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Research article

Fate of per- and polyfluoroalkyl substances (PFAS) in anaerobic digestion of tannery sludge and their removal from clarified digestate by adsorption

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ABSTRACT

Anaerobic digestion (AD) is a well-established technology for sludge stabilization and energy recovery; however, its application to PFAS-contaminated industrial sludge remains poorly understood. In this study, tannery sludge was treated under mesophilic (37 °C) and thermophilic (55 °C) conditions in continuous stirred tank reactors, to evaluate process performance and assess the fate of per- and polyfluoroalkyl substances (PFAS). Subsequently, adsorption was investigated as a post-treatment for PFAS removal from the clarified digestate (CD) using granular activated carbon (GAC) and a strong-base anion exchange resin (AER). Both reactors achieved stable operation, with thermophilic conditions resulting in higher specific gas production (0.62 vs 0.49 m^3/kg VS) and volatile solids removal (54% vs 42%) compared with mesophilic digestion. However, PFAS concentrations in the CD remained above 10,000 ng/L; no significant differences were observed between mesophilic and thermophilic conditions, suggesting that AD thermal regime had a limited influence on PFAS occurrence in the liquid fraction.

Adsorption experiments showed that AER exhibited a higher adsorption capacity than GAC for both total PFAS and individual congeners. The adsorbent dosages required to achieve target PFAS concentrations (500 and 100 ng/L) were estimated, and a preliminary full-scale assessment highlighted substantial differences in adsorbent inventory requirements and infrastructure footprint between the two technologies. Overall, the results demonstrate that, while AD is effective for energy recovery from tannery sludge, it does not mitigate PFAS contamination and therefore requires dedicated post-treatment. These findings provide new insights into the integrated management of PFAS-contaminated industrial sludge, supporting the development of scalable treatment strategies.

1. Introduction

The management of industrial sludge from the tanning industry represents a significant environmental challenge due to its complex composition and potential environmental impact. Tannery sludge is typically characterized by high organic content and the presence of heavy metals and recalcitrant organic compounds, which limit conventional treatment and disposal options (Bresolin et al., 2025). In recent years, increasing attention has been devoted to the occurrence of emerging contaminants in these waste streams, particularly per- and polyfluoroalkyl substances (PFAS).

PFAS comprise a class of highly persistent synthetic chemicals widely used for their hydrophobic and oleophobic properties in numerous industrial applications, including leather finishing processes (Glüge et al., 2020). As a result, tannery sludge can act as a relevant reservoir of these compounds because of their exceptional resistance to environmental degradation. In addition to their persistence, PFAS have been associated with adverse effects on human health and the environment and are therefore subject to increasingly stringent regulatory controls, particularly with respect to water discharge limits (Cousins et al., 2020). Consequently, the presence of PFAS in tannery sludge raises critical concerns regarding its safe treatment, disposal and overall management.

Anaerobic digestion (AD) is a well-established technology for the stabilization of sludge and other organic waste streams, as well as for energy recovery. By reducing the mass of the treated waste while simultaneously producing biogas, AD represent an attractive treatment option, particularly when sludge is used as co-substrate (Li et al., 2024). However, the application of AD to tannery sludge has been only marginally explored, mainly due to concerns that heavy metals, such as

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Abbreviations

(AD) Anaerobic digestion
(AER) anion-exchange resins
(BOD) biochemical oxygen demand
(CAPEX) capital expenditures
(CD) clarified digestate
(CSTR) continuous stirred tank reactor
(EBCT) Empty Bed Contact Time
(GPR) gas production rate
(GAC) granular activated carbon
(HRT) hydraulic retention time
(OPEX) operational expenditures
(OLR) organic loading rate
(PFAS) per- and polyfluoroalkyl substances
(SRT) sludge retention time
(SGP) specific gas production
(TKN) total Kjeldahl nitrogen
(TS) total solids
(VFA) volatile fatty acids
(VS) volatile solids
(WWTP) wastewater treatment plant

chromium, and other persistent contaminants may inhibit methanogenic bacteria (Mpofu et al., 2021). Furthermore, the behavior and fate of PFAS during AD (or in leachate matrices from landfills) remain poorly understood, and the available literature reports limited and sometimes contradictory findings (Gallen et al., 2017). In particular, to the best of the authors' knowledge, comparative studies evaluating the treatment of PFAS-contaminated tannery sludge under mesophilic and thermophilic conditions have not yet been reported.

Given the persistence of PFAS, additional treatment steps are often required to ensure compliance with discharge standards. Among the available technologies, adsorption onto granular activated carbon (GAC) and anion-exchange resins (AER) is widely recognized as one of the most effective approaches for PFAS removal from aqueous matrices (Rahman et al., 2014; Meng et al., 2019). However, most studies have focused on relatively clean water systems or laboratory-scale artificially contaminated waters (Senofonte et al., 2026), whereas investigations involving complex matrices, such as anaerobic digestates, remain limited.

In this context, the present study addresses this knowledge gap by investigating the AD of tannery sludge under both mesophilic and thermophilic conditions, evaluating process performance and assessing PFAS occurrence in the resulting clarified digestate (CD). Subsequently, an adsorption-based post-treatment was applied using GAC and AER to remove PFAS from the digestate. The adsorption process was modeled to describe equilibrium behavior and support process design. Based on the modeling outcomes, the adsorbent dosage required to achieve target PFAS concentrations in the treated effluent were estimated, considering regulatory thresholds of 100 and 500 ng/L. Furthermore, a preliminary design of the adsorption system was developed by estimating the minimum number of adsorption columns required as a function of the digestate flow rate.

