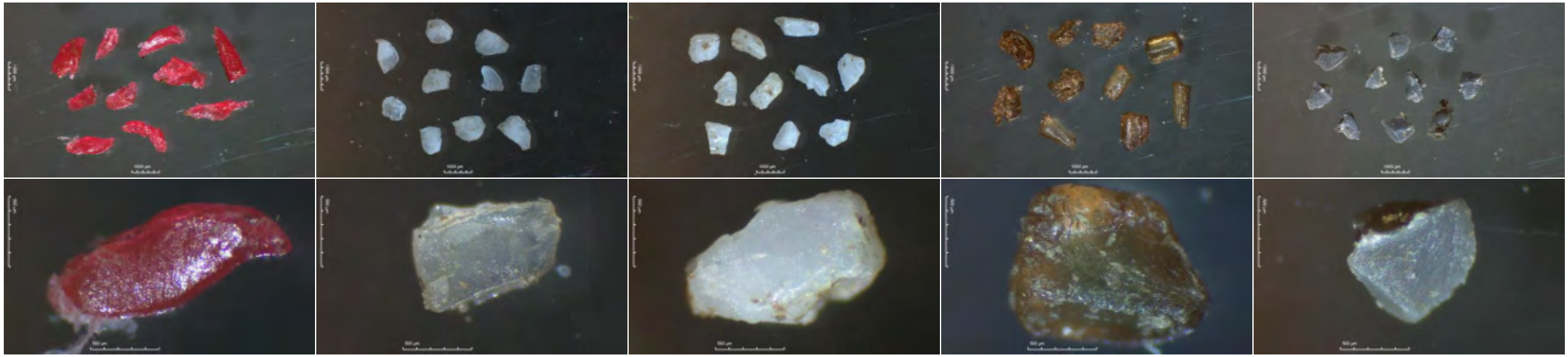
- Microscopie
- Spectroscopie FTIR

Polyéthylène	Polypropylène	Polystyrène	Polyamide	PVC

Avant



Après



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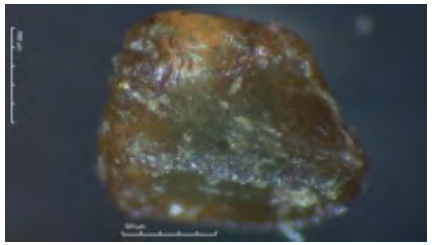
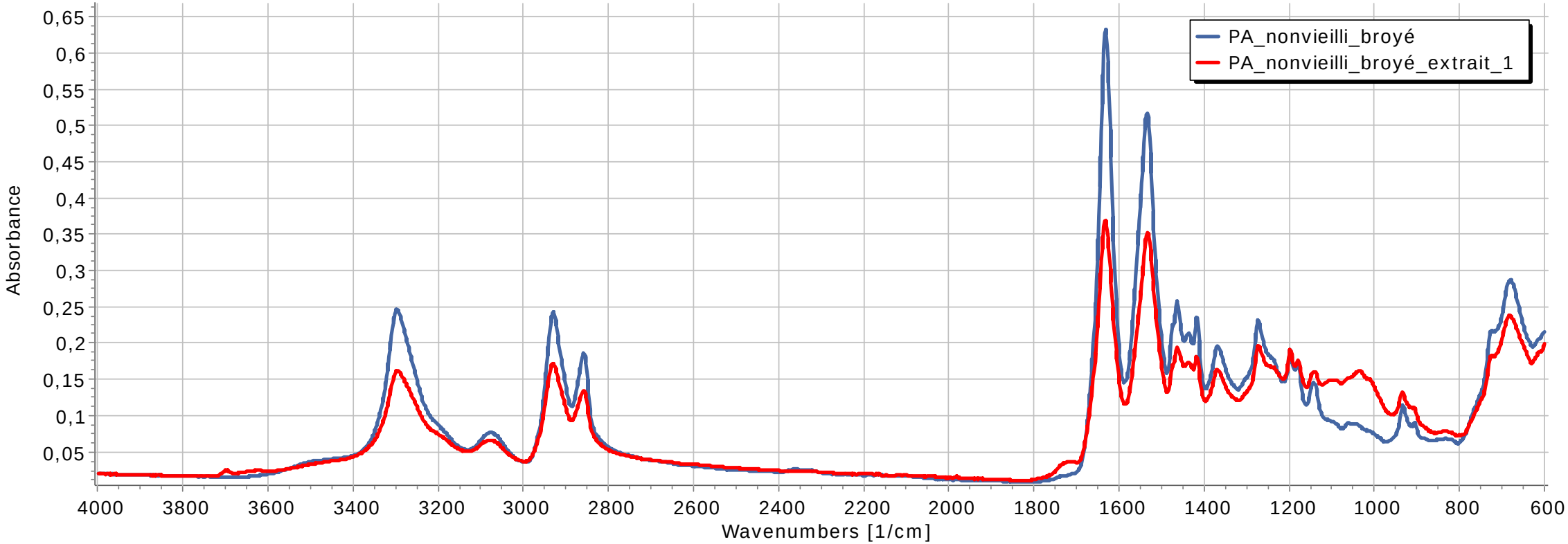


Polyamide



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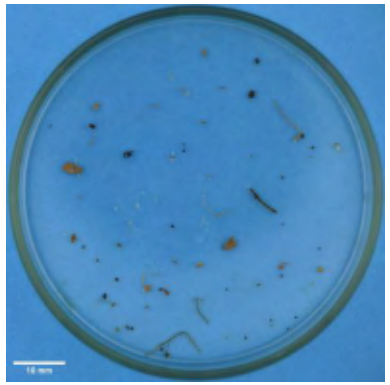
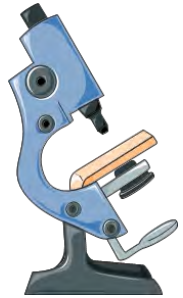
Matériels et méthodes

