Supplementary Material: Developing a systematic method for extraction of microplastics in soils

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Supplementary Materials

Table S 1. Particle size distribution (proportion of clay, silt and sand) for inorganic soils used in the experiment measured using the hydrometer method (Sheldrick and Wang, 1993).

	Clay (%)	**Silt (%)**	**Sand (%)**
Soil 1	0.0	14.4	85.6
Soil 2	14.4	24.0	61.6
Soil 3	21.2	29.2	49.6
Soil 4	36.2	26.5	37.3
Soil 5	42.6	37.3	20.2
Soil 6	52.7	46.5	0.8

Table S 2. Statistical analysis results for Kruskal Wallis tests to measure differences in: the amount of organic removed from samples of initial high and low organic content by different chemicals and by those chemicals at different temperatures; the recovery efficiency of microplastics from inorganic soils using density separation methods; the recovery efficiency of microplastics from organic soils using a combination of digestion and density separation methods; and the ease of identification of microplastics subjected to different treatments, split into temperatures and chemicals.

		Kruskal Wallis			
		$\boldsymbol{X_2}$	**df**	**p**	
Organic matter removed	Low organic	11.41	2	<0.01	Reagent only (H_2O_2, Fenton's and KOH)
	High organic	9.58	2	<0.01	
	Low organic	10.43	5	>0.05	Reagent and temperature(H_2O_2, Fenton's and KOH at 40 and 50^oC)
	High organic	15.69	5	<0.01	
Recovery efficiency (inorganic soil)	fibres	17.26	2	<0.01	By treatments (NaCl, $ZnCl_2$ and canola oil)
	big	12.29	2	<0.01	
	PET fragments	48.78	2	<0.01	
	PET fibres	59.25	2	<0.01	
	HDPE (0.5-1 mm)	8.16	2	<0.05	
	HDPE (0.25- 0.5 mm)	15.6	2	<0.01	
	Big PVC	44.31	2	<0.01	
	PVC (0.25- 0.5 mm)	46.33	2	<0.01	
	Big PS	8.32	2	<0.05	
	PS (0.25- 0.5 mm)	9.66	2	<0.01	
	PP fragments	11.99	2	<0.01	
Recovery efficiency (organic soil)	Fibres	9.14	8	>0.05	By treatments (H_2O_2 followed by NaCl, $ZnCl_2$ and canola oil)
	Fragments (0.25- 0.5 mm)	14.84	8	>0.05	
	PET fragments	18.89	2	<0.01	
	PET fibres	6.49	2	<0.05	
	PVC big	25.98	2	<0.01	
	PVC (0.25- 0.5 mm)	23.06	2	<0.01	
	PS (0.25- 0.5 mm)	20.64	2	<0.01	
HQI	Treatments	5.69	5	>0.05	At 40 and 50^oC: H_2O_2, Fentons and KOH. NaCl, $ZnCl_2$ and canola oil, ultrasound.
	Chemicals	2.96	2	>0.05	
	Temperatures	0.02	1	>0.05	

Table S 3. Statistical analysis results for Wilcox tests to measure differences in: the amount of organic matter removed from samples of high and low initial organic at 40 and 50^{o}C; the amount of organic removed across all treatments in low and high organic; and the recovery efficiency in inorganic soils using canola oil with and without the use of ultrasound.

		Wilcox Test		
		W	p	
Organic matter removed	Low organic	34	>0.05	Between temperatures (40 and 50^{o}C)
	High organic	60	>0.05	
	All treatments	104	>0.05	Between low and high organic
Recovery efficiency (inorganic soil)	With and without ultrasound	-1.805	>0.05	Canola oil only

Table S 4. Statistical analysis results for one-way ANOVA's to measure differences in: the amount of organic removed across reagents with and without the inclusion of dispersant; recovery efficiency of total microplastics, fragments and small microplastics from inorganic soils using different density separation treatments; and recovery efficiency of total microplastics, fragments and big microplastics from organic soils using different treatments combining digestion and density separation.