This work provides new insights into the integrated treatment of PFAS-contaminated tannery sludge through the combination of AD and adsorption processes and contributes to the development of scalable treatment strategies capable of meeting increasingly stringent environmental regulations.

2. Materials and methods

2.1. Tannery sludge collection and features

The tannery sludge used in this study was collected from the wastewater treatment plant (WWTP) of Montebello Vicentino (northeast Italy), which treats more than 10,000 m^3/d of tannery wastewaters generated by local industries. These wastewater undergoes a physico-chemical primary treatment followed by sedimentation to remove a portion of the chromium and settleable organic matter. The resulting primary effluent is then treated in a secondary biological process (anoxic/aerobic) for nitrogen and biochemical oxygen demand (BOD) removal. The final sludge consists of approximately 60% primary sludge and 40% secondary biological sludge (v/v). Due to the high content of proteins and other macromolecular organic compounds, as well as the relatively high salinity of the tannery wastewaters, the WWTP is operated at a sludge retention time (SRT) exceeding 30 days, which is substantially longer than the SRT typically adopted in municipal WWTPs. After its collection, the sludge is subjected to a drying process prior to landfilling.

In terms of physical-chemical parameters, the tannery sludge showed the following characteristics: 830 ± 14 g TS/kg (total solids), 590 ± 4 g VS/kg (volatile solids), 793 ± 18 g COD/kg TS as chemical oxygen demand, 32.8 ± 0.9 g N/kg TS and 7.9 ± 0.4 g P/kg TS as total Kjeldahl nitrogen (TKN) and phosphorus content.

2.2. Mesophilic and thermophilic anaerobic digestion

Prior to use, the tannery sludge was diluted with tap water to a solids content of 7% (w/v), simulating the sludge stream exiting a dynamic thickener in a typical WWTP sludge line. The diluted sludge was subsequently subjected to a mild thermal pretreatment (70 °C, 24 h) to enhance the bioavailability of the organic matter. This protocol was adapted from Tuci et al. (2024). Two parallel plexiglass continuous stirred tank reactors (CSTRs; Idea Bioprocess Technology S.r.l.) were used for the anaerobic treatment of tannery sludge. Both reactors were inoculated with a methanogenic consortium obtained from the digestate of a full-scale anaerobic digestion plant located in Treviso (northeast Italy) (Micolucci et al., 2014). Each reactor had a working volume of 4.0 L and was equipped with a thermostatic jacket for temperature control and a mechanical stirrer. The reactors were manually fed the diluted tannery sludge and discharged once daily. A volume of 200 mL was withdrawn from each reactor according to the chosen hydraulic retention time (HRT, 20 d). The temperature was set at 37 °C in the mesophilic CSTR (CSTR37) and at 55 °C in the thermophilic CSTR (CSTR55). The effluents were periodically analyzed for soluble COD (COD_{SOL}), volatile fatty acids (VFA), ammonia, partial alkalinity, and pH. The OLR was equal to 2.5 kg VS/(m^3 d) in both reactors, under the stability period.

2.3. Digestate clarification and PFAS adsorption tests

Digestate samples from both mesophilic and thermophilic reactors were collected and subjected to solids removal trough an assisted physical-chemical settling process. An $FeCl_3$ solution was added to adjust the digestate pH to 4.5 (approximately 4.5 L per m^3 of digestate). Subsequently, a commercial polyelectrolyte solution was dosed to promote solids coagulation (approximately 20 L of a 0.1% w/v polyelectrolyte solution per m^3 of digestate). The suspension was then allowed to settle for 24 h, yielding the final clarified digestate (CD), which was first analyzed for PFAS quantification and then used in the adsorption tests. The PFAS removal was investigated through batch adsorption tests using two commercially available adsorbent materials,

representative of the main adsorbent classes employed for PFAS removal: a granular activated carbon (GAC; DARCO®, Sigma-Aldrich/Merck, 8–20 mesh) and a strong-base anion exchange resin (AER; C.T. S. S.r.l., Italy). The AER consisted of a styrene-divinylbenzene (DVB) copolymer matrix functionalized with quaternary amine groups, supplied in the OH^- ionic form, with an exchange capacity of 3.9 eq/L and particle size of 30–200 μm. GAC and AER were selected to compare two distinct adsorption mechanisms involved in PFAS removal: hydrophobic adsorption onto activated carbon and ion-exchange/electrostatic interactions onto anion exchange resin.

Both adsorbents were tested at four dosage levels (0.25, 0.50, 1.0 and 2.0 g/L) using 0.5 L of CD. Each adsorption batch tests were carried out in duplicate, at 20 °C under a fixed stirring rate (Nautilus machine, Anaero Technology) for 96 h. This contact time was selected as a conservative condition to ensure the attainment of equilibrium, or near-equilibrium conditions, prior to isotherm modeling. Preliminary contact-time observations showed that PFAS residual concentrations measured after 24 h were comparable to those measured after 96 h for all detected congeners, indicating that adsorption had effectively reached equilibrium within 24 h under the tested conditions. The Langmuir and Freundlich models were applied to describe PFAS adsorption from the CD and to estimate the adsorbent dosage required to achieve residual PFAS concentrations of 500 ng/L and 100 ng/L. These target values correspond to the thresholds established by the former Drinking Water Directive 98/83/EC for total PFAS (500 ng/L, where an appropriate analytical method is available) and for the sum of 20 regulated PFAS (100 ng/L). Both limits were subsequently confirmed and incorporated into the current Drinking Water Directive (EU, 2020).