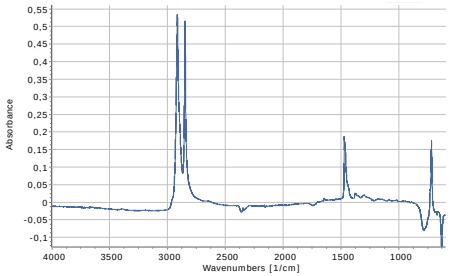
Caractérisation



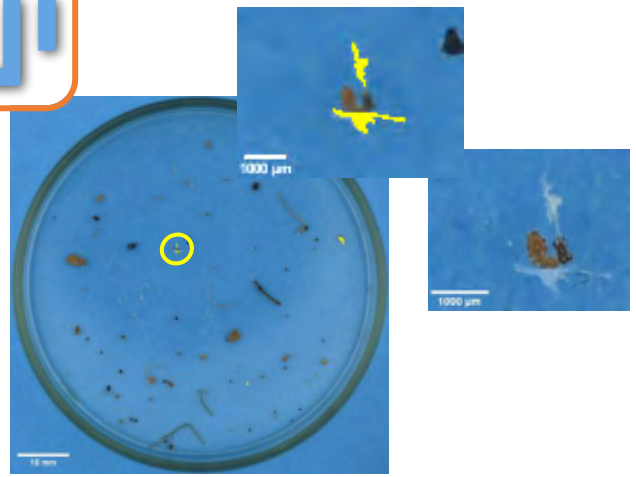
Photographie des boîtes de Pétri



Détection visuelle des MP



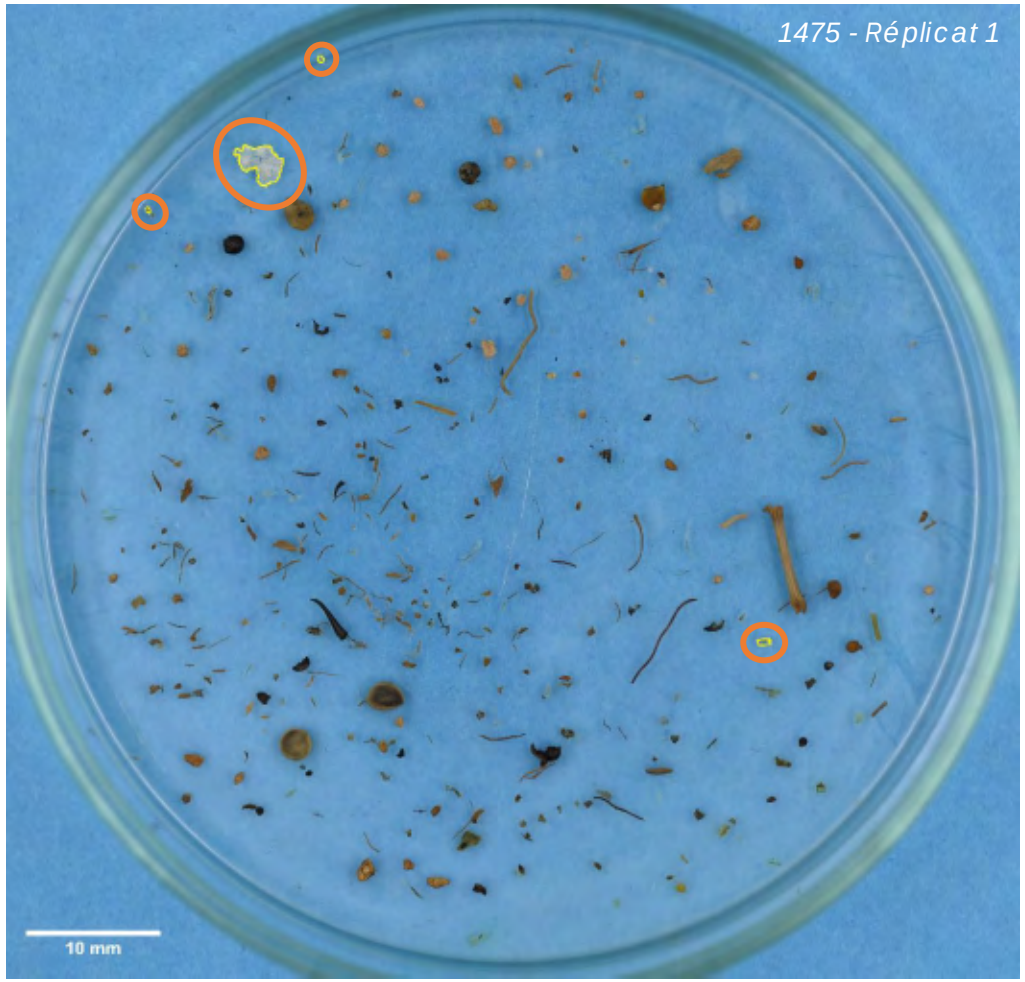
Identification des MP à l'ATR-FTIR



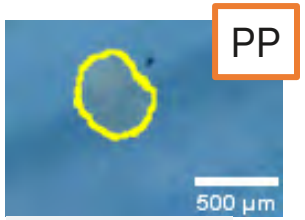
Analyse d'image

Diamètre de Feret

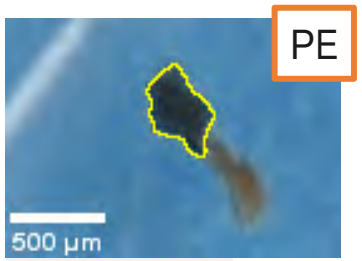
Quelques exemples…



Feret : 3 847 µm



Feret : 526 µm



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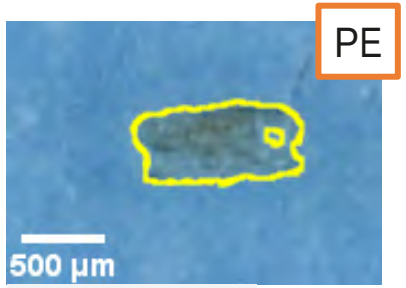


