Supporting Information

Identification of Microplastics Extracted from Field Soils Amended with Municipal Biosolids

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Municipal biosolids (MBs), sourced from a secondary-level wastewater treatment facility with anaerobic digestion, were applied to an agricultural field, shown in Figure S1.

Figure S1 Image of MBs application to the field that highlights the heterogeneity in the field application.

In order to compare the measured FTIR data with the Open Specy and FLoPP / FLoPP-e spectral libraries, library polymers were filtered such that only polymers with 10+ entries are included, leaving 14 possible search polymers (Table S1).

Table S1 List of 14 selected polymers from OpenSpecy, FLoPP/FLoPP-e, and SLoPP/SLoPP-e

Polymer	Entries (FTIR)	Entries (Raman)
Polyethylene	233	86
PET	175	21
Polypropylene	94	52
Polyamide	34	30
Polystyrene	34	29
Cellulose	31	25
Polyester	29	27
Polyurethane	27	16
PVC	21	20
Polyethylene vinyl acetate	18	5
ABS	17	11
Polycarbonate	16	11
PMMA	11	7
Polylactic acid	10	2

Multiple extractions were performed on the PE spiked sandy soil samples for each of $CaCl_2$, canola oil, and NaI/NaCl flotation solutions. Each of the TGA curves can be found in Figure S2.

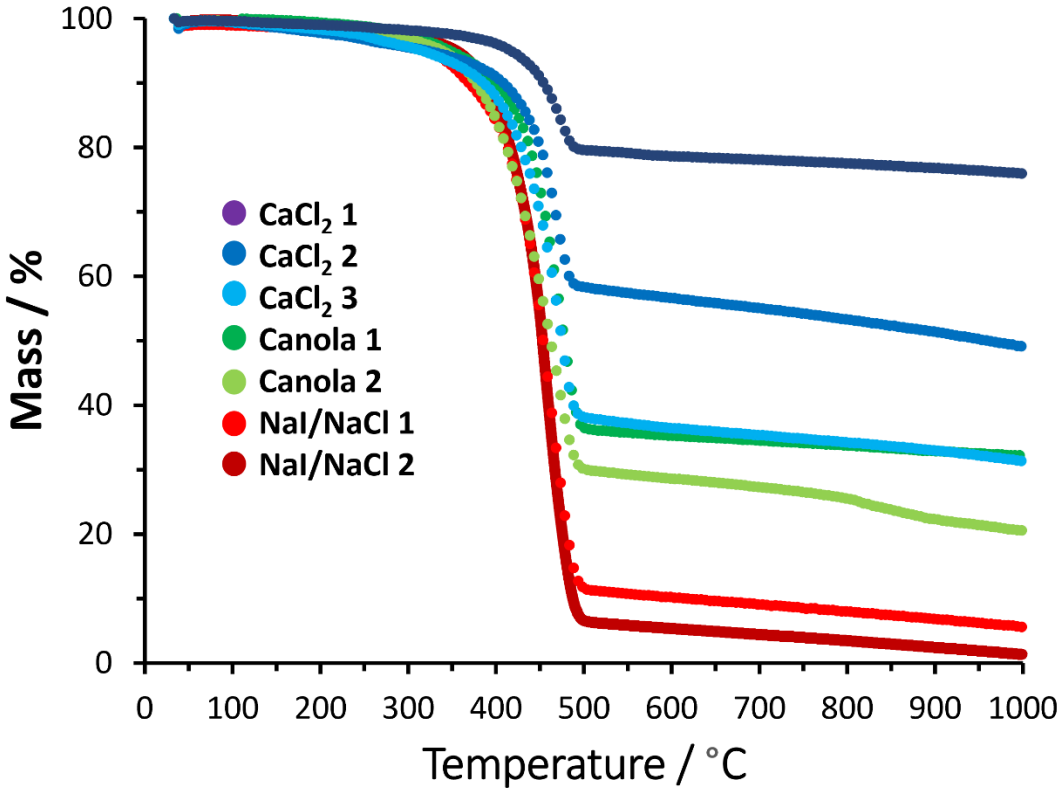

Figure S2 TGA plots comparing the efficacy of different density separation solutions ($CaCl_2$ at 1.32 g/cm^3, canola oil, and NaI/NaCl at 1.5 g/cm^3) used in the extraction of spiked PE beads in a sandy soil.

The NaI/NaCl flotation solution was also tested on a higher density polymer (PTFE) to evaluate its extraction efficiency (Table S2).

Table S2 PTFE extraction from spiked sandy soil by density separation (flotation) in NaI/NaCl solution (indicative values are shown).