2.4. Sampling and analytical methods

Reactor effluents were monitored approximately twice per week for TS, VS, COD, TKN and P. Similarly, process stability parameters, including pH, volatile fatty acid (VFA), partial alkalinity and ammonia, were measured in duplicate two/three times per week. All analyses were performed according to Standard Methods (Apha, 2005). VFA concentrations were determined using an Agilent 6890N gas chromatograph equipped with a flame ionization detector (100 °C) and a fused silica capillary DB-FFAP column (15 m length, 0.53 mm × 0.5 mm film thickness). Analyses were conducted on filtered samples (cellulose acetate filter of 0.20 μm porosity) following centrifugation (4000 rpm; 5 min). Hydrogen was used as the carrier, and the oven temperature was increased from 80 °C to 200 °C at a rate of 10 °C/min. Biogas production was monitored with an internal programmable logic controller (Idea bioprocess technology srls, Italy) which provided data on daily biogas flow rate (Q_{biogas}); three measurements were usually taken to quantify the daily gas production rate (GPR). The methane content in the biogas was quantified using a gas chromatograph 6890N (Agilent TechnologyTM) equipped with an HP-PLOT MOLESIEVE column (30 m length, 0.53 mm ID x 25 mm film thickness) maintained at 80 °C for 8 min, by using a thermal conductivity detector (250 °C) and argon as carrier.

PFAS analyses on the CD were performed twice weekly by an external accredited laboratory according to ASTM D7979-20, using liquid chromatography coupled with mass spectrometry. This method is designed for the determination of selected PFAS in water, sludge, influent, effluent, and wastewater matrices by liquid chromatography coupled with tandem mass spectrometry. Additional details regarding sample handling, quantification procedures, and quality assurance/quality control (QA/QC) protocol are provided in the Supplementary m aterial.

The complete list of PFAS compounds analyzed in this study is reported in Table 1. The table includes the 20 PFAS regulated under the former Drinking Water Directive 98/83/EC, whose thresholds were later confirmed and incorporated into the current Drinking Water Directive (EU) 2020/2184, together with additional PFAS and precursors commonly included in advanced analytical workflows.

Table 1
PFAS compounds analyzed in the CD.

Compound	CAS	Compound	CAS
PFOS	1763-23-1	PF-3,7-DMOA	172,155-07-6
PFOA	335-67-1	4:2 FTS	757,124-72-4
PFBA	375-22-4	6:2 FTS	27,619-97-2
PFBS	375-73-5	8:2 FTS	39,108-34-4
PFPeA	2706-90-3	4:2 FTOH	2043-47-2
PFHxA	307-24-4	6:2 FTOH	647-42-7
PFHxS	355-46-4	8:2 FTOH	678-39-7
PFHpA	375-85-9	PFOSA	754-91-6
PFNA	375-95-1	N-MeFOSA	31,506-32-8
PFDeA	335-76-2	N-EtFOSA	4151-50-2
PFUnA	2058-94-8	N-MeFOSE	24,448-09-7
PFDoA	307-55-1	N-EtFOSE	1691-99-2
PFHpS	375-92-8	Me-FOSAA	2355-31-9
HFPO – DA (GenX)	13,252-13-6	Et-FOSAA	2991-50-6

2.5. Calculation

The performance indicators of the AD process were defined based on biogas production and VS removal. The specific gas production (SGP; Nm^3/kg VS) was calculated as a function of the biogas flow rate (Q_{biogas}), the OLR and reactor volume (V):

$$SGP = \frac{Qbiogas}{OLR \cdot V}$$

The GPR (Nm^3/m^3 d) was calculated according to the biogas flow rate (Q_{biogas}) and reactor volume (V):

$$GPR = \frac{Qbiogas}{V}$$

For the two CSTRs, the steady state was considered achieved when the GPR was constant enough (below 10% of deviation from average values).

The VS removal efficiency (η%) was defined as the degree of organic matter conversion into biogas and indicated the percentage of removed VS; the equation was defined according to the flow rate (Q; inlet and outlet flow rates were the same), VS_{IN} and VS_{OUT} as the VS concentration in and out of the CSTR:

$$\eta(\%) = \frac{(Q \cdot VS \in) - (Q \cdot VSOUT)}{(Q \cdot VS \in)}$$

For the investigation of the PFAS adsorption on CD, the Langmuir equation was chosen to describe the isotherm:

$$q_e = q_m \frac{K_L \cdot C_e}{1 + K_L \cdot C_e}$$

where "q_e" indicates the mass of adsorbate per unit mass of adsorbent material at equilibrium, "q_m" indicates the maximum adsorption capacity under the Langmuir assumptions, "K_L" is the Langmuir constant and "C_e" is the concentration of the species of interest in the solution at equilibrium. The related model was fitted using its linear form:

$$\frac{C_e}{q_e} = \frac{1}{q_m \cdot K_L} + \frac{C_e}{q_m}$$

To further evaluate the adequacy of the selected adsorption model, the experimental data were also fitted using the Freundlich isotherm, which is commonly applied to describe adsorption on heterogeneous surfaces and complex matrices. This model was fitted using the following equation:

$$\log(q_e) = \log(K_F) + \frac{1}{n} \cdot \log(C_e)$$

where K_F and n are the Freundlich constants related to adsorption ca-

pacity and adsorption intensity, respectively. The resulting fitting parameters and goodness-of-fit indicators are reported in the Supplementary material.