Feret : 1 043 µm

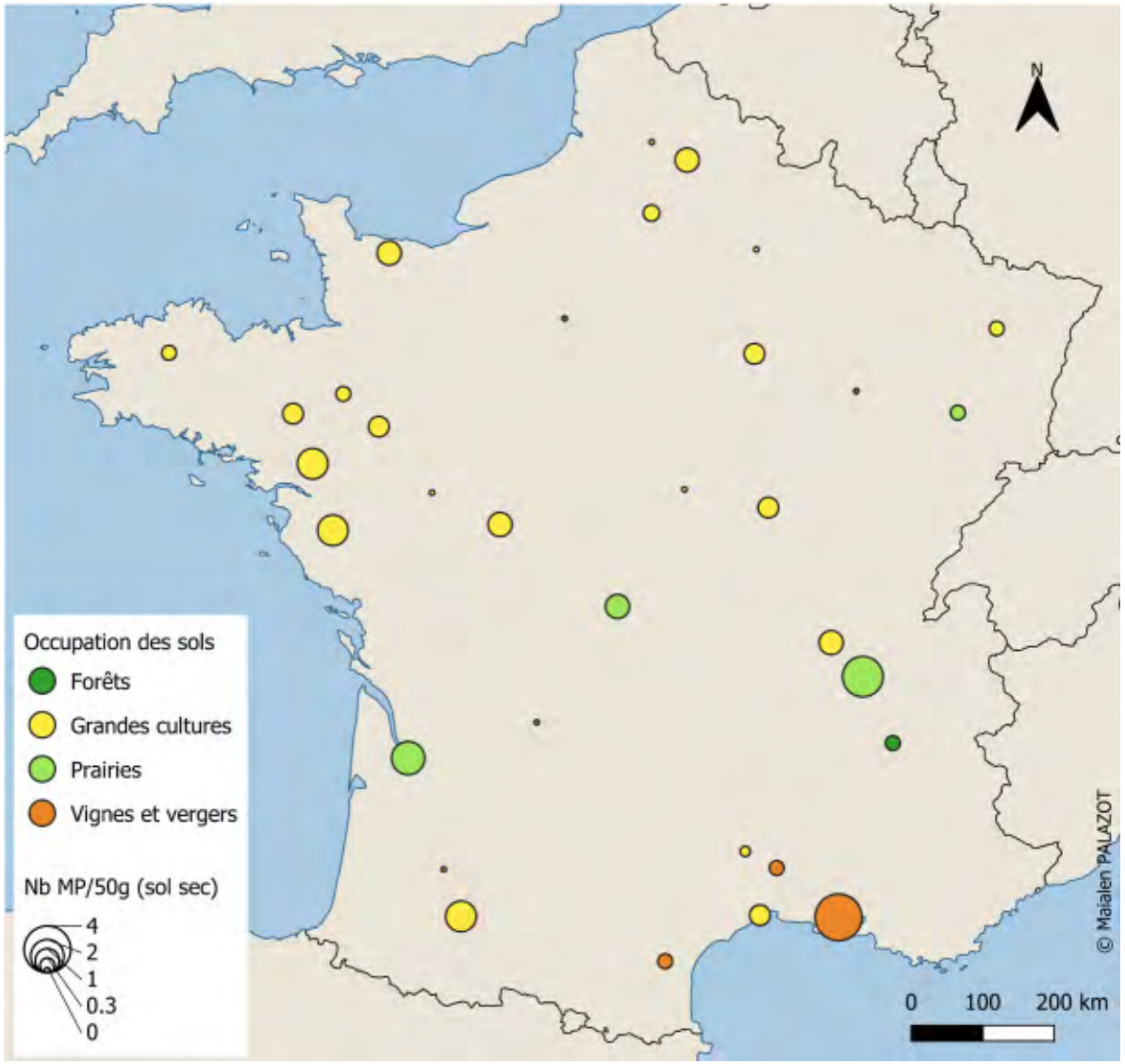
Résultats

Y a-t-il des MP dans les sols Français ?

MP retrouvés dans 76% des échantillons (25/33)