		ANOVA			
		F	df	p	
Organic matter removed	Dispersant vs. no dispersant	20.61	2,12	<0.01	Between reagents (H_2O_2, Fenton's and KOH)
Recovery efficiency (inorganic soil)	Total microplastics	20.77	2,51	<0.01	Difference between treatments (NaCl, $ZnCl_2$ and canola oil)
	Fragments	7.34	2,51	<0.01	
	small	4.11	2,51	<0.05	
Recovery efficiency (organic soil)	Total microplastics	7.95	2,51	<0.01	Difference between treatments
	fragment	8.08	2,45	<0.05	
	big	5.83	2,45	<0.01	

Table S 5. Statistical analysis results for t-tests to measure differences in the recovery efficiency in inorganic soils using ZnCl2 and NaCl with and without the use of ultrasound.

		T Test		
		t	df	p
Ultra sound vs. no ultrasound	$ZnCl_2$	1.14	32	>0.05
	NaCl	1.18	34	>0.05

Table S 6. Statistical analysis results for Spearman's Rank correlations to determine relationships between; the amount of clay in inorganic soils and the recovery efficiency; the organic content in a sample and the recovery of total microplastics, fragments, small microplastics, PET fragments and small LDPE.

		Spearman's rank	
		r_s	p
	Amount of clay in sample vs. recovery of total microplastics	0.04	>0.05
Organic matter content vs. recovery with canola oil	Total microplastics	0.5	<0.05
	Fragment	0.57	<0.05
	small	0.48	<0.05
	PET fragments	0.69	<0.01
	Small LDPE	0.55	<0.05

Figure S 1. Percentage organic matter removed from soils with initially high organic content (~70%) using three digestion methods (Fenton's reagent, hydrogen peroxide and potassium hydroxide) with and without the use of a dispersant (sodium hexametaphosphate)

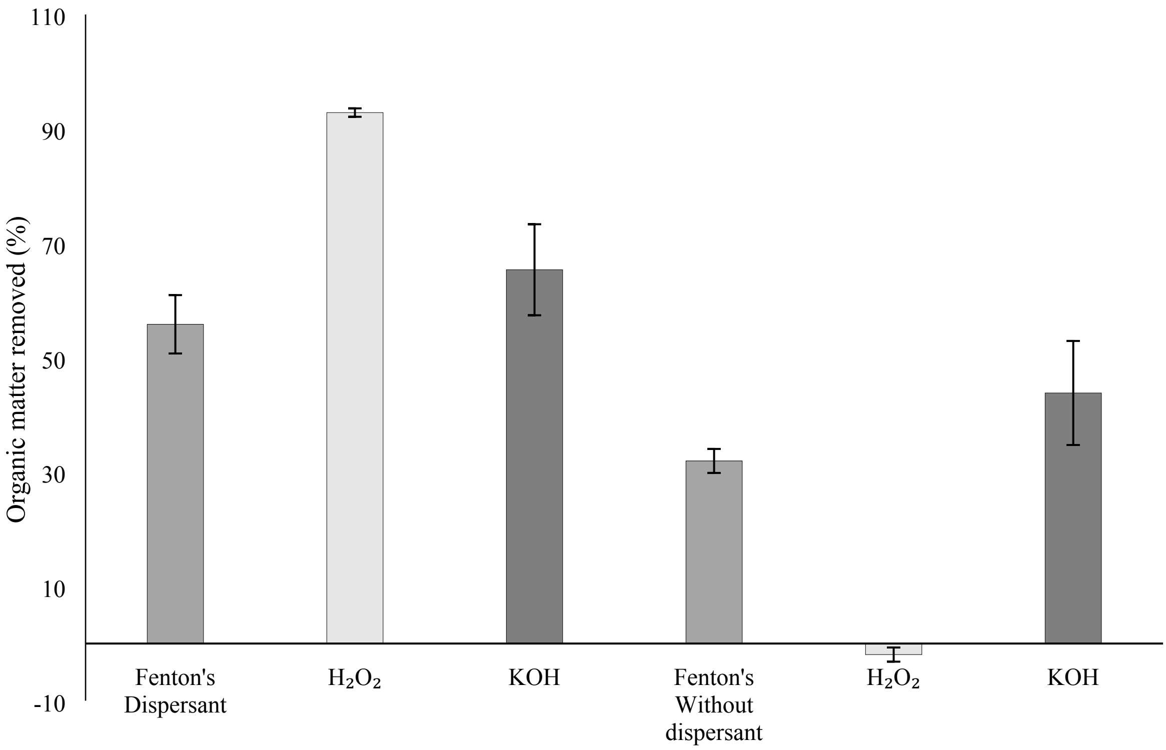

Figure S 2. Total microplastic recovery from low organic (left) and organic (right) soils using the three density separation methods ($ZnCl_2$, NaCl and canola oil) in the first and second round of extraction.

