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Piezoelectric Ball Milling Treatment of PFAS-Laden Spent Resins

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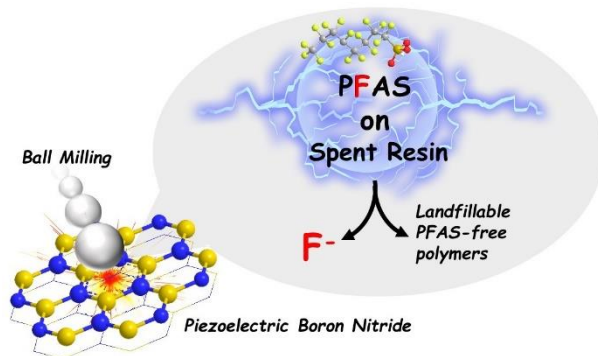
Abstract

Anion exchange resins (AERs) have been widely used for the removal of per- and polyfluoroalkyl substances (PFAS) from water. However, regardless of whether AERs are regenerable or single-use, the disposal of end-of-life AERs presents significant challenges. Conventional waste management strategies, such as landfill disposal and incineration, are increasingly under scrutiny and may face impending bans due to environmental concerns. This study introduces a non-thermal, solvent-free process for the effective destruction of PFAS on AERs by co-milling piezoelectric boron nitride (BN) powders with contaminated AERs and stainless steel (SS) balls. The approach demonstrated the destruction of PFOS and achieved 80% defluorination when applied to two types of AERs (PFA694E and AmberLite IRA 67) pre-loaded with PFOS. Comprehensive targeted PFAS analysis and suspect screening analysis of transformation products elucidated the reaction mechanisms and confirmed that the functional groups of AERs did not participate in the reaction. The process's effectiveness was further validated in treating field-collected single-use AERs (PFA694 and AmberLite PSR2 Plus) contaminated with PFAS mixtures of varying chain lengths and head groups. After treatment, PFAS levels on the AERs were reduced to below ng/g detection limits, and no PFAS release was observed in standard leaching tests.

Keywords: per- and polyfluoroalkyl substances, anion exchange resins, piezoelectric materials, boron nitride, ball milling

Synopsis: This study presents a non-thermal, solvent-free piezoelectric ball milling process for the destructive treatment of PFAS on AERs.

Graphical Abstract



Introduction

Per- and polyfluoroalkyl substances (PFAS) are a group of chemical pollutants highly recalcitrant to conventional water treatment processes. With the United States Environmental Protection Agency (USEPA) enforcing stringent PFAS standards, PFAS adsorption has become widely adopted in water treatment plants across the country. Anion exchange resins (AERs) are garnering increasing attention for PFAS removal, as they show higher adsorption capacity and resistance to natural organic matters (NOMs) than granular activated carbon (GAC).^{1,2}

Regeneration of AERs requires salt brine amended with organic solvent.³ The resulting regenerant, also known as still bottoms, from this process poses significant disposal challenges. The reported treatment strategies for the destruction of PFAS in still bottoms include electrochemical oxidation, UV/sulfite reduction, sonolysis, and plasma treatment.⁴⁻⁸ However, each method has its limitation: electrochemical oxidation of still bottom with high chloride concentrations can lead to the formation of halogenated byproducts, UV/sulfite reduction can be hindered by NOMs, sonolysis requires high energy input with sluggish PFAS degradation kinetics, and plasma treatment is less efficient for the removal of short-chain PFAS. It remains uncertain under what conditions the cost of regeneration, including the treatment of PFAS-laden still bottom solutions, can be justified by the savings achieved through the repetitive reuse of the AERs. For example, the USEPA concluded that the regeneration practice is less feasible at the full scale for drinking water facilities.⁹ More importantly, the loss of adsorption capacity (i.e., faster breakthrough) was observed after AER regeneration.¹⁰ Therefore, even regenerable AERs will eventually need to be disposed of after repeated service cycles. Manufacturers have introduced single-use AERs targeted specifically for PFAS. Some single-use AERs were found to outperform regenerable AERs.¹¹ However, the question remains: how should the spent single-use AERs be disposed of after service?