64% d'entre eux (16/25) :
MP dans au – 2 réplicats

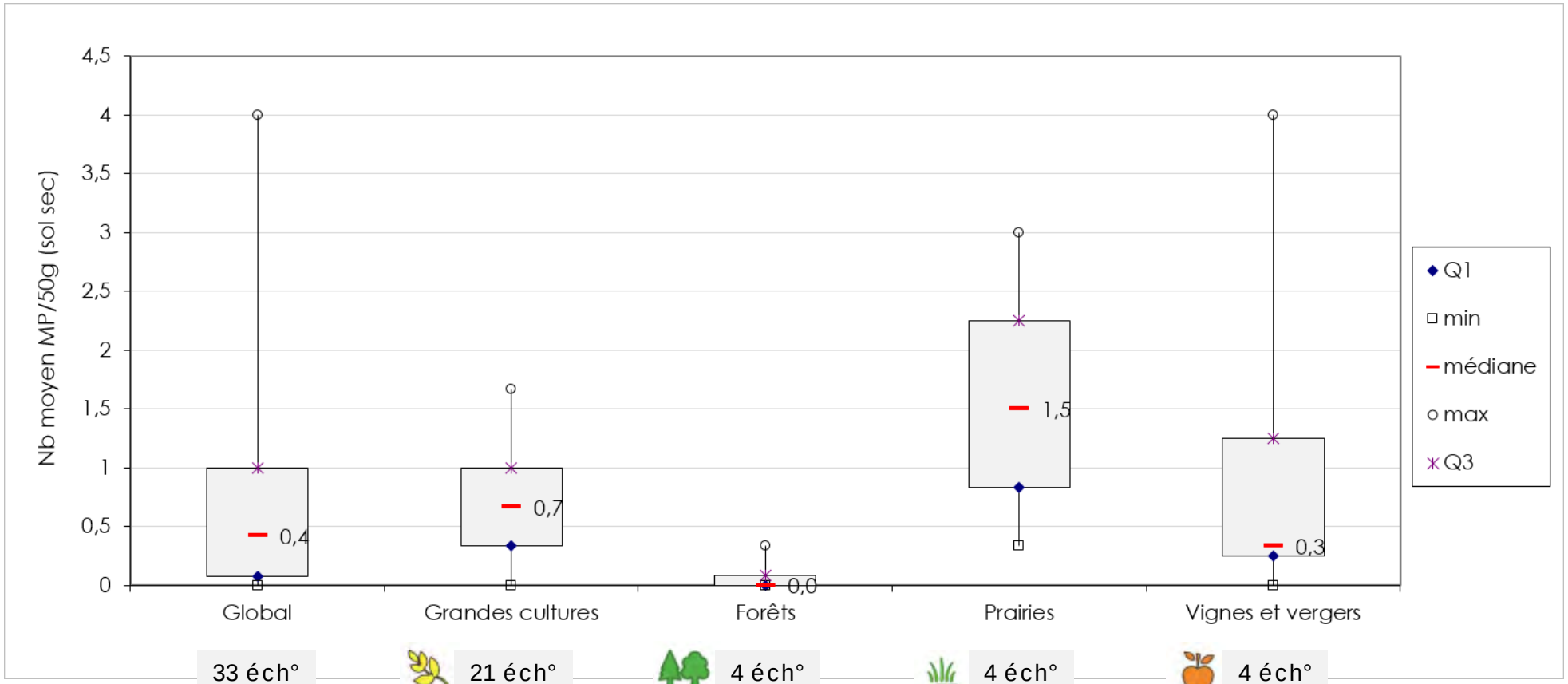
Forêts	1/4
Grandes cultures	17/21
Prairies	4/4
Vignes et vergers	3/4



Résultats

Quantification (nb MP/50g sol sec)

[315 µm ; 5mm[

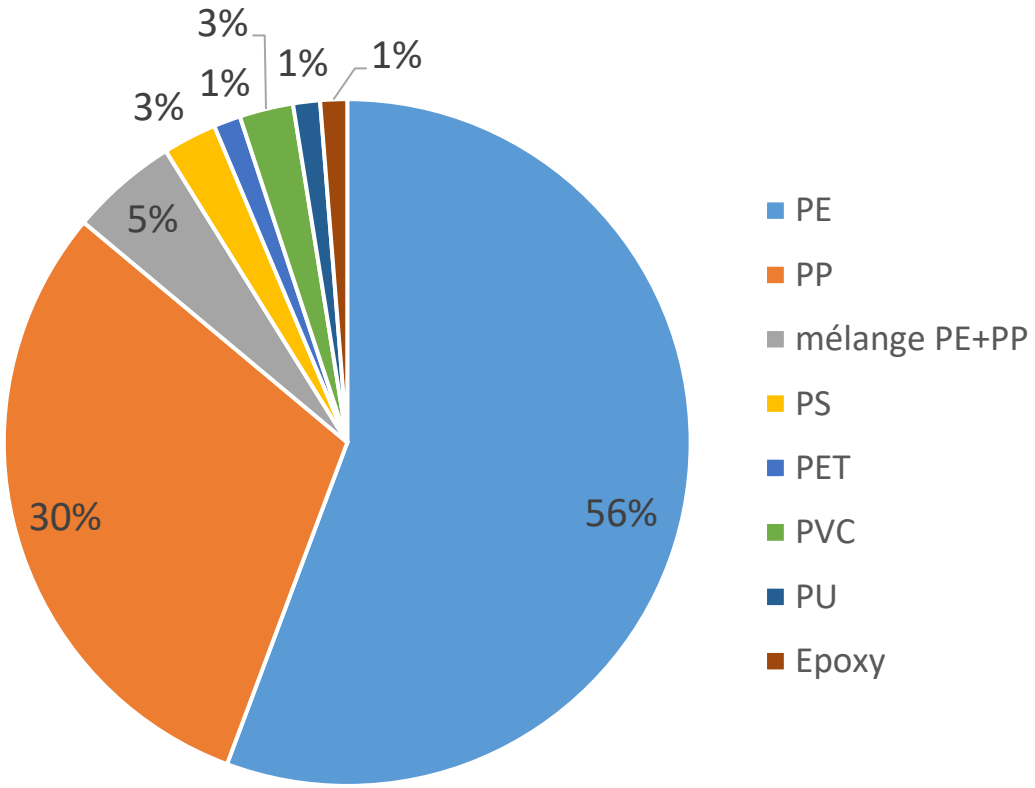


33 éch°	21 éch°	4 éch°	4 éch°	4 éch°
n_{MP} = 79	n_{MP} = 45	n_{MP} = 1	n_{MP} = 19	n_{MP} = 14

