

# STABILITY OF PFAS IN SPENT FLUORO-SORB® 200 ADSORBENT

## LEACHING OF PFAS USING THREE EPA PROTOCOLS

### INTRODUCTION

A landfill in the northeastern part of the United States used FLUORO-SORB® 200 Adsorbent for removal of per- and polyfluoroalkyl substances (PFAS) from a landfill leachate treatment system. FLUORO-SORB 200 Adsorbent is a granular media (shown in Figure 1) that has a high adsorption capacity for a variety of PFAS and resists the competitive effects of other contaminants and water quality parameters that can interfere with PFAS treatment.<sup>1</sup> The influent for the FLUORO-SORB 200 Adsorbent treatment system was raw landfill leachate that was previously treated with a membrane bioreactor, which provides biological treatment, as well as ultrafiltration. The resulting treated landfill leachate (see Table 1 for chemistry parameters) had high concentrations of PFAS, as well as total organic carbon (TOC) and total dissolved solids (TDS) and was piped to the FLUORO-SORB 200 Adsorbent treatment system. The treatment system consisted of two 6-foot diameter vessels, arranged in series in a lead-lag configuration, and each vessel contained 120 cubic feet or 6,000 lbs. of FLUORO-SORB 200 Adsorbent. System parameters are given in Table 2.

**TABLE 1: MBR TREATED LANDFILL LEACHATE CHEMISTRY PARAMETERS**

PARAMETER (AVERAGE) VALUES	VALUE
Total MA-6 PFAS	10,700 ng/L
PFOA	6,500 ng/L
PFHpA	1,800 ng/L
PFNA	415 ng/L
PFDA	450 ng/L
PFOS	590 ng/L
PFHxS	870 ng/L
TOC	280 mg/L
TDS	16,000 mg/L
TDS Nitrate (as nitrogen)	1,100 mg/L

**FIGURE 1: FLUORO-SORB® 200 ADSORBENT TREATMENT MEDIA**



Note: MA-6 PFAS, Massachusetts PFAS standard for public drinking water supplies (sum of six PFAS must be less than 20 ng/L).

**TABLE 2: PFAS TREATMENT SYSTEM PARAMETERS - TWO VESSELS**

PARAMETER (AVERAGE) VALUES	VALUE
Vessel Diameter	6 feet
Vessel Height	6 feet
FLUORO-SORB 200 Adsorbent Bed Height	4.25 feet
FLUORO-SORB 200 Adsorbent Bed Volume	900 gallons/120 cubic feet
Average Flow Rate	100 gallons per minute (gpm)/144,000 gallons per day
Empty Bed Contact Time (EBCT)	9 minutes
Hydraulic Loading Rate	3.5 gpm/ft <sup>2</sup>

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UPDATED: JULY 2024

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FORM: FS-109 STABILITY OF PFAS IN SPENT FLUORO-SORB ADSORBENT\_AM\_EN\_202407

The FLUORO-SORB® 200 Adsorbent treatment system continuously removed the sum of six PFAS (PFOA, PFHpA, PFNA, PFDA, PFOS, and PFHxS) from the influent to below 20 ng/L for 5,000 to 10,000 bed volumes (30 to 60 days) before breakthrough occurred from the lead vessel. The initial compound to breakthrough is typically PFHpA, which is considered a short-chain PFAS and is harder to remove from contaminated water than the longer chain PFAS. To avoid further break through, the spent FLUORO-SORB 200 Adsorbent, which is laden with PFAS from the treatment system is physically removed from the treatment vessel and fresh FLUORO-SORB 200 Adsorbent is loaded so that PFAS removal operations can continue.

## LEACHING TESTS DESCRIPTION

A sample of spent FLUORO-SORB 200 Adsorbent was obtained and sent to Eurofins USA, Lancaster, Pennsylvania for PFAS testing. The goal of the testing was to assess the stability of PFAS in the spent FLUORO-SORB 200 Adsorbent and understand the potential for leaching of PFAS from the solid. This information can aid in making informed decisions about disposal of spent FLUORO-SORB 200 Adsorbent. The PFAS content of the spent FLUORO-SORB 200 Adsorbent was determined using a modified EPA Method 537. The water leachable PFAS from the spent media was determined using the leaching protocols EPA Methods 1311, 1312 and 1313, described in Table 3. A total of 14 PFAS were measured using Eurofins 537 IDA, which is a modified version of US EPA Method 537 with Isotope Dilution.<sup>2</sup> The 14 PFAS compounds analyzed for were perfluorobutanoic acid (PFBA), perfluoropentanoic acid (PFPeA), perfluorohexanoic acid (PFHxA), perfluoroheptanoic acid (PFHpA), perfluorooctanoic acid (PFOA), perfluorononanoic acid (PFNA), perfluorodecanoic acid (PFDA), perfluorobutanesulfonic acid (PFBS), perfluorohexanesulfonic acid (PFHxS), perfluorooctanesulfonic acid (PFOS), perfluorodecanesulfonic acid (PFDS), perfluorooctanesulfonamide (PFOSA), 6:2 FTS, and 8:2 FTS.