Langmuir model was retained for the process-oriented estimation of adsorbent requirements because it provides a finite maximum adsorption capacity (q_m), enabling a direct estimation of the required adsorbent inventory. The Freundlich model was employed as a complementary model to account for possible adsorption heterogeneity in the CD matrix.

Once the Langmuir coefficients had been determined and the model reliability had been assessed, the model was used to estimate the theoretical adsorbent dosage required to achieve PFAS concentrations of 500 ng/l and 100 ng/l in the liquid matrix, as previously specified. For this purpose, the initial PFAS concentration in the CD was considered, and the following mass balance for a batch reactor at equilibrium was used:

$$q_e \cdot m = V \cdot C_0 - V \cdot C_e$$

Where "m" indicates the mass of the adsorbent.

To design a full-scale continuous fixed-bed system for PFAS adsorption, both flow rate (Q) and Empty Bed Contact Time (EBCT) have been considered; the required volume (V) is calculated according to the formula:

$$V = EBCT \cdot Q$$

For statistics, the t - tests were conducted to detect the significance of the results. Shapiro–Wilk test was performed to check whether the data were normally distributed. F - test was used to verify the homoscedasticity between the different groups of data. The null hypothesis was rejected when the calculated p-values were equal to or less than the level of significance (α). Statistical analyses were performed using the open-source program, R (The R Foundation for Statistical Computing, version 4.2.2).

3. Results and discussions

3.1. Process management in the start-up and in the steady state phases

Both mesophilic and thermophilic reactors followed the same operational strategy in terms of OLR and HRT. In the first two weeks, no feeding was applied in order to allow the inoculum to acclimate to the new substrate. At the end of this period, both CSTR37 and CSTR55 were started at an OLR of 0.5 kg VS/(m^3 d), corresponding to a relatively long HRT (approximately 50 days). This low OLR was maintained for nearly one month to prevent the onset of fermentation phenomena, which are often promoted by a sudden increase in the organic load within the reactor (Valentino et al., 2019). Subsequently, the OLR was gradually increased to progressively enhance the activity of the methanogenic consortium, ultimately reaching the target value of approximately 2.5 kg VS/(m^3 d). This conservative start-up strategy is generally recommended for the anaerobic digestion of a wide range of substrates and is particularly suitable for wastes rich in readily biodegradable organic matter (Yan et al., 2023). Although AD represents an environmentally sustainable option for tannery sludge management, enabling both biogas recovery and sludge mass reduction, remarkable attention to its application for this specific waste stream has only emerged in recent years (Bresolin et al., 2025). According to the available literature, which mainly focuses on mesophilic mono-digestion of primary and secondary tannery sludges, either separately or in combination, a cautious operational approach was adopted in this study. Regarding HRT, values higher than the target operating condition were maintained in the initial stages of operation, in both CSTR37 and CSTR55, to favor biomass retention and minimize the risk of the methanogenic biomass wash-out. Starting from the 8th week onward, both reactors were operated at the target HRT of 20 days.

3.2. Stability parameters and process performances

Fig. 1A-B-C shows the pH, VFA/Alkalinity ratio and the GPR observed in CSTR37 and CSTR55, according to the average values recorded on each sampling day. It is well known from the literature that the methanogenic consortia require an optimal pH between 7.5 and 8.5 to ensure a stable activity (Tsegaye and Leta, 2022). In the first two weeks, pH exhibited some variability but remained within the stability range of 7.5 – 8.5 (Fig. 1A). As the OLR increased from 0.5 to 2.5 kg VS/(m^3 d), both reactors showed a slight decrease in pH, likely due to a temporary accumulation of VFA. However, once the OLR was maintained at a constant level, pH stabilized within the optimal range (around 7.6 and 8.4 in CSTR37 and CSTR55 respectively).