Résultats

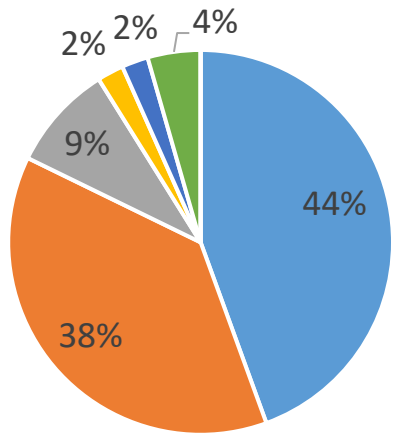
Nature chimique des MP

[315 µm ; 5mm[



Global

n_{MP} = 79



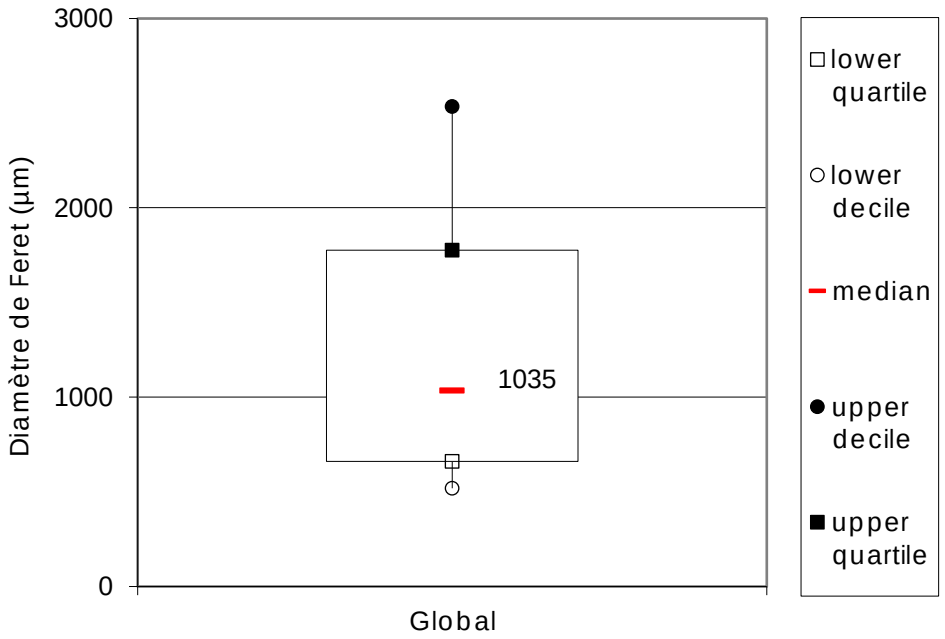
Grandes cultures

n_{MP} = 45

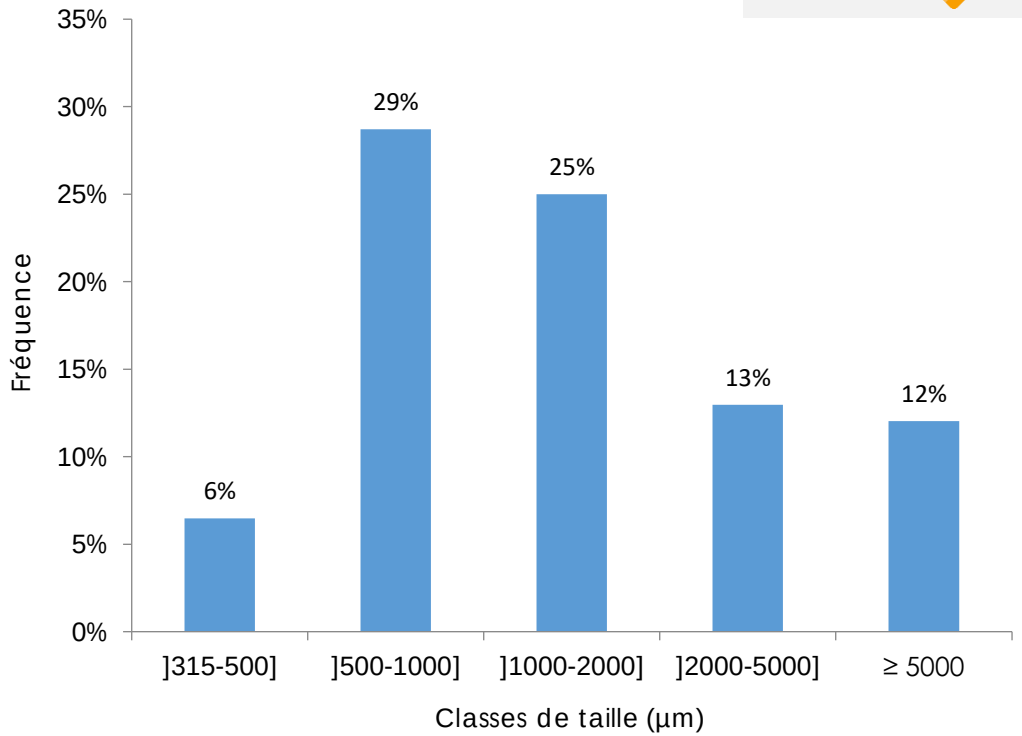
Résultats

Tailles des MP (diamètre de Feret, µm)