Landfilling and incineration are two interim options for managing spent sorbents.⁹ Landfilling without PFAS destruction merely postpones PFAS discharge until they migrate into the leachate. Incineration of PFAS-laden sorbents may emit greenhouse gas, hydrogen fluoride, and the product of incomplete combustion.¹² Though the thermal treatment of PFAS-laden GAC has been extensively summarized,^{13,14} studies specifically on the thermal treatment of PFAS-laden AERs are relatively scarce. A recent study proved the decomposition of AERs spiked with target PFAS

by inductive heating.¹⁵ However, the study did not close the fluorine balance, and fluorocarbon byproduct formation was not investigated. There has been no full-scale study evaluating the incineration of PFAS-laden AER.

This study provides a solvent-free and non-thermal option to treat PFAS-laden AERs at ambient conditions. We report that the co-milling of PFAS-loaded AERs (prepared in the lab and collected from the field) with piezoelectric boron nitride (BN) can readily destroy PFAS on AERs to fluoride (F⁻). Targeted analysis and suspect screening of transformation products were used to elucidate the reaction mechanisms. Leaching tests on treated AERs were conducted, confirming that PFAS release is not a concern if the milled resins are exposed to rainfall or snow melt.

Materials and Method.

Chemicals and AERs

All chemicals were used as received: Heptadecafluorooctanesulfonic acid potassium salt (PFOS, Sigma-Aldrich), boron nitride (BN, Sigma-Aldrich), sodium chloride (NaCl, Sigma-Aldrich), methanol (MeOH, Sigma-Aldrich), PFA694E single-use AERs (Purofine), and AmberLite IRA67 regenerable AERs (DuPont). The desired amounts of PFOS were loaded on AERs through adsorption (details see [Text S1](#)) to prepare model AERs for treatment and mechanism study. Two types of PFAS-containing spent single-use AERs, PFA694 and AmberLite PSR2 Plus, derived from PFAS site remediation projects, were provided by WSP and Haley & Aldrich, respectively.

Ball Milling Treatment

AERs (0.28 g) and BN (0.5–1 g) were added into a 100 mL stainless steel (SS) jar containing 160 g of SS balls (fifteen 10 mm balls and one hundred and ten 5 mm balls). The jar was sealed with a lid and placed on a planetary ball mill (Across International, PQ-N04), which was then rotated at a jar speed of 580 rpm.

Extraction and Analysis of PFAS

The PFOS loads on the model AERs, both before and after the ball milling treatment, were verified through solvent extraction. The selection and optimization of the extraction solvent are detailed in a subsequent section. For the AERs without subjecting to ball milling, solvent extraction of AER beads was conducted in a centrifuge tube mounted on a shaker rotating at 60 rpm for 24 hours. For AERs subjected to the ball milling treatment, the solvent was added directly to the ball milling jars

to extract residual PFAS from the AERs, milling balls, and jar walls. The jars were then subjected to the same 24-hour extraction process. The extraction of the PFAS mixture from the field-collected spent AERs follows the same protocol. PFAS analysis was conducted by ultra-high-performance liquid chromatography (UHPLC, Thermo Vanquish) integrated with a triple quadrupole mass spectrometer (MS/MS, Thermo Altis). Suspect screening of transformation products was performed using high-performance liquid chromatography coupled with quadrupole time-of-flight mass spectrometry (HPLC-QToF-MS, SCIEX). Details of instrument setup and data acquisition can be found in [Text S2](#) and [S3](#). Procedures for the extraction of F⁻ by 0.1 M NaOH and analysis by ion chromatography (IC; Dionex) were described in [Text S4](#).