Flotation Solution	PTFE mass fraction (in %) in extracted sample	PTFE recovery efficiency (in % of initial PTFE spike)
NaI/NaCl	35	9

Visual analysis was performed on the debris extracted from the clay loam soils before and after biosolid amendment, with plastics visible in Figure S3.

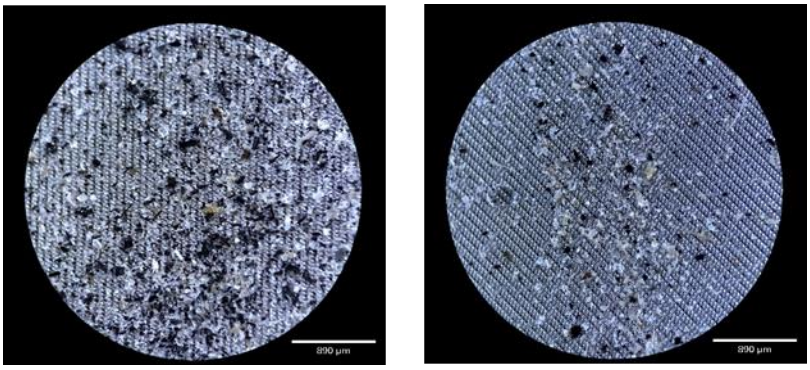

Figure S3 Plastic debris extracted from clay loam soil without biosolids amendment (left) and with biosolids amendment (2 wt. %) (right).

TGA curves of MBs and MB-spiked clay loam soils were analyzed in order to compare the DTG curve area against the MB concentration. The results of this analysis can be found in Table S3.

Table S3 TGA measurement results (integrated DTG peak areas and corresponding standard uncertainties) of 5 g MB (no soil) and 0-5 wt.% MB addition to clay loam soil.

Sample	(400-500) °C DTG peak	total (200-500) °C DTG signal
5 g MB	178 ± 59	586 ± 87
5 wt. % MB	91 ± 49	295 ± 76
2 wt. % MB	17 ± 11	119 ± 17
0 wt. % MB	24± 59	91 ± 33

Analysis was performed on samples from the agricultural field before, 1 month after, and 1 year after MB treatment. The % mass change for each sample in TGA can be found in Table S4.

Table S4 Indicative TGA values of mass fraction loss (in %) in the (400–500) ℃ temperature range for soil collected from an agricultural field before and after MB treatment.

Sample	Mass fraction loss (in %) attributed to MB
Pre-Biosolid	14
1 month post	16
1 year post	10

Multiple samples containing varied initial sample mass of 1 month post MB treatment soils were analyzed using TGA and their DTG curves were analyzed in the 400-500 ˚C and 200-500˚C range, as found in Table S5.

Table S5 TGA measurement results (integrated DTG peak areas and corresponding standard uncertainties) of multiple extractions on clay loam soil 1-month post-biosolids application.

Sample	(400-500) °C DTG peak	total (200-500) °C DTG signal
50 g A	< 13	285.0 ± 14.2
50 g B	92.7 ± 3.6	142.8 ± 4.3
50 g C	43.8 ± 2.6	90.2 ± 3.3
750 g A	< 7	145.1 ± 8.5
750 g B	33.2 ± 1.2	> 33.2
2500 g	102.5 ± 3.1	404.9 ± 13.5

Plastic pieces found post-extraction were examined using Raman and FTIR (Agilent Cary 630 FTIR with a diamond ATR accessory was used here) to verify their composition, with representative FTIR and Raman spectra found in Figure S4, respectively.