[315 µm ; 5mm[



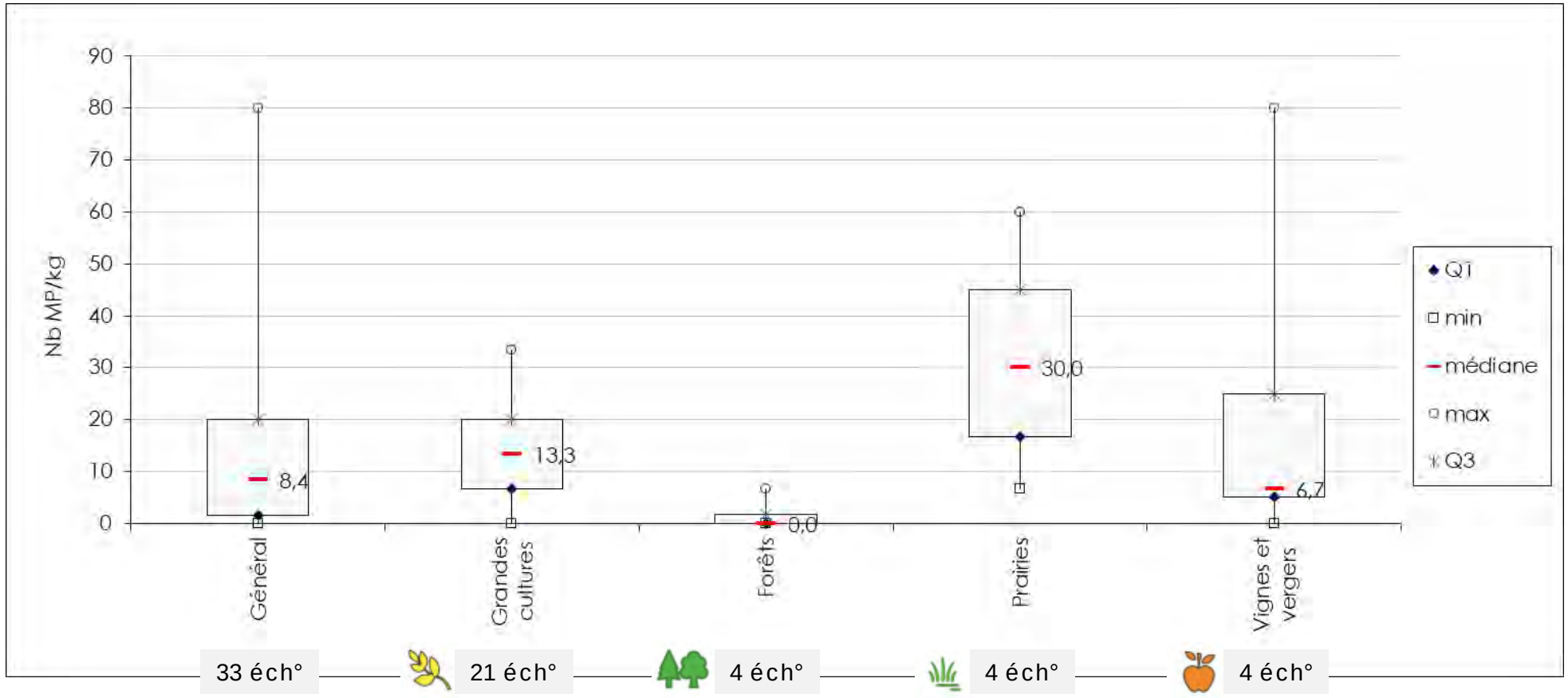
≤ 315 µm



Résultats

Quantification (nb MP/kg sol sec)

[315 µm ; 5mm[



Results

Quantification (nb MP/kg sol sec)

Global concentrations of microplastics in soils – a review

Frederick Büks and Martin Kaupenjohann
Chair of Soil Science, Department of Ecology, Technische Universität Berlin, 10587 Berlin, Germany
Correspondence: Frederick Büks (frederick.bueks@tu-berlin.de)

Received: 10 August 2020 – Discussion started: 1 September 2020
Revised: 9 November 2020 – Accepted: 11 November 2020 – Published: 17 December 2020

region	sites	land use	entry pathway	measured plastic type (***bold*** =found)	items kg^{-1} dw mv ± sd median (min–max)	size span [µm]	reference
Austria/Southern Germany	11	NA	NA	NA	$11x10^6$ ($2x10^6$–$26x10^6$)	5–1,000	Meixner et al. (2020)
Mittelfranken (Germany)	1	agr	none	**PE, PP, PS**	0.3 (0.0–1.3)	1,000–5,000	Piehl et al. (2018)
Denmark	1	agr	sew	**PP > PE >> nylon**	71,000 (0–165,000)	20–500	Vollertsen (2017)
	1	agr	no sew	**PE >> nylon > PP**	145,000 (53,000–528,000)		
València (Spain)	1	agr	sew (1x)	NA	1,499 (999–1,998)	>11	van den Berg et al. (2020)
	1	agr	sew (3x)		2,664 (999–3,996)		
	1	agr	sew (3x)		1,998 (999–3,663)		
	1	agr	sew (3x)		2,830 (1,998–3,330)		
	1	agr	sew (4x)		5,328 (1,332–6,327)		
	1	agr	sew (4x)		3,330 (1,998–3,996)		
	1	agr	sew (5x)		7,659 (7,326–7,992)		
	1	agr	sew (5x)		3,330 (1,998–5,328)		
	1	agr	sew (6x)		2,997 (2,331–5,994)		
	1	agr	sew (8x)		3,996 (1,998–8,658)		
	1	agr	sew (8x)		2,831 (1,665–5,994)		
	1	agr	no sew		2,498 (333–4,662)		
	1	agr	no sew		999 (333–2,331)		
	1	agr	no sew		500 (0–1,332)		
	1	orch	no sew		999 (0–1,332)		
	1	orch	no sew		2,664 (999–2,664)		
Southeast Ontario (Canada)	1	agr	no sew	**PS, PE, PP, PU, polyester, others**	4 ± NA	NA	Crossman et al. (2020)
	2	agr	sew (1x)		103 ± 52		
	1	agr	sew (2x)		541 ± 305		
Wuhàn, 武汉市 (China)	10	hort	pm, sew, ww	**PE > PA, PP, PS**	43,000–620,000	10–5,000 82%<100	Zhou et al. (2019)
	7	for	pm, sew, ww		96,000–690,000		
	7	fal	pm, sew, ww		22,000–200,000		

[315 µm ; 5mm[

Médiane (min-max) = 8,4 (0,0 – 80) MP/kg (dw)

Moyenne (± écart-type) = 15 (± 23) MP/kg (dw)

Global

Médiane (min-max) = 13 (0,0 – 33) MP/kg (dw)

Moyenne (± écart-type) = 13 (± 17) MP/kg (dw)

Grandes cultures

Table 1. Studies on microplastic concentrations with characterization of sites, applied methods and extracted microplastic samples. The abbreviations used in this table are as follows: hort – horticulture; agr – agriculture; orch – orchard; for – forest; sew – sewage sludge application; pm – plastic mulching; ww – waste water; dw – dry weight. . NA denotes that information was not available