TABLE 3: LEACHING TESTS APPLIED TO SPENT FLUORO-SORB® 200 ADSORBENT				
	EPA Method 537 Modified for the preparation of soil samples <sup>3</sup>	TCLP, US EPA Method 1311	SPLP, US EPA Method 1312	LEAF, US EPA Method 1313
Full name	Determination of Selected Per- and Polyfluorinated Alkyl Substances in Drinking Water by Solid Phase Extraction (SPE) and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS).	Toxicity Characteristic Leaching Procedure	Synthetic Precipitation Leaching Procedure	Leaching Environmental Assessment Framework, Liquid-Solid Partitioning as a Function of Extract pH
Solution composition	Sample treated with proprietary solvent using ultrasonic extraction. Extractant was concentrated with heat/nitrogen and reconstituted in methanol.	Acetic acid	Mix of sulfuric acid and nitric acid in a 60 to 40 ratio	Concentrated nitric acid and potassium or sodium hydroxide or deionized water for natural pH
Solution pH	Does not apply	pH 4.2 to 5	pH 2.88	pH 2, pH 4, pH 5.5, Natural pH, pH 10, pH 13 (see Note)
Liquid-to-solid ratio	Proprietary	20 ml/g	20 ml/g	10 ml/g
Extraction time	1 hour	18 hours	18 hours	24 to 48 hours depending on particle size

Note: pH 13 data not presented as it was replicative of the pH 10 data.

## RESULTS

Results for PFAS leached from spent FLUORO-SORB 200 Adsorbent using the three leaching tests are presented in Figures 2 – 3 and Table 4. The 11 PFAS compounds included in the figures are PFDA, PFNA, PFOA, PFHpA, PFHxA, PFPeA, PFBA, PFOS, PFHxS, PFBS, and 6:2 FTS. The amount of PFAS leached by each leaching protocol, as compared to the amount measured in the spent FLUORO-SORB 200 Adsorbent, is presented for the sum of 11 PFAS in Figure 2 and for each PFAS in Table 4. Figure 3 shows the percent of PFAS in the spent FLUORO-SORB 200 Adsorbent that was leached as a result of applying each leaching protocol.