Results and Discussion

Extraction of PFAS and F⁻

It is important to emphasize that the ball milling treatment is a non-solvent process, where dry AERs were co-milled with BN without the use of solvents. However, to assess the efficiency of PFAS destruction, samples were extracted with solvents both before and after treatment, and the PFAS content in the extract was analyzed. For regenerable AER, the extraction process aligns with the principles of regeneration, using solvents to desorb PFAS.¹⁶ Extracting PFAS from single-use AERs poses a greater challenge due to its high affinity for long-chain PFAS, but it is still achievable.¹⁵

A combination of water, methanol, and NaCl has proven to be the most effective extraction solvent.^{16–19} Based on this, we tested nine different solvents following a previously reported method for extracting PFOS from PFA694E single-use AERs preloaded with a known quantity of PFOS ([Figure S1a](#)). The results show that the mixture of 80% methanol and 3% NaCl was the most effective, recovering 80% of the spiked PFOS. Milling AERs with SS balls without BN for one hour did not result in PFOS destruction but did increase the amount of PFOS extractable from the pulverized AERs to 97% ([Figure S1a](#)). This increase is likely because the milling process broke down the porous structure of the AERs, allowing improved contact between the AERs and solvent, which facilitated the extraction of PFAS. This ‘pre-milling + extraction’ strategy was also used to measure the PFOS sorbed on IRA 67 AERs ([Figure S1b](#)) and the PFAS mixture sorbed on the field-collected resin ([Figure S2](#)) in subsequent experiments.

Fluoride, as the degradation end product, was extracted using either 6% NaCl or 0.1 M NaOH, based on recommendations from previous studies focused on regenerating AERs after F⁻ adsorption.^{20,21} The results of extraction of a known amount of F⁻ spiked on AERs are shown in [Figure S1c](#). 0.1 M NaOH demonstrated superior performance over 6% NaCl, achieving a 90% F⁻ extraction efficiency. These results also suggest that if PFAS are fully mineralized to F⁻, which is subsequently immobilized by AERs, approximately 90% should be found in the extract solution. The limitation in F⁻ recovery prevents the fluorine balance from being fully closed (see discussion below). However, it is important to note that achieving 100% F⁻ recovery from AERs is rarely possible, as has been shown previously.^{20,21} Because the F⁻ extraction efficiency is controlled by the concentration equilibrium between F⁻ and competing ions. Additionally, AERs have a higher affinity to F⁻ than to Cl⁻ and OH⁻ in the extract solvent.²²

Treatment of Model AERs Spiked with PFOS

AERs spiked in the lab with PFAS were pulverized by milling with stainless steel (SS) balls without BN for one hour and extracted to yield time-zero PFOS concentrations ([Figure 1](#)). Control tests were conducted by further milling the pulverized AERs for an additional eight hours in the absence of BN. The key finding from these tests is that ball milling alone, without the aid of BN, could not destroy PFOS.

To initiate PFAS destruction, BN powders were added following one hour of pulverization. In the presence of BN, upon ball collision, the piezoelectric BN can generate kV-level potentials for the destruction of PFAS chemicals, as detailed extensively in our recent studies.^{23,24} Measurable destruction of PFOS adsorbed on AERs was observed after the addition of BN, with degradation kinetics accelerating with the increased BN loading on PFA694E AERs ([Figure 1a](#)). Co-milling of 750 mg BN with 280 mg AERs (BN/AER mass ratio of 2.7) resulted in near-complete destruction of PFOS after three hours of treatment. The faster PFOS degradation kinetics at higher BN doses can be attributed to the enhanced contact between BN and the AERs. This is further supported by scanning electron microscope (SEM) imaging, which shows a well-mixed homogenized mixture of BN and AER debris after milling ([Figure S3](#)).