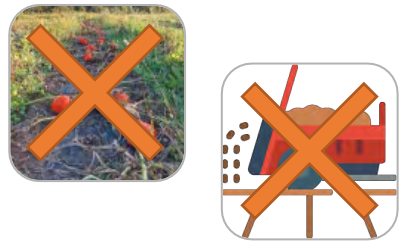
Adapted from Büks and Kaupenjohann (2020)

Conclusion & perspectives

MICROSOF : Etablir les premières références nationales sur la contamination des sols français par les microplastiques

33 échantillons

4 occupations



MP retrouvés dans 76% des échantillons (25/33)

Quantités < sols avec application de PRO et/ou paillage plastique

Quantités dépendent des tailles étudiées

PE & PP en majorité

MICROSOF (2020-2022)

BIOMALEG (2021-2023)

PRO (2021-2023)

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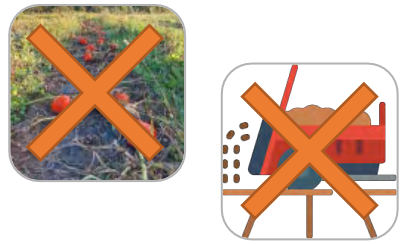

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IRDI Institut de Recherche Dupuy de Lôme UMR CNRS 6027

ADEME AGENCE DE LA TRANSITION ÉCOLOGIQUE

MICROSOF (2020-2022)

BIOMALEG (2021-2023)

PRO (2021-2023)

Merci

mikael.kedzierski@univ-ubs.fr

Büks, F., & Kaupenjohann, M. (2020). Global concentrations of microplastics in soils–a review. *Soil*, *6*(2), 649-662.