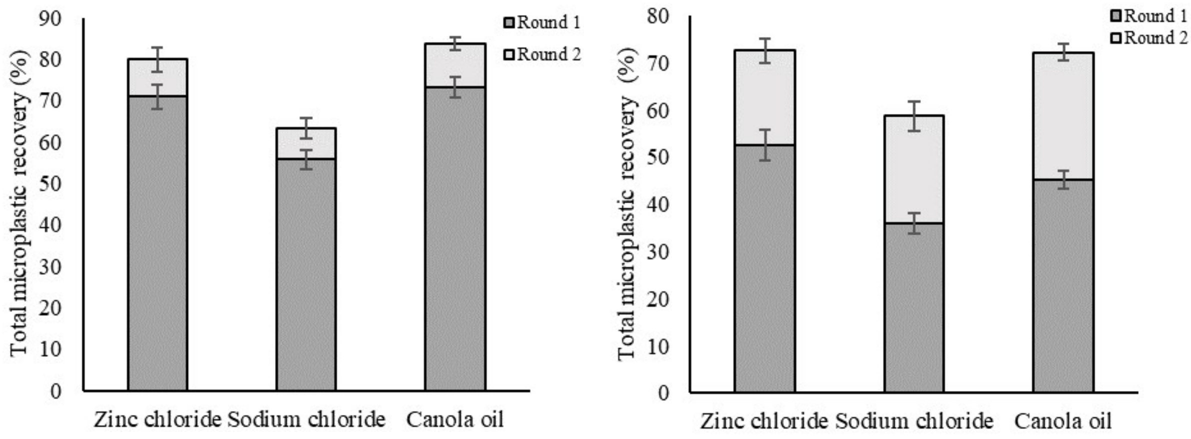

Figure S 3. Total microplastic recovery from inorganic soils using the three density separation methods ($ZnCl_2$, NaCl and canola oil) with (+US) and without (-US) the inclusion of an ultrasound step

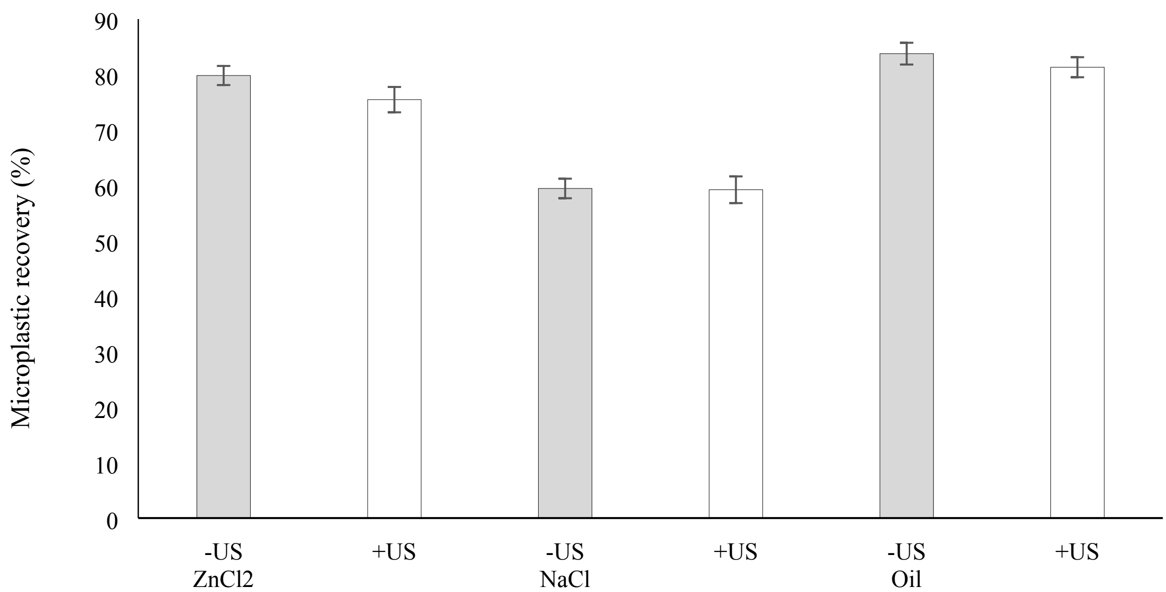

Figure S 4. FT-IR spectra for a sample of microplastic spikes using the experiment when treated with density treatments involving NaCl, $ZnCl_2$, oil and ultrasound (a. big PVC b. small PVC c. small PS d. small LDPE).