The treatment of PFOS on AERs was compared with the destruction of PFOS in solid form at equivalent amounts to determine the role of the resin in PFOS destruction ([Figure 1b](#)). As expected,

the PFOS embedded in AERs exhibited slower degradation kinetics, suggesting that the AERs act as an inert matrix and shield the impact force exerted on BN, thereby attenuating the piezoelectric potential. The impact force can be increased by an increase of ball to powder mass ratio (B/P ratio, where the powder includes BN particles and AERs). As a result, the PFOS degradation kinetics are accelerated at a higher B/P ratio (Figure S4). By optimizing the BN-to-AER ratio, near-complete destruction of PFOS on regenerable IRA 67 AERs was achieved (Figure 1c).

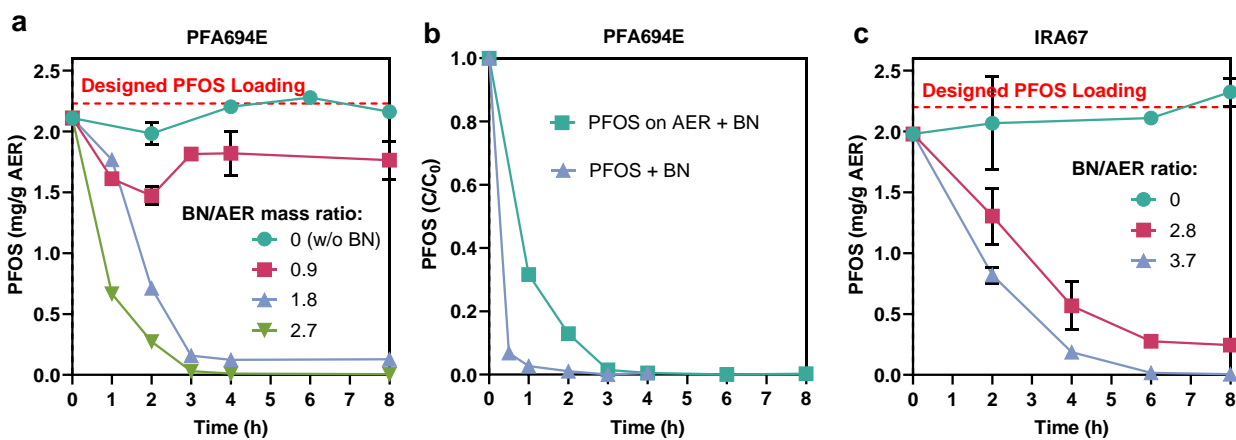


Figure 1. BN-assisted ball milling treatment of PFOS adsorbed on model AERs. (a) PFOS degradation on single-use PFA694E AERs. (b) Comparative analysis of PFAS degradation kinetics when present in powder form versus adsorbed on PFA694E AERs. (c) PFOS degradation on regenerable IRA67 AERs. PFA694E (280 mg) or IRA67 (268 mg) was milled with BN powders at mass ratios specified in the figure legend, using a jar rotation speed of 580 rpm. For Figure 1b, two conditions were tested: (i) 280 mg PFA694E loaded with 0.6 mg PFOS was milled with 750 mg BN, and (ii) 0.6 mg PFOS (added as a methanol stock solution and evaporated methanol at room temperature) was milled with 750 mg BN. Error bars represent the standard deviation of triplicate results.

During the ball milling treatment, the formation of shorter-chain perfluorinated carboxylate acids (PFCAs) was observed both when PFOS was adsorbed onto the AERs and when it was treated as a standalone solid (Figure S5). The formation of PFCAs indicates a $-\text{CF}_2-$ elimination mechanism initiated by the cleavage of the sulfonate group (Route 1 in reaction pathways

illustrated in Figure S7). This oxidative pathway has been widely reported in electrochemical oxidation processes.^{25,26} It aligns with the nature of BN-assisted ball milling, which relies on high piezoelectric potential to oxidize PFAS.²³