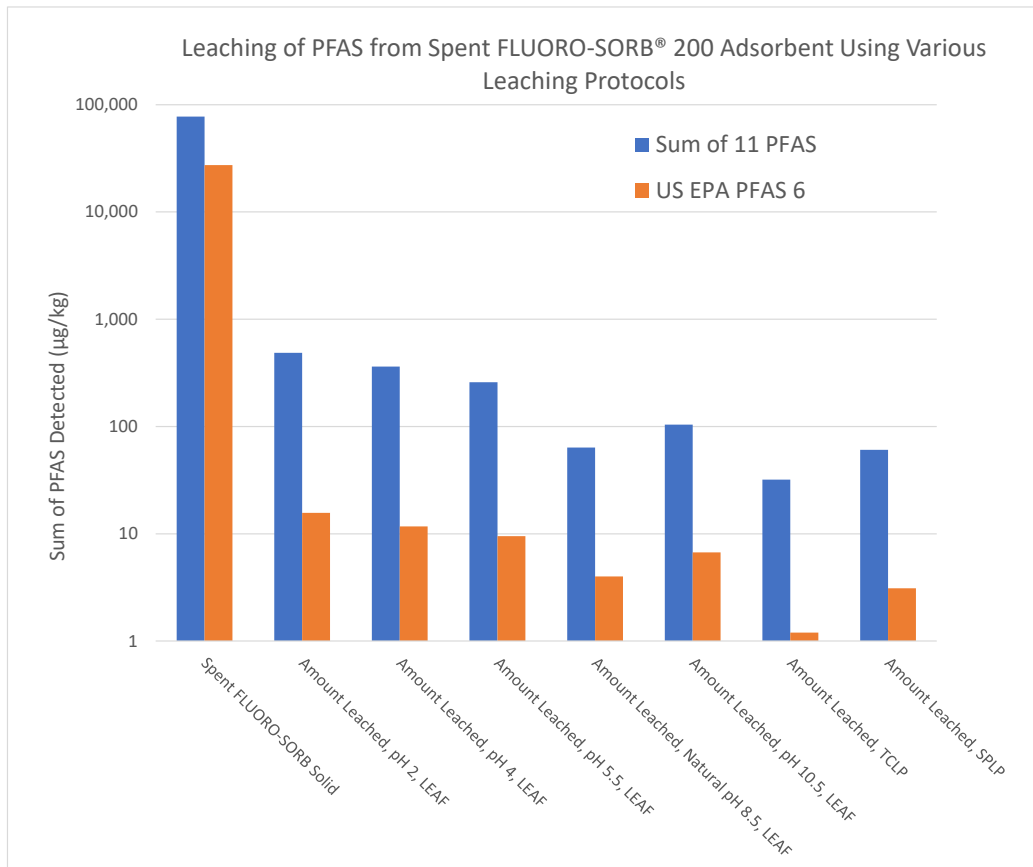


Figure 2: Sum of 11 PFAS Leached from Spent FLUORO-SORB® 200 Adsorbent

**TABLE 4: PFAS LEACHED FROM SPENT FLUORO-SORB® 200 ADSORBENT AS COMPARED TO ADSORBED AMOUNT, µg/kg**

PFAS	Spent FLUORO-SORB® 200 Adsorbent Solid	Amount Leached, pH 2, LEAF	Amount Leached, pH 4, LEAF	Amount Leached, pH 5.5, LEAF	Amount Leached, Natural pH 8.5, LEAF	Amount Leached, pH 10.5, LEAF	Amount Leached, TCLP	Amount Leached, SPLP
PFDA	420	0.5	0.5	0.5	0.5	0.5	0.01	0.01
PFNA	590	0.5	0.5	0.5	0.5	0.5	0.01	0.01
PFOA	9,400	1.2	0.92	1.2	0.5	0.75	0.18	0.56
PFHpA	7,800	10	8.9	6.3	1.6	2.5	0.68	1.82
PFHxA	35,000	200	150	120	27	47	14.6	28
PFPeA	4,600	140	100	64	16	27	7.8	14.4
PFBA	790	120	90	58	14	20	7.6	13
PFOS	330	0.5	0.5	0.5	0.5	0.5	0.01	0.15
PFHxS	1,000	0.5	1	0.5	0.5	0.5	0.01	0.02
6:2 FTS	1,400	0.5	0.5	0.5	0.5	0.5	0.08	0.22
PFBS	16,000	13	8.8	6.8	2	4.5	0.94	2.4
<b>Total PFAS</b>	<b>77,330</b>	<b>487</b>	<b>362</b>	<b>259</b>	<b>64</b>	<b>104</b>	<b>32</b>	<b>61</b>