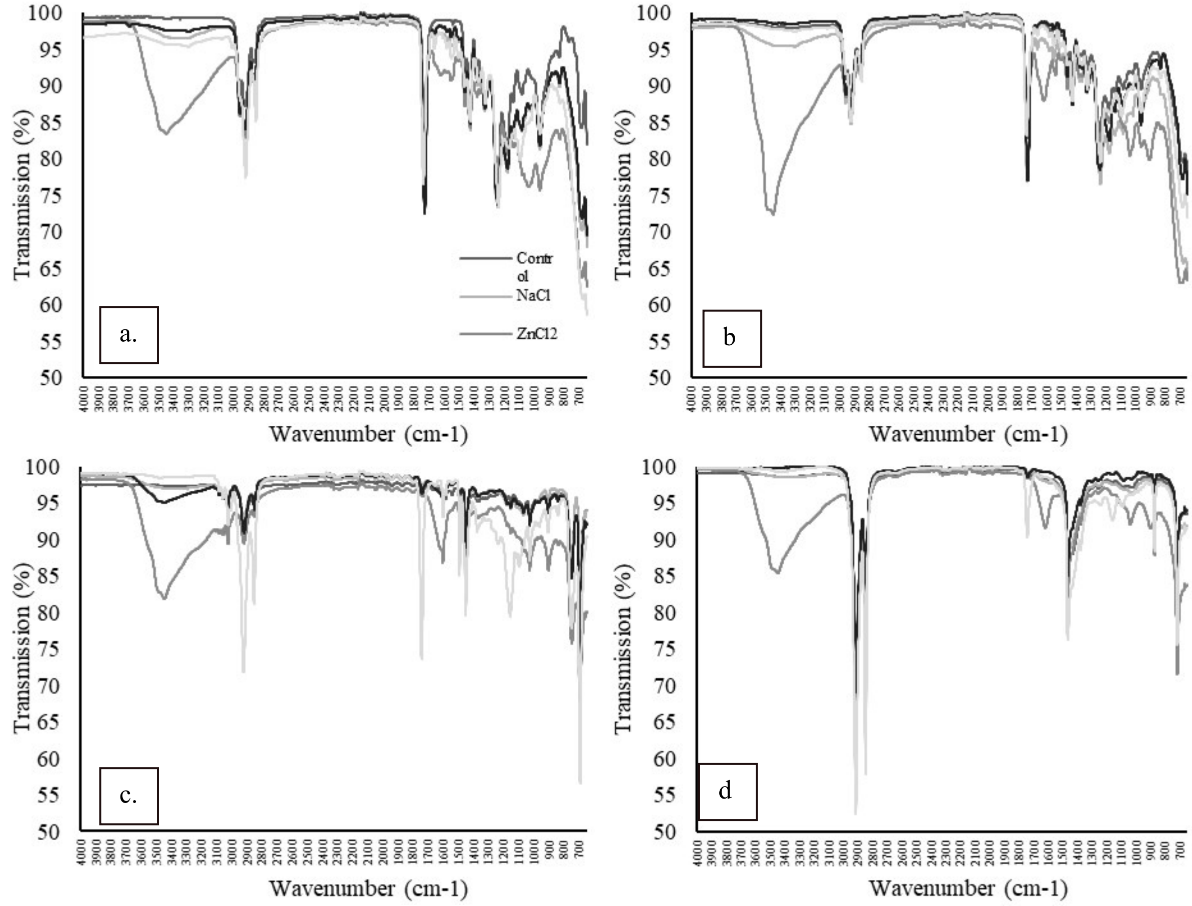

Figure S 5. FT-IR spectra for a sample of microplastic spikes using the experiment when treated with digestion treatments involving Fenton's reagent, potassium hydroxide and hydrogen peroxide at 40 and 50^{o}. (a. small LDPE b. PET fragments c. PP fragments d. large PVC)