An HPLC-QToF-HRMS was used to identify suspect transformation products (TPs) not captured by the targeted UPLC-MS/MS analysis. As revealed in our recent study, ball collisions can impart boron nitride (BN) with both positive and negative charges, with the negative charge facilitating reductive destruction mechanisms.²³ It is also critical to note that H₂O, present in the feedstock and air, can be split into hydrogen atoms (•H) and hydroxyl radicals (•OH) under high potentials,^{27,28} such as piezoelectric potentials generated in our ball milling system. The generation of free electrons and the subsequent formation of hydrogen atoms (•H) leads to the formation of six 1H-perfluoroalkanes TPs (C_nF_{2n+1}H; n = 3-8; Figure S6). These TPs may originate from the cleavage of C-S or C-C bonds followed by the adduct of •H, as depicted in route 2 in Figure S7. The reductive destruction following the H/F exchange (route 3 in Figure S7) may also occur on the side-chain C-F bonds without breaking the sulfonate head groups. This potential reaction is supported by the detection of two H/F exchange TPs, C₈F₁₆HSO₃⁻ and C₈F₁₅H₂SO₃⁻ (Figure S6). The H/F exchange is suspected to take place at the beta-carbon position due to the lower C-F bond energy at this site.²⁹ The cleavage of the C-F side chain may also be followed by the attack of •OH, representing a combination of reduction and oxidation pathways (shown as route 4 in Figure S7). This process explains the formation of the OH/F exchange product (C₈F₁₆HSO₄⁻) and the associated ketonic structure (C₈F₁₅SO₄⁻) after HF elimination (Figure S6).

We found no evidence that the presence of AERs contributed to new reaction pathways or generated new TPs. For instance, we did not observe the formation of Cl⁻ or nitrogen-containing TPs, which could have resulted from the quaternary ammonium groups or the preloaded Cl⁻ in the strong-base AER.

Comparing the TP profiles observed during BN-assisted ball milling of PFOS loaded on AERs with those from solid PFOS treatment, we found that the presence of AERs produced the same TPs but in lower quantities (Figure S8). Although we could not quantify the TPs due to the lack of chemical standards, it is important to note that all TPs were eliminated with extended ball milling treatment. No peak intensities exceeding 100 were observed after 4 hours of treatment.

After 24 h of milling, 80% of the fluorine on PFOS loaded on the AERs was recovered as F⁻ (Figure S9). The gap in the fluorine balance may be attributed to the difficulty of completely extracting F⁻ from the AERs (Figure S1c).

Treatment of Field Exposed Spent AERs

We applied the BN-assisted ball milling approach in the treatment of field-exposed spent AERs. The milling treatment was performed at a BN-to-AER mass ratio of 2.7:1 (Figure 2). The AERs before and after treatment were extracted by a mixing solution of 80% MeOH and 3% NaCl for 24 hours (Figure S2), followed by the analysis of thirty target PFAS by UPLC-MS/MS. The extracts from the two AERs, sourced from different groundwater remediation projects, exhibited diverse PFAS profiles, covering C4-C8 PFSA and PFCAs. The BN-assisted ball milling treatment effectively eliminated all target PFAS below the detection limits (Table S1), irrespective of their chemical structures, after 24 h of milling.