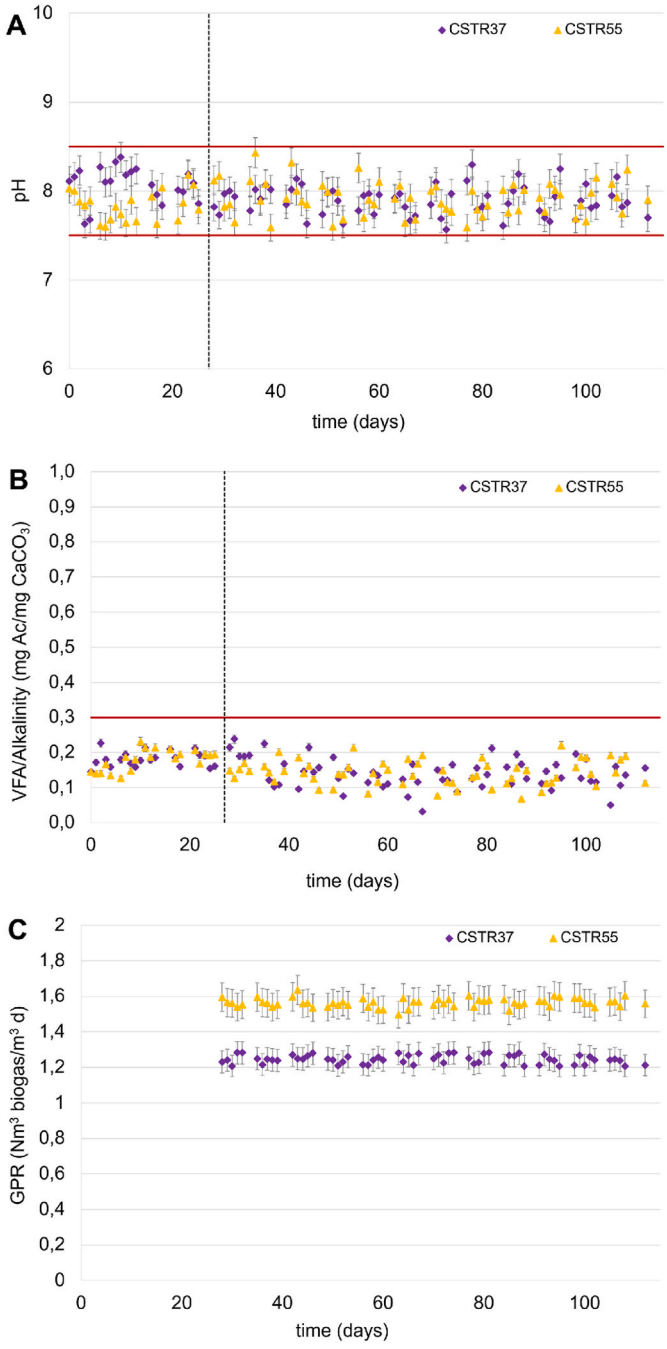

Fig. 1. Trend of pH (A), VFA/Alkalinity ratio (B) and GPR (C) quantified in mesophilic and thermophilic reactors. Error bars represent standard deviations. The vertical line indicates the start of the stability phase of the process.

Regarding partial alkalinity, this parameter was closely related to the adopted OLR under both thermal regimes, increasing as a function of the amount of sludge fed per day. Despite of the presence of lime, which could act as buffering agent (Tuci et al., 2024), it was necessary to gradually increase the OLR to avoid any proliferation of fermentative bacteria (over methanogenic consortia) as a consequence of excessive VFA accumulation. In the start-up phase of both CSTR37 and CSTR55, partial alkalinity progressively increased with increasing OLR, mainly due to the higher biogas production and, in turn, to the CO_2 dissolution in form of HCO_3^- (Du et al., 2025). Once the stability phases were reached, the distributions of stability parameters (pH, VFA, partial and total alkalinity, biogas and its composition production) in both thermal regimes were found to be characterized by a normal distribution (Shapiro – Wilk test, p value > 0.05), demonstrating the presence of the steady states (from the 28th day of operation). Regarding the VFA concentrations, the highest and most variable values were detected in the first weeks of operation in both CSTR37 and CSTR55. This behavior was likely associated with the relatively low alkalinity (850 – 1000 mg $CaCO_3$/L) and the incomplete acclimation of the methanogenic biomass, which was not yet able to efficiently utilize the VFA produced by fermentative bacteria. The ratio between VFA concentration and partial alkalinity is widely recognized as a key indicator of AD stability. The trend of this index is reported in Fig. 1B and it is almost overlapped to the VFA trend (data not shown). In the first two weeks of operation, the VFA/alkalinity ratio exhibited relatively high values, exceeding the threshold of 0.3 mg acetate/mg $CaCO_3$, commonly considered indicative of stable AD process (Elazhar et al., 2022). Throughout the whole experimental period, both CSTR37 and CSTR55 showed VFA/alkalinity values always below the aforementioned threshold.

3.2.1. Biogas production and composition

Biogas production was evaluated from the 28th day onward, as shown in Fig. 1C, which reports the GPR trend until the end of both experimental runs. Given the quantified initial values, both CSTR37 and CSTR55 were characterized by a GPR increasing trend in the start-up phase (as expected) with a stable values reached from the 14th week. On average, the GPR values were equal to 1.24 and 1.56 $Nm^3/(m^3\ d)$ for CSTR37 and CSTR55 respectively. These results are aligned and in the same order of magnitude as those reported for highly biodegradable substrates, such as the mixture of sewage sludge and organic fraction of municipal solid waste (1.38-1.79 $Nm^3/(m^3\ d)$; Valentino et al., 2019), and food waste (>2.0 $Nm^3/(m^3\ d)$; Micolucci et al., 2014). The biogas composition was also monitored starting from the 28th day onward, when both reactors had nearly reached the stability periods. Unlike the GPR, no significant variations in biogas composition were observed throughout the experimental period. The biogas produced in CSTR37 was characterized by an average CH_4 and CO_2 content equal to 56.2% (±1.4%) and 43.8% (±1.4%) respectively. Similarly, the biogas generated in CSTR55 had the following composition: 56.7% (±1.2%) and 43.3% (±1.2%) for CH_4 and CO_2 respectively. These methane contents are comparable to those reported for other commonly digested organic substrates (Ayantokun et al., 2025). Moreover, the relatively high methane fraction suggests that the protein-rich and biodegradable organic matter in tannery sludge was suitable for an effective conversion through all the AD steps, from hydrolysis to methanogenesis. Finally, the low variability in gas composition further confirms the stability of the process, demonstrating that tannery sludge is a suitable substrate for renewable energy generation in continuous anaerobic systems.

Consistent with the higher GPR observed in CSTR55 compared with CSTR37, the thermophilic reactor achieved a significantly higher SGP (0.62 m^3/kg VS) compared to mesophilic trial (0.49 m^3/kg VS) (T-test; p-value <0.05). The observed SGP values were within or exceed typical SGP ranges reported for other organic substrates. For example, the SGP in AD process of raw sewage sludges alone usually remains below 0.30 m^3/kg VS (Szelag-Sikora et al., 2025); improvements are usually observed when municipal sludge is mixed with other organic waste fractions, reaching up to 0.80 m^3/kg VS as a result of improved substrate biodegradability (Azarmanesh et al., 2023).

