Occurrence of Microplastics In Biosolids In Wastewater Treatment Plants in Deep East Texas

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Abstract

Wastewater treatment plants (WWTPs) act as secondary sources of microplastic pollution into the environment. Microplastics (MPs) come in various types, namely; filaments, fragments, rods, and beads. They range in size from 5 mm to 1 µm. In this study, spectroscopic, chromatographic, and optical properties of biosolids from Deep East Texas were analyzed via Fourier transform infrared spectroscopy, ion chromatography (IC), and optical microscopy. Samples were taken from San Jacinto (SJWWTP), San Augustine (SAWWTP), Jasper (JWWTP), Lufkin (LWWTP), Nacogdoches (NWWTP), and the Neches Compost Facility (Soil Therapy Compost, STC). Using FT-IR spectral peaks at ν(O-H) and ν(N-H) (~3300 cm^{-1}), ν(C-H) 2930 cm^{-1}, ν(C=O) 1677 cm^{-1}, and δ(Si-O-Si) 815 cm^{-1}. Via IC analysis PO_4^{3-} concentrations were determined above USEPA drinking water regulations in all samples. Concentrations of F^-, Cl^-, NO_2^-, NO_3^-, Br^-, SO_4^{3-} were below USEPA drinking water regulations. Via optical microscopy of LWWTP sample, many types of MPs were identified. This study is useful because it adds to the emerging research on microplastic contamination from WWTPs in Deep East Texas

Objectives for the Study

- Evaluate the presence of microplastics in biosolids from wastewater treatment plants in Deep East Texas.
- Analyze the efficiency of wastewater treatment plants from Deep East Texas in removing microplastics from biosolids.
- Use spectroscopy (FT-IR, SEM/EDX,) and ion chromatography (IC) to characterize microplastics in biosolids.

Biosolid Samples

Figure 1: Unextracted Biosolid Samples

Materials and Methods

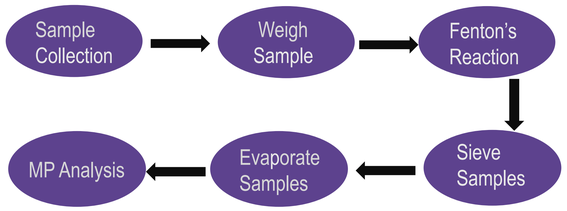
1. Reagents

- Combined Seven Anion Standard (ThermoScientific Dionex) was used for IC. It was diluted 50x, 40x, 30x, and 20x for preparation of standard for calibration curve.
- Fenton's reagent: was used for extraction of MPs.
  - 20 mL of 30% hydrogen peroxide and 250 mL of 0.05 M $FeSO_4.7H_2O$ was used (Flinn Scientific laboratory grade).

2. Instrumentation

- PerkinElmer Spectrum 100 FT-IR Spectrometer
- ThermoScientific Dionex ICS-2100
- Olympus Brightfield Microscope (1X-HOS, CKX3-SLP) with Infinity 3 Lumera attachment

3. Extraction



Results (FT-IR)

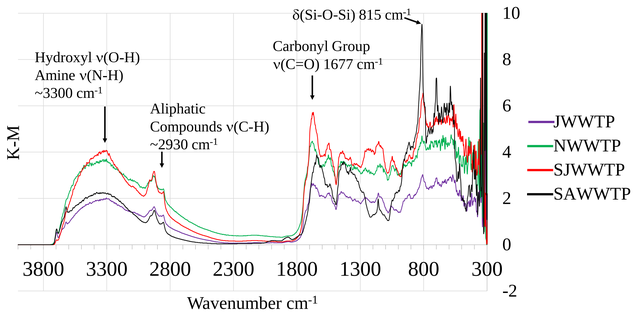


Figure 2: FT-IR Overlay of unextracted biosolid samples from Jasper, Nacogdoches, San Augustine, and San Jacinto wastewater treatment plants (JWWTP, NWWTP, SAWWTP, and SJWWTP)

Results (IC)

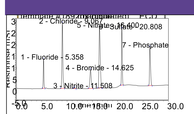


Figure 3: Chromatograph showing retention times of anions (Dionex ICS-2100 ion chromatograph, a DionexIonPac AS19 analytical column (2 x 250 mm) thermostated at 30 ºC, guard column (IonPac AG19), KOH eluent (ECG III KOH), flow rate of 0.25 mL/min, a suppressor column ASRS-2 mm, and operating pressure of 1200-2300 PSI.