Region	Sites	Land use	Entry pathway	Vicinity	Sampling depth (cm)	Method of extraction	Method of quantification/ qualification	Measured plastic type (bold=found)	mg kg^{-1} dw mv ± sd median (min–max)	Items kg^{-1} dw mv ± sd median (min–max)	Size span (µm)	Shape	Reference
Wùhàn, 武汉市 (China)	8 12	hort hort	NA road	Municipal	NA	S-DF ($ZnCl_2$ 1.5 g cm^{-3})	LM, Raman	**PA,PP > PS,PVC > PE**	NA	1083 (600–3167) 1142 (300–12500)	20–5000 70% <200	fib,mb>frag	Chen et al. (2020)
Región Metropolitana de Santiago (Chile)	1 1 1 1 1	agr agr agr agr agr	Sew (1x) Sew (2x) Sew (3x) Sew (4x) Sew (5x)	Municipal	0-25	S-DF (water, NaCl 1.2 g cm^{-3}, $ZnCl_2$ 1.55 g cm^{-3})	LM, shape to mass	NA	1.4 (0.7–2.2) 2.0 (1.8–3.2) 2.2 (0.6–4.6) 2.9 (1.8–12.9) 4.4 (1.8–10.3)	1200 (0–2200) 1800 (1200–3200) 1200 (200–4400) 2200 (800–12400) 3600 (1000–10200)	<5000 Mainly <1000x50	fib>>others	Corradini et al. (2019)
Southeast Ontario (Canada)	1 2 1	agr agr agr	No sew Sew (1x) Sew (2x)	Rural	0-5, 5-10, 10-15	Fenton's reagent, DF	LM, FTIR	**PS, PE, PP, PU, polyester, others**	NA	4 ± NA 103 ± 52 541 ± 305	NA	fib>frag	Crossman et al. (2020)
Köln (Germany)	1	NA	Road	Municipal	NA	PFE	Pyr-GC-MS	**PE > PP, PS**	915 ± 63	NA	NA	NA	Dierkes et al. (2019)
Shanxi Province, 陕西省 (China)	9	agr	pm, sew	Municipal	NA	S-DF (NaCl 1.2 g cm^{-3}, $CaCl_2$ 1.5 g cm^{-3}), H_2O_2	LM, FTIR	**PE, PET, PP, PS, PVC**	NA	2131 ± 371	<5000 Mainly <500	fib>frag>other	Ding et al. (2020)
Sydney (Australia)	1	NA	NA	Industrial	NA	PFE	Gravimetric, FTIR	**PE, PS, PVC**, PP, PET	2400 (300–67500)	NA	<1000	NA	Fuller and Gautam (2016)
Tiānjīn, 天津市 (China)	1	NA	NA	Municipal	NA	AM-DF (NaCl+NaI 1.5 g cm^{-3}), H_2O_2	LM, FTIR	**PP**	NA	95	<3200	frag	Han et al. (2019)
Shíhézǐ, 石河子市 (China)	1 1 1	agr agr agr	pm (5 yr) pm (15 yr) pm (24 yr)	Municipal	0-40	S-DF (NaI 1.85 g cm^{-3}), H_2O_2	LM, FTIR	**PE**	NA	80 ± 49 308 ± 138 1076 ± 347	<5000	frag	Huang et al. (2020)
Yucatan Peninsula (Mexico)	10	hort	lit	Rural	0-10, 10-20	U-DF (water)	LM	NA	NA	870 ± 1900	<2000 95% <50	NA	Huerta Lwanga et al. (2017)
Shànghǎi, 上海市 (China)	20	hort	pg, pm	Municipal	0-3, 3-6	U-DF (NaCl 1.2 g cm^{-3}), H_2O_2	LM, FTIR	**PP, PE > polyester**	NA	70 ± 13	20–10000 54% <1000	fib>frag> films>pel	Liu et al. (2018)
Malmö (Sweden)	1 1 1	agr agr agr	No sew Sew (1 t ha^{-1}a^{-1}) Sew (3 t ha^{-1}a^{-1})	Municipal	0-20	H_2O_2+enzymes, DF ($ZnCl_2$ 1.7 g cm^{-3})	LM, FTIR, shape to mass	Diverse	0.3 0.3 3.4	NA	10–5000	NA	Ljung et al. (2018)
Shànghǎi, 上海市 (China)	3 3	rice rice-fish	Ponding	Municipal	0-10	S-DF (NaCl 1.24 g cm^{-3}), H_2O_2	LM, FTIR	NA	NA	12 ± 4 4 ± 2	>20	fib, frag	Lv et al. (2019)
Austria/Southern Germany	11	NA	NA	Municipal	NA	H_2O_2, U-DF ($ZnCl_2$ 1.45 g cm^{-3})	LM	NA	NA	11x10^6 (2x10^5–26x10^6)	5–1000	NA	Meixner et al. (2020)
Mittelfranken (Germany)	1	agr	None	Rural	0-5	H_2O_2, sieving	LM, FTIR	**PE, PP, PS**	NA	0.3 (0.0–1.3)	1000–5000	films>frag> others	Piehl et al. (2018)
Fars province, فارس (Iran)	5 5	agr gra	pm NA	Rural	0-10	S+U-DF (water)	LM, shape to mass	NA	1.2 ± 0.6 0.2 ± 0.1	205 ± 186 38 ± 17	40–740 Mainly <100	NA	Rezaei et al. (2019)
València (Spain)	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	agr agr agr agr agr agr agr agr agr agr agr agr agr agr agr agr orch orch	Sew (1x) Sew (3x) Sew (3x) Sew (3x) Sew (4x) Sew (4x) Sew (5x) Sew (5x) Sew (6x) Sew (8x) Sew (8x) No sew No sew No sew No sew No sew No sew	Rural Rural NA Rural Rural NA Rural NA Rural Rural NA Municipal NA Rural Rural NA	0-10, 10-30	S-DF (water, NaI 1.7 g cm^{-3})	LM, FTIR	NA	NA	1499 (999–1998) 2664 (999–3996) 1998 (999–3663) 2830 (1998–3330) 5328 (1332–6327) 3330 (1998–3996) 7659 (7326–7992) 3330 (1998–5328) 2997 (2331–5994) 3996 (1998–8658) 2831 (1665–5994) 2498 (333–4662) 999 (333–2331) 500 (0–1332) 999 (0–1332) 2664 (999–2664)	>11	frag>>fib,films	van den Berg et al. (2020)
Denmark	1 1	agr agr	Sew No sew	NA	0-15	SDS, S-DF (water, $ZnCl_2$ 1.7 g cm^{-3})	LM, FTIR, shape to mass	**PP > PE >> nylon** **PE >> nylon > PP**	5.8 (0.0–16.5) 12.0 (0.1–224)	71000 (0–165000) 145000 (53000–528000)	20–500	NA	Vollertsen and Hansen (2017)
Kūnmíng, 昆明市 (China)	2 (G) 2 (N) 1 (G)	agr agr for	pg, sew, ww pg, sew, ww NA	Municipal	0-5, 5-10	NaOH+H_2O_2, U-DF (water, NaI 1.8 g cm^{-3})	LM	NA	NA	26070 (13470–42960) 12050 (7100–26630) 14440 (8180–18100)	50–10000, 95% <1000, 82% <250	fib>>others	Zhang and Liu (2018)
Loess Plateau, 黄土高原 (China)	1 1 1	agr orch hort	pm NA NA	Rural	0-10, 10-30	S+U-DF (water)	LM, shape to mass	**LD-PE, PP**	0.3 ± 0.5 0.5 ± 0.7 0.1 ± 0.1	80 ± 136 187 ± 222 87 ± 213	Mainly >100	NA	Zhang et al. (2018)
Hāěrbīn, 哈尔滨市 (China)	2 2	agr agr	pm pm	Municipal Rural	0-20 (0-30)	DF (water)	LM, FTIR, shape to mass	NA	0.1 ± 0.6 0.0 ± 0.0	163 ± 250 75 ± 130	50–5000 Mainly >100	NA	Zhang et al. (2020)
Wuhàn, 武汉市 (China)	10 7 7	hort for fal	pm, sew, ww pm, sew, ww pm, sew, ww	Mainly industrial	at 5	KOH+NaClO, S+U-DF (NaCl 1.19 g cm^{-3}, $ZnCl_2$ 1.55 g cm^{-3})	LM, Raman	**PE > PA, PP, PS**	NA	43000–620000 96000–690000 22000–200000	10–5000 82%<100	frag>>others	Zhou et al. (2019)
Hangzhou Bay, 杭州湾 (China)	60	agr	pm	Municipal	0-10	DF (NaCl 1.2 g cm^{-3}, NaI 1.6 g cm^{-3}), H_2O_2	LM, FTIR	Diverse	NA	310 (0–2760)	>60 Mainly 500–3000	frag, fib>films	Zhou et al. (2020)
Ithaca (USA) Cobleskill (USA)	1 3	orch NA	Sew Sew	Municipal	0-10, 10-25, 25-50	S-DF (water)	LM	NA	NA	1250 ± 60 1240 ± 87	NA	fib	Zubris and Richards (2005)

Table 1. Studies on microplastic concentrations with characterization of sites, applied methods and extracted microplastic samples. The abbreviations used in this table are as follows: hort – horticulture; agr – agriculture; gra – grassland; orch – orchard; for – forest; fal fallow; sew – sewage sludge application; pm – plastic mulching; pg – plastic greenhouses; lit – littering; ww – waste water; S – stirring; U – ultrasonication; AM – air mixing; DF – density fractionation; SDS – sodium dodecyl sulfate; LM – light microscopy; FTIR – Fourier transform infrared spectroscopy; Pyr–GC–MS – pyrolysis–gas chromatography–mass spectrometry; Raman – Raman spectroscopy; fib fibers; mb – microbeads; frag – fragments; pel – pellets; G – Gleysol; N – Nitisol; dw – dry weight. Information indicated in bold means that the specific type of microplastic was found. NA denotes that information was not available. Detailed data are listed in the Supplement.