Regarding the solid's abatement efficiency (η), thermophilic and mesophilic regimes caused a VS reduction of 54% and 42% respectively. Even though these performances were promising, as they substantially reduce the solids amount requiring final disposal, higher VS reduction can be obtained with highly putrescible substrate such as food waste, for which reductions of up to 80% have been reported, even under co-digestion conditions with municipal sludge (Arelli et al., 2021). When anaerobically digested as a single substrate, primary and secondary sewage sludge typically shows a VS reduction in the range 40-70% and 30-50%, respectively, depending on the operating temperature and process conditions (Picard et al., 2026). Therefore, the η values obtained here are comparable to those reported for conventional sludge digestion systems.

3.3. PFAS quantification in mesophilic and thermophilic clarified digestate (CD)

As reported in Fig. 2, the AD of tannery sludge produced a CD with PFAS concentration consistently exceeding 10.000 ng/L. PFAS analyses were conducted on CD samples collected twice weekly throughout the entire steady-state period for each thermal condition. No clear temporal trends were observed in either total PFAS concentration or congener distribution, with concentrations remaining consistently above 10,000 ng/L under both mesophilic and thermophilic operation. These results are consistent with previous studies reporting that conventional anaerobic biological processes are unable to effectively degrade PFAS

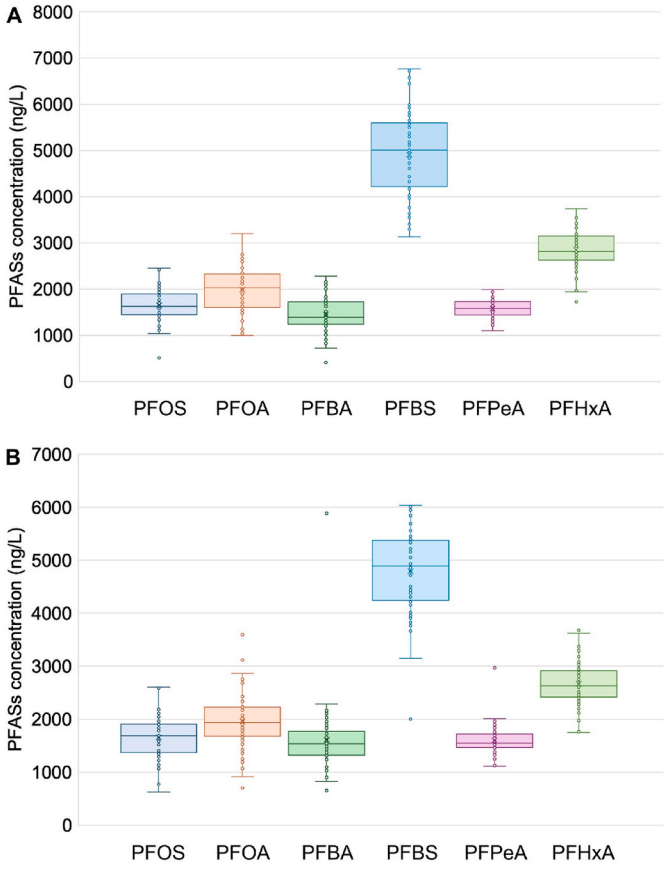

Fig. 2. Boxplot indicating the PFAS congeners in the mesophilic clarified digestate (A) and in the thermophilic clarified digestate (B). Samples were collected twice per week throughout the entire steady-state period for each condition. Boxes represent the median and interquartile range; "x" symbols indicate mean values, whiskers indicate variability, and points indicate outliers.

compounds. The degradation efficiency of PFAS by mixed microbial cultures is extremely low, and the biotransformation times required are not compatible with the typical retention time of AD systems (Niu et al., 2024). In this study, PFAS were consistently detected in the CD, with no significant differences observed between mesophilic and thermophilic operation (*t*-test, p-value >0.05). However, because PFAS concentrations were not measured in either the raw sludge or the solid fraction of the digestate, a complete solid–liquid mass balance could not be established. Consequently, the available data do not allow discrimination among potential mechanisms responsible for PFAS occurrence in the liquid phase, including limited biodegradation, redistribution between solid and liquid fractions, concentration effects associated with solids reduction, or the transformation of precursor compounds. Furthermore, a recent study demonstrated that measurable biodegradation of PFOA and PFOS generally requires reaction times substantially longer than those adopted in the present study, even when pure microbial cultures are employed (Huang and Jaffe, 2019). This suggests that significant PFAS degradation was unlikely under the 20-day HRT applied to the anaerobic mixed microbial consortia used in this work. Nevertheless, the similar PFAS concentrations and congener profiles observed in mesophilic and thermophilic CDs indicate that the thermal regime adopted during AD did not substantially influence PFAS occurrence in the liquid fraction.

The thermal regime adopted in the biological process did not result in any significant variation in PFAS congeners distribution. Among the 20 PFAS congeners analyzed, only six compounds were consistently detected above the method quantification limit (LOQ <5 ng/l) in all the samples, namely perfluorooctanesulfonic acid (PFOS), perfluorooctanoic acid (PFOA), perfluorobutanesulfonic acid (PFBS), perfluorobutanoic acid (PFBA), perfluoropentanoic acid (PFPeA), and perfluorohexanoic acid (PFHxA), in both mesophilic and thermophilic digestates. The targeted PFAS precursors included in the analytical method, such as fluorotelomer alcohols (FTOHs), fluorotelomer sulfonates (FTSs), and FOSA/FOSE/FOSAA-related compounds, were not detected above the quantification limit in any CD samples. However, because non-target screening was not performed, the presence of unidentified PFAS compounds or non-target PFAS precursors in the CD cannot be excluded. This aspect warrants further investigation through studies incorporating comprehensive precursor characterization and complete solid-liquid PFAS mass balance assessments.