Results (IC) (cont'd)

Table 1: Average and Standard Deviations of ions in WWTP Biosolid Samples

	F^- (mg/g)	Cl^- (mg/g)	NO_2^- (mg/g)	Br^- (mg/g)	NO_3^- (mg/g)	SO_4^{2-} (mg/g)	PO_4^{3-} (mg/g)
STC	0.03 ± 0.06	0.17 ± 0.04	0.48 ± 0.23	0.00 ± 0.00	3.97 ± 0.16	4.10 ± 0.22	2.52 ± 0.70
JWWTP	0.01 ± 0.01	0.23 ± 0.03	1.51 ± 0.13	0.02 ± 0.00	0.08 ± 0.04	0.27 ± 0.02	4.16 ± 1.23
LWWTP	0.05 ± 0.02	0.96 ± 0.03	0.57 ± 0.34	0.01 ± 0.00	3.24 ± 0.19	48.99 ± 9.50	6.12 ± 1.18
NWWTP	0.02 ± 0.02	0.62 ± 0.03	1.48 ± 1.57	0.01 ± 0.00	2.87 ± 0.09	2.25 ± 0.05	8.25 ± 2.46
SAWWTP	0.01 ± 0.00	0.04 ± 0.01	0.50 ± 0.09	0.00 ± 0.00	0.06 ± 0.03	0.52 ± 0.14	2.62 ± 2.72
SJWWTP	0.01 ± 0.01	0.48 ± 0.01	1.27 ± 0.77	0.00 ± 0.00	0.05 ± 0.03	0.54 ± 0.02	7.77 ± 1.36

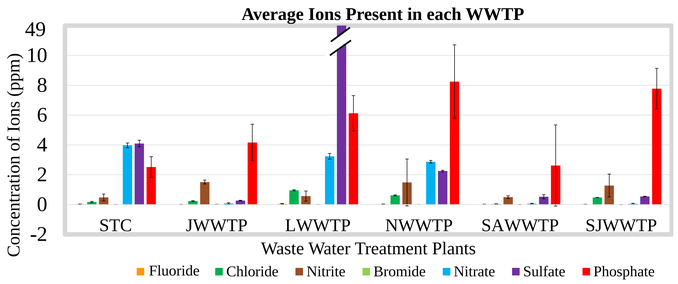


Figure 5: Average Ions Present in each WWTP (Soil Therapy Compost, Jasper, Lufkin, Nacogdoches, San Augustine, San Jacinto)

Results (Optical Microscopy)

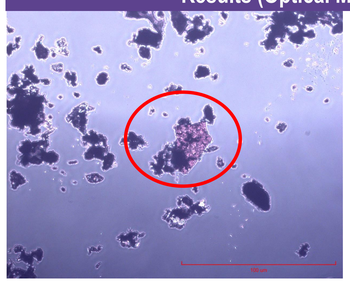


Figure 6: LWWTP biosolid sample: 150 µm < x < 850 µm **fragment**

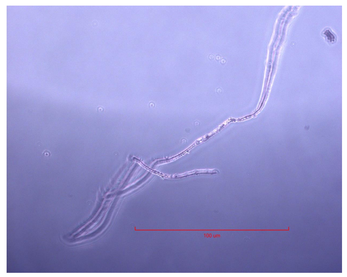


Figure 7: LWWTP biosolid sample: 150 µm < x < 850 µm **filament**

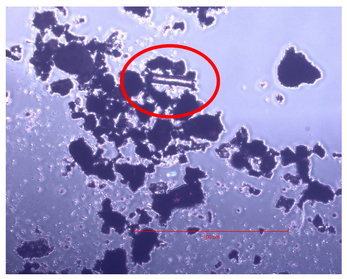


Figure 8: LWWTP biosolid sample: 150 µm < x < 850 µm **rod**

Results (Optical Microscopy) (cont'd)

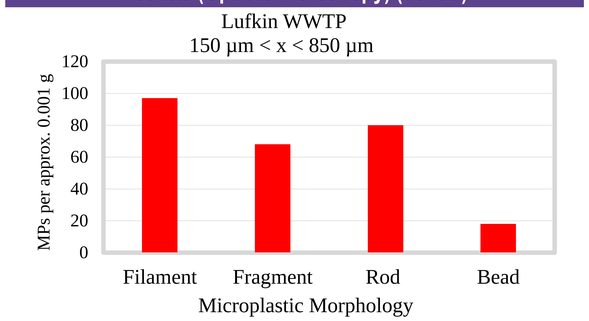


Figure 9: LWWTP biosolid sample 150 µm < x < 850 µm number of microplastic types

Conclusions

FT-IR

- ν(O-H) (phenol/water/alcohol) or ν(N-H) amine ~3300 cm^{-1}.
- ν(C-H) at 2930 cm^{-1} aliphatic .
- Carbonyl group ν(C=O) at 1677 cm^{-1}. Potential carboxylic acid and polypeptides.
- Silicon δ(Si-O-Si) at 815 cm^{-1}.
- Similar shape and peaks across samples. Same functional groups across samples.

IC

- PO_4^{3-} had the highest concentration across samples. Above USEPA regulated concentrations for drinking water (0.015 ppm).
- SO_4^{2-} and NO_3^- were high in the STC, LWWTP, and NWWTP samples. (SO_4^{2-} = 250 ppm, NO_3^- = 10 ppm (USEPA)).
- F^-, Cl^-, NO_2^-, and Br^- were low across the samples. Under USEPA regulated concentrations.

Optical Microscopy

- Filaments, rods, and fragments were the most common type of microplastic found in the LWWTP sample. Beads were the least common.

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