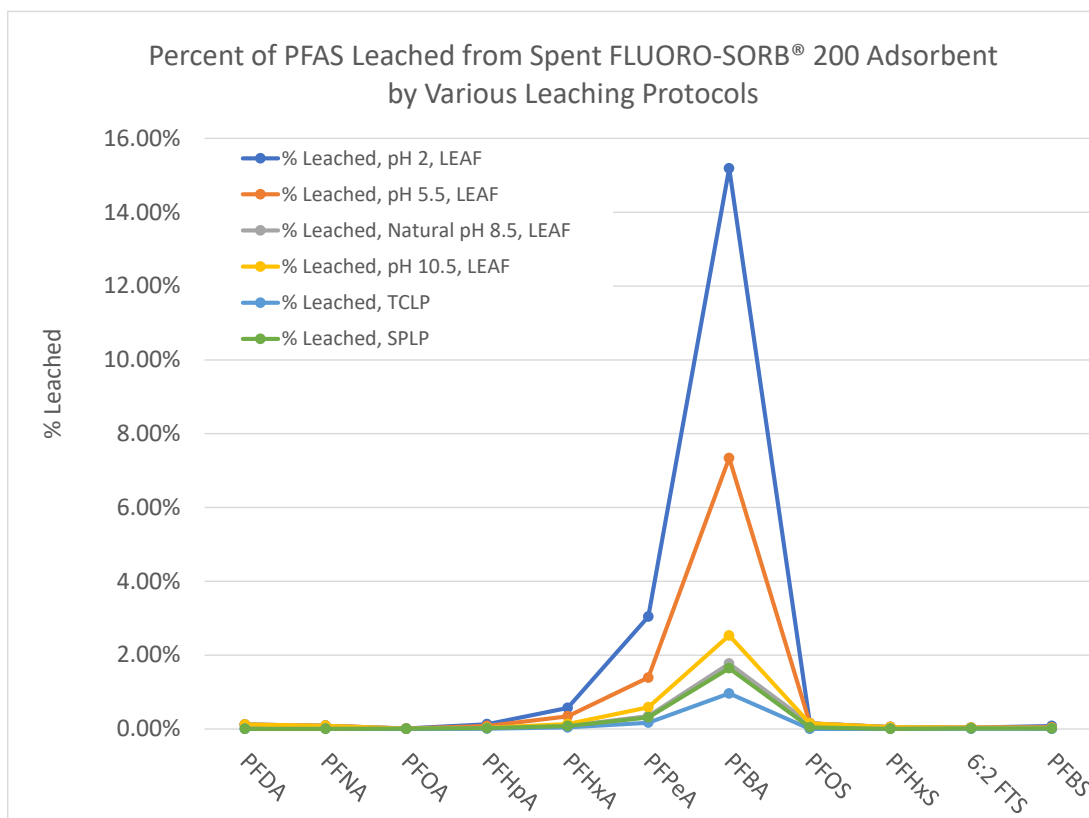


Figure 3: Percent of PFAS Leached from Spent FLUORO-SORB® 200 Adsorbent

## DISCUSSION

The total extractable PFAS content of the FLUORO-SORB® 200 Adsorbent spent media in this study was determined to be 77,330 µg/kg. The amount of PFAS leached from spent FLUORO-SORB 200 Adsorbent is significantly lower and, in some cases, below method detection limits for all the US EPA leaching protocols. Note that the liquid based concentrations that result from the leaching protocols were converted to solid based units using the liquid-to-solid ratio that was used for each test. The landfill leachate and the spent FLUORO-SORB 200 Adsorbent were dominated by the perfluoroalkyl carboxylic acids, like PFOA, PFHpA, PFHxA, PFPeA, and PFBA, showing that FLUORO-SORB 200 Adsorbent can be an effective adsorbent for short- and long-chain PFAS. The chemistry of FLUORO-SORB 200 Adsorbent allows PFAS adsorption by hydrophobic and electrostatic driving forces. These driving forces are important for short-chain PFAS adsorption, which rely primarily on the electrostatic Adsorbent attraction. The long-chain PFAS (i.e., any PFAS with 6 or more fluorinated carbons) were not able to be leached from the spent FLUORO-SORB 200 Adsorbent in any appreciable amount. The short-chain PFAS like PFPeA and PFBA, which typically do not have regulatory limits, did show 3 to 15% leaching in the US EPA LEAF 1313 protocol under the low pH conditions, as shown in Figure 3. It may be that cations and anions in the extraction fluid used to adjust the pH either competed with the anionic short-chain PFAS for adsorption on FLUORO-SORB 200 Adsorbent or neutralized the negative charge on these PFAS, thus reducing the electrostatic driving force for adsorption on FLUORO-SORB 200 Adsorbent. Note that the Natural pH 8.5 condition, which uses deionized water as an extraction fluid, showed a similar leaching profile to the TCLP and SPLP results. This indicates that the composition of the US EPA LEAF extraction fluid may be extracting more PFAS from the spent media than the other protocols.

## CONCLUSION

Based on the leaching protocols applied to the spent FLUORO-SORB 200 Adsorbent, leaching of regulated PFAS is not expected under environmental conditions, other than under very low pH conditions that likely are not be encountered in waste disposal facilities.

## REFERENCES

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2. Testing data contained in Eurofins test reports: J140291-1 and J140312-1.
3. Eurofins Test Document: T-PFAS-WI12031 Version 10 (Redacted)16-JUN-2020. Old reference 1-P-QM-WI9039643 (1-P-QM-WI9035864). A redacted version of this SOP is available in G-DC-FRM23907 due to the proprietary nature of its contents.