Regarding the relative distribution of PFAS congeners in the two digestates analyzed, PFBS was the dominant compound, accounting on average for 35% and 33% of the total PFAS concentration in the mesophilic and thermophilic digestates, respectively. PFHxA was the second most abundant congener, representing approximately 20% of the total PFAS concentration in both digestates, followed by PFOA (14%), PFOS (11% and 13% in the mesophilic and thermophilic digestates, respectively), and finally PFBA and PFPeA, each contributing approximately 10% in both digestates.

3.4. *PFAS adsorption tests with thermophilic clarified digestate (CD) and practical implications*

The presence of PFAS in the digestate raises significant concerns regarding the use of anaerobic digestion as a treatment option for tannery sludge. This issue is particularly critical because the liquid fraction of the digestate, which may contain these contaminants, is typically directed to a biological treatment process at WWTP. To the best authors' knowledge, this is the first study reporting the occurrence of PFAS in digestate derived from tannery sludge anaerobic treatment. However, similar findings have already been reported for sewage sludge digestate (Deligiannis et al., 2024). Moreover, a recent review extensively discussed the challenges associated with PFAS recirculation through liquid streams, including digestate and internal recirculation flows, coupled with the limited effectiveness of conventional biological treatments for these contaminants (Liu et al., 2026). As highlighted in the review, conventional WWTPs are generally ineffective at removing PFAS compounds; as a result, the accumulation of PFAS in the treated effluent could lead to environmental contamination, affecting both surface and groundwater quality. This situation underscores the need for advanced treatment technologies capable of effectively removing these persistent pollutants.

To address this issue, the application of adsorption processes based on GAC and AER has been investigated for the removal of PFAS from the liquid fraction of the digestate. Fig. 3 shows the experimental equilibrium data and the corresponding Langmuir isotherms fitted to the adsorption results obtained from batch tests performed with the two tested adsorbent materials (AER and GAC). The tests were conducted using the CD sample characterized by the highest total PFAS concentration (16,515 ng/L; CSTR55). Four adsorbent dosages were evaluated for each material (0.25, 0.50, 1.0 and 2.0 g/L).

Before adsorption, the clarification step substantially modified the digestate matrix. COD decreased from 1540 to 543 mg/L and TOC from 531 to 200 mg/L, corresponding to reductions of approximately 65% and 62%, respectively. TKN decreased from 611 to 498 mg/L, whereas chloride concentration increased from 803 to 2420 mg/L, likely due to the $FeCl_3$-based acidification-coagulation process. Therefore, although clarification significantly reduced the organic load and suspended solids content, the CD used for adsorption tests still represented a complex matrix, characterized by residual organic matter, nitrogen compounds and high salinity. These constituents may affect PFAS adsorption by competing for adsorption sites, modifying electrostatic interactions, and promoting to fouling phenomena.

As shown in Fig. 3, the anionic resin exhibited a higher adsorption capacity for PFAS compounds compared to activated carbon. The q_m values were equal to 32,228 ng/g and 21,943 ng/g for AER and GAC respectively; accordingly, K_L values were equal to 0.0004 l/ng and 0.00005 l/ng for AER and GAC respectively.

The comparison with the Freundlich model showed that the Langmuir model provided an excellent fit for total PFAS adsorption onto AER ($R^2 = 0.99$), whereas the Freundlich model provided a better fit for total PFAS adsorption onto GAC ($R^2 = 0.97$ compared to $R^2 = 0.95$ for Langmuir) (Table S1 and Table S2, Supplementary material). These results suggest that PFAS adsorption onto GAC may be characterized by a greater degree of heterogeneity, likely due to the simultaneous contribution of congeners with different chain lengths and adsorption affinities. Nevertheless, the Langmuir model adequately described most congener-specific adsorption trends, particularly for GAC, and was therefore selected for the preliminary process design and sizing calculation. Also, Langmuir model provides a finite maximum adsorption capacity (q_m), which allows a direct estimation of adsorbent requirement. Freundlich model was used as a complementary approach to

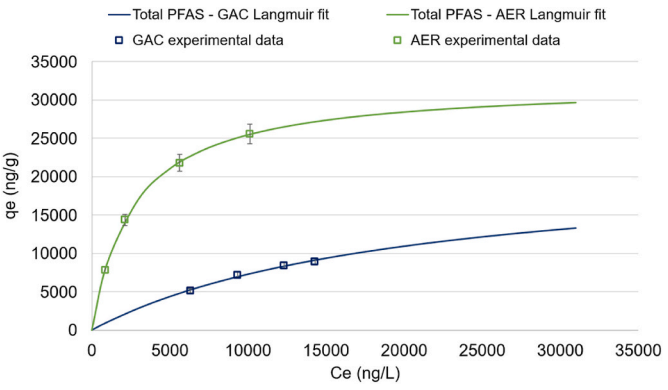

Fig. 3. Experimental equilibrium data points and fitted Langmuir isotherms for total PFAS adsorption using GAC and AER. Symbols indicate experimental equilibrium data obtained at four adsorbent dosages (0.25, 0.50, 1.0 and 2.0 g/L); solid lines indicate fitted Langmuir isotherms.

describe adsorption heterogeneity within the CD matrix. The complete set of Langmuir and Freundlich fitting parameters, together with the corresponding goodness-of-fit indicators, is reported in the Supplementary material (Tables S1 and S2).