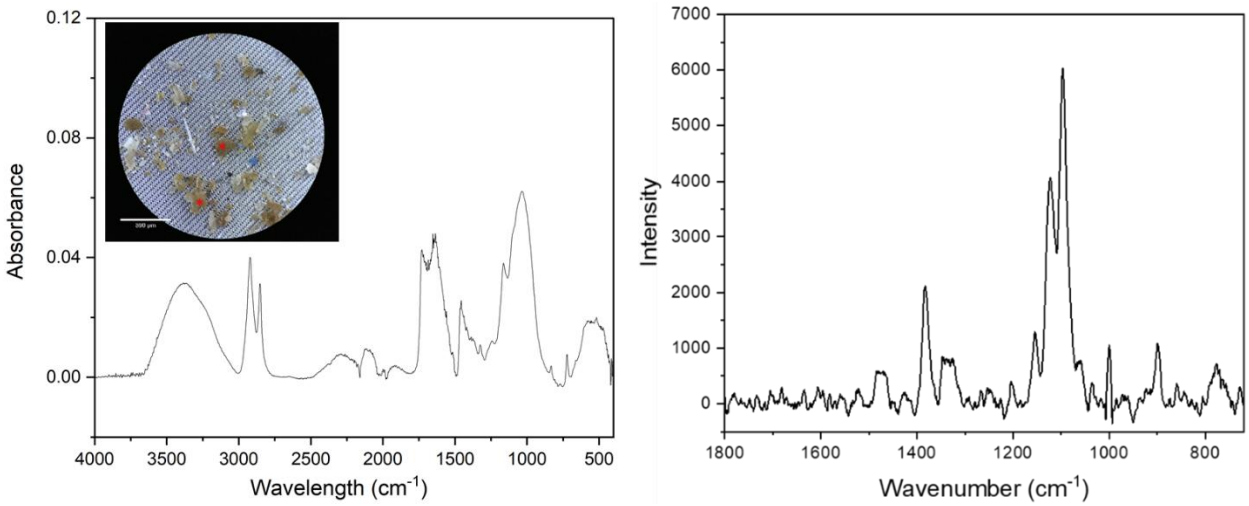

Figure S4. ATR-FTIR and Raman spectra of selected pieces from 5 g municipal biosolids filter debris (labeled with red stars). The functional groups match the characteristics of cellulose.

Typical 3D FTIR spectra against temperature and a representative absorbance versus wavelength FTIR spectrum are presented in Figure S5 to illustrate one set of measurements on 5 g MB sample (Jung et al., 2023).

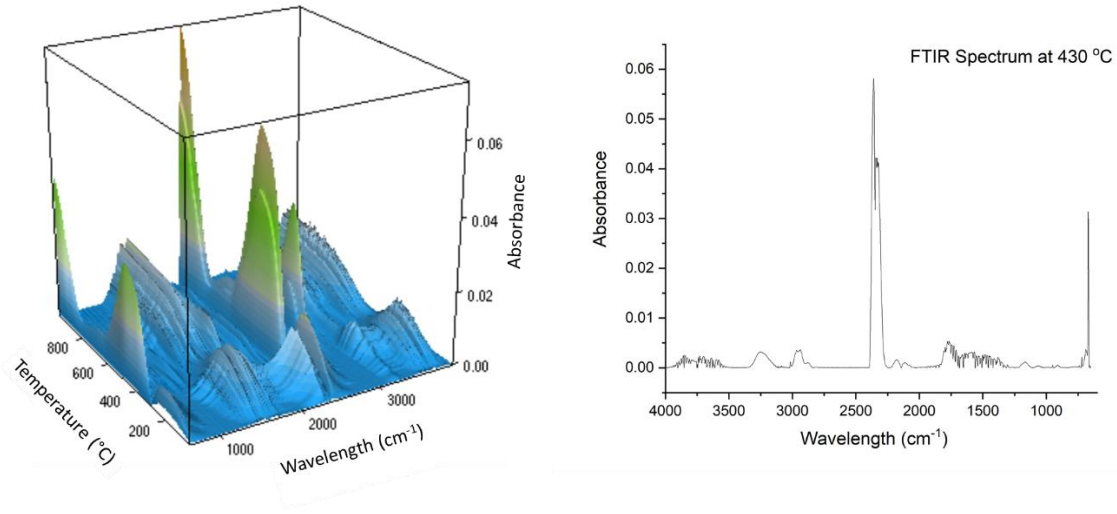

Figure S5. 3D FTIR spectrum of 5 g of municipal biosolids and representative FTIR spectrum at 430 °C.

Polymer Identification (PID) heat maps were created from the FTIR data over a range of temperatures. Figure S6 shows these plots for a number of control samples, as well as a clay loam samples with 5 g of MB.