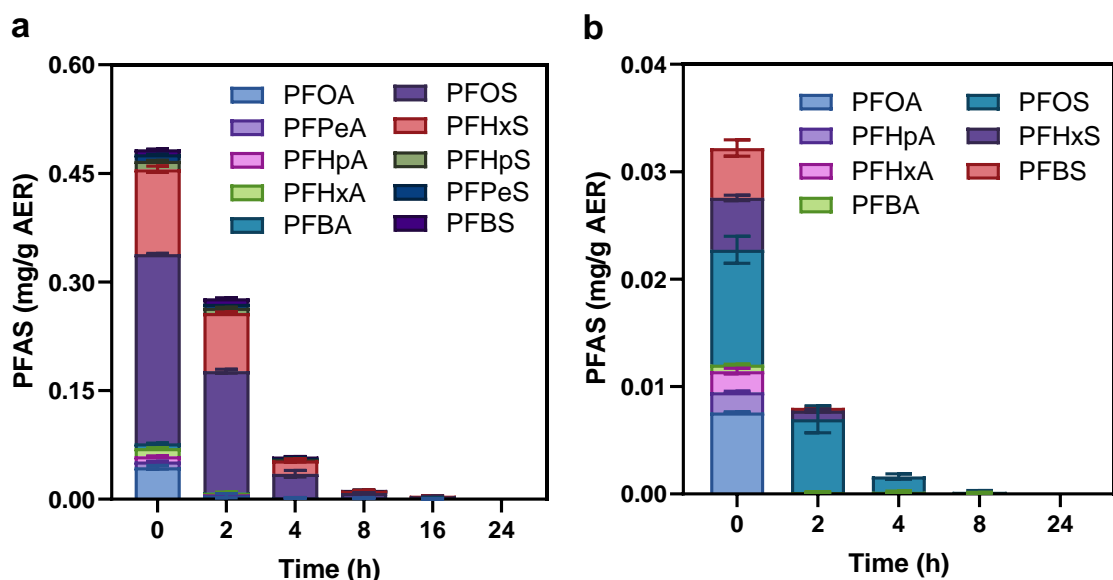


Figure 2. Destruction of PFAS on field-collected spent AERs. (a) Treatment of PFA 694. (b) Treatment of Amberlite PSR2 Plus. AERs (280 mg) were co-milled with 750 mg BN and 160 g SS balls at a jar rotation speed of 580 rpm. Error bars represent the standard deviation of triplicate results.

To address the concern about the leaching of PFAS residuals from AERs after ball milling, we performed leaching tests following EPA method SW-846 1312 (method description in [Text S5](#)). The treated AERs (without extraction by organic solvents) were immersed in pH 4.2 deionized water to simulate acid rainfall or snow melt. The water-to-solid mass ratio was 20:1 in compliance with the standard method. After 20 h of leaching tests, thirty target PFAS were all found below the detection limits in the leaching solution ([Table S2](#)).

Environmental Implications

This study presents a novel, non-thermal, solvent-free approach for treating PFAS in AERs. The method involves a simple setup that co-mills AERs with BN and SS balls. BN is a widely available industrial material. Given the economic considerations, we do not expect the recovery and reuse of low-cost BN (\$30-50/kg from Alibaba.com) to be beneficial. Therefore, the BN will be blended with the treated AERs after treatment. During the PFAS degradation process, BN is expected to decompose into ammonium and borate.²³ Even if residual BN particles remain, they are known to be biocompatible and non-cytotoxic.³⁰

Ball mills, as mature industrial equipment, can be repurposed as effective treatment units. However, a significant gap exists in developing models and methodologies to translate lab-scale results obtained from planetary ball mills to larger-scale mills, such as tumbler mills. This challenge is currently under investigation. Nonetheless, our previous theoretical analysis indicates that the ball impact velocity in planetary ball milling, measured at 1.5 m/s,²³ is in the same order of magnitude as the value generated by tumbler mills.³¹ Additionally, a peer study has demonstrated the feasibility of scaling up PFAS-contaminated soil treatment using a tumbler mill, though their approach differs by employing KOH as the co-milling reagent.³² Therefore, scaling up the treatment of AERs using BN-assisted ball milling presents a promising avenue approach.

Lastly, it is important to emphasize that this technology is specifically designed for the disposal of single-use and end-of-life regenerable AERs, not for regeneration. The ball mill treatment unit can be constructed as a modular, trailer-based platform. This onsite, non-thermal PFAS elimination process allows the PFAS-free milled AERs to be further disposed of as non-hazardous material through landfilling, ensuring regulatory compliance.

Supporting Information

The Supporting Information is available free of charge at [link]

Description of methods, extraction efficiencies of PFAS, analyte list, SEM images, transformation product analysis, and defluorination efficiencies.

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