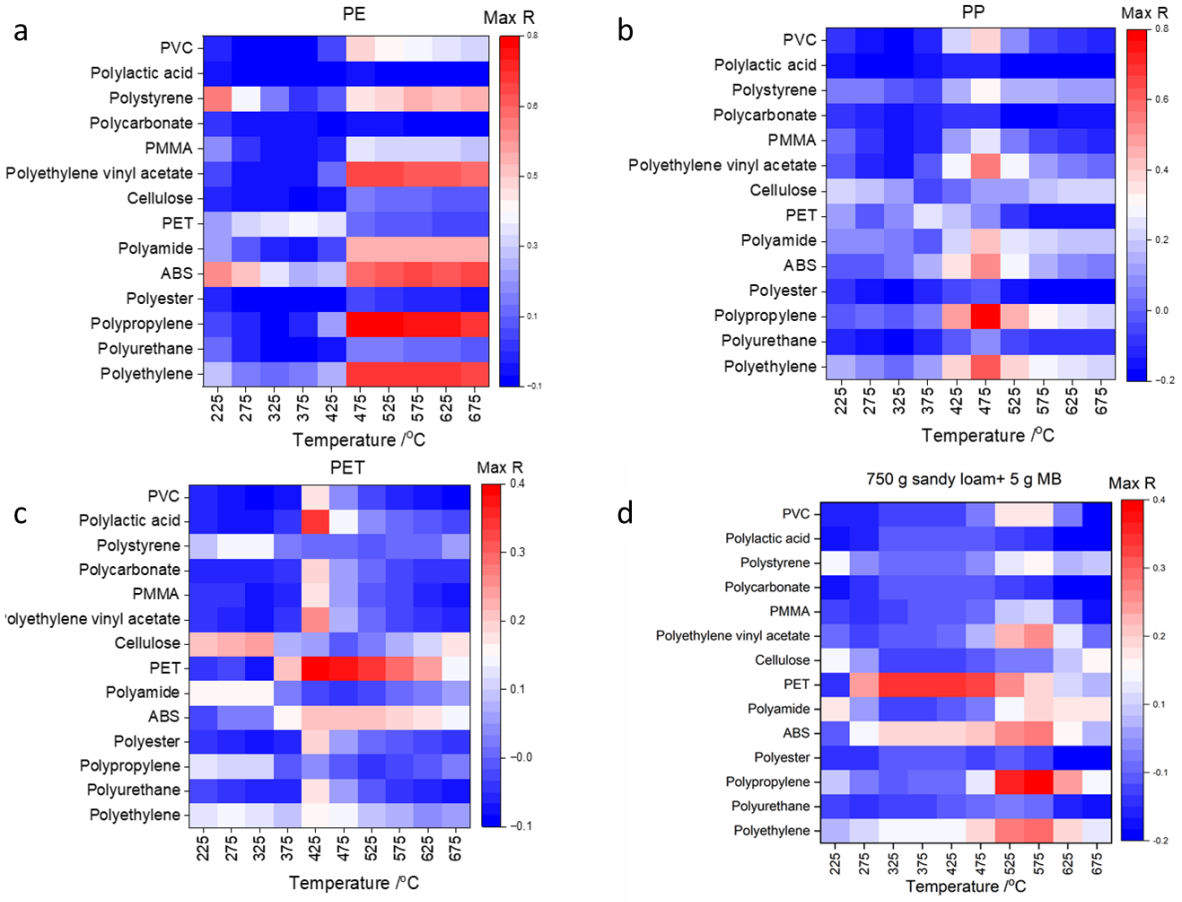

Figure S6 PID heat-map of control plastics a) PE, b) PP, c) PET and d) 750 g clay loam with 5 g spiked MB.

Different microplastic particles were identified using Raman microscopy. Many measured particles suffered from relatively high background fluorescence and low signal-to-noise, but plastics such as PE and PP were observed (Figure S7). Significant amounts of cellulosic material remained.

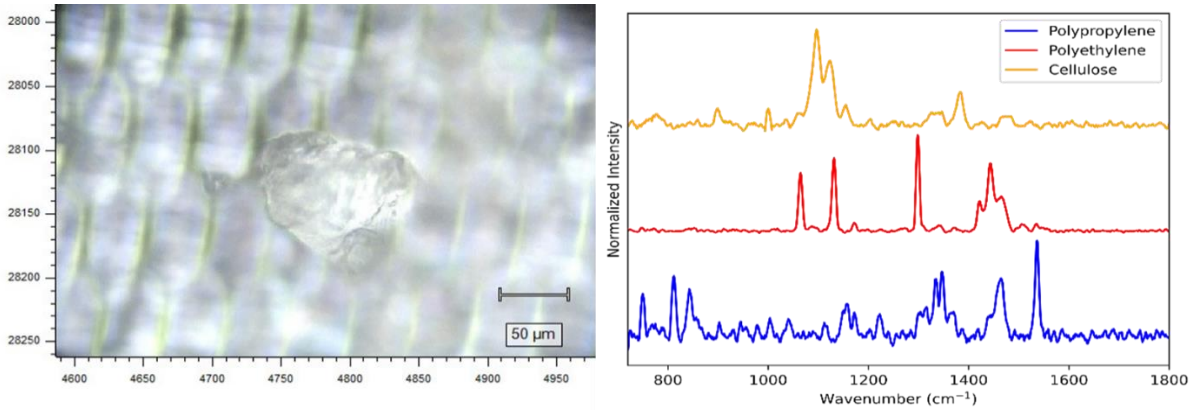

Figure S7 Example processed Raman spectra of polypropylene (with an optical image on the left), polyethylene and cellulose.

References

Jung, S., Raghavendra, A., Patri, A., 2023. Comprehensive Analysis of Common Polymers using Hyphenated TGA-FTIR-GC/MS and Raman Spectroscopy towards a Database for Micro- and Nanoplastics Identification, Characterization, and Quantitation. NanoImpact 30, 1000467. https://doi.org/10.1016/j.impact.2023.100467