Despite these differences in model fitting, the higher adsorption capacity observed for AER is consistent with its ion-exchange-based removal mechanism, which exhibits greater selectivity towards PFAS anions than the predominantly hydrophobic adsorption mechanism of activated carbon.

Most previous investigations have been conducted using either synthetic solution or real PFAS-contaminated groundwater; these studies have shown that anion exchange resins generally exhibit higher adsorption capacities for PFAS than GAC (up to 2400 mg PFOS/g and 525-1500 mg PFOA/g), as their ion-exchange mechanism promotes stronger electrostatic interactions with PFAS anions than the predominantly hydrophobic adsorption mechanism of activated carbon (Loganathan et al., 2024). Furthermore, long-term pilot-scale studies have confirmed the superior PFAS removal of anion-exchange resins compared with GAC under continuous operation (Chow et al., 2022), in agreement with the results observed in this study using CD from tannery sludge treatment.

With respect to the individual congeners, Fig. 4 shows the Langmuir adsorption isotherms obtained for the six detected compounds (PFOS, PFOA, PFBS, PFBA, PFHxA, and PFPeA). For readability, only the fitted Langmuir curves are shown, whereas the corresponding experimental equilibrium data are reported in the Supplementary material (Figure S1 and Figure S2). Consistent with the results for total PFAS, AER showed higher adsorption efficiency than GAC for all analyzed congeners. Additionally, while GAC isotherms varied considerably among compounds, AER showed a more homogeneous adsorption behavior. Activated carbon displayed lower affinity for short-chain congeners, particularly PFBA, which had the lowest log K_{ow} among the detected compounds. This is consistent with previous findings obtained in full-scale tannery wastewater treatment systems (Gottardo et al., 2023). Similar trends have been reported for synthetic contaminated waters, where AER outperformed activated carbon in the removal of short-chain ($C \leq 4$; PFBS and PFBA) and ultrashort-chain PFAS (e.g., perfluoropropionic acid, PFPrA), although at higher concentration ranges than those investigated in the present work (Lenka et al., 2024). The lower affinity of GAC for short-chain PFAS is attributed to its predominantly hydrophobic adsorption mechanism, which depends on interactions between the carbon surface and the fluorinated chain. In contrast, AER removal is mainly governed by electrostatic interactions, resulting in a more uniform affinity across congeners with different chain lengths.

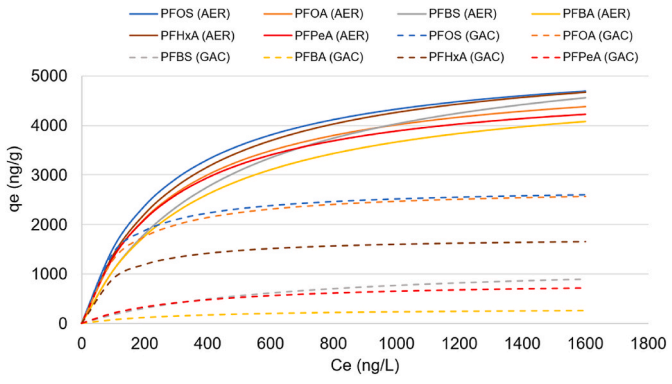

Fig. 4. Fitted Langmuir isotherms for individual PFAS congeners using GAC and AER as adsorbent materials. Experimental equilibrium data points used for model fitting are reported in the Supplementary material.

3.5. Engineering implications for full-scale implementation

To provide a preliminary order-of-magnitude assessment, a scaling analysis was conducted assuming a CD treatment capacity of 1000 m^3/d. As it was based on equilibrium-derived adsorbent inventories, the analysis should be regarded as a preliminary engineering estimate rather than a full-scale design. Based on the Langmuir model, approximately 3 ton of AER and 30 ton of GAC would be required to achieve a target PFAS concentration of 500 ng/L, while 14 ton of AER and 151 ton of GAC would be needed to reach a more stringent target of 100 ng/L. Assuming representative bulk densities of 500 kg/m^3 for GAC (Crittenden et al., 2012) and 700 kg/m^3 for strong-base anion exchange resins (consistent with manufacturer specifications and with the material used in this study), the corresponding bed volumes would range from a few cubic meters up to ~20 m^3 for AER and from 60 to 300 m^3 for GAC. Under representative design conditions (e.g., bed depth of 1.5 m), these volumes would translate into only 1-2 adsorption columns for AER, whereas GAC could require several parallel units (up to ~30 columns under stringent targets), highlighting a substantial difference in infrastructure footprint.

In continuous fixed-bed systems, design is governed by both adsorption capacity and hydraulic contact time (Tchobanoglous et al., 2014). For the assumed flow rate ($Q \approx 41.7$ m^3/h), full-scale GAC system treating PFAS-contaminated water typically operate with EBCT values of 10-13 min (Appleman et al., 2014), corresponding to bed volume of ~7–9 m^3. For ion-exchange resins, pilot-scale studies reported EBCT values of ~3 min (Chow et al., 2022), although these values are strongly dependent on resin properties and operating conditions (Loganathan et al., 2024). These volumes are substantially smaller than those required by adsorbent inventory under stringent treatment targets, indicating that system sizing may be primarily affected by adsorption capacity rather than hydraulic contact-time. This does not imply that longer EBCT values should be adopted in full-scale applications; rather, it indicates that the adsorbent inventory needed to achieve low residual PFAS concentrations may exceed the bed volume dictated by hydraulic requirements alone. Consequently, increasing EBCT would not necessarily be the most effective design strategy when adsorption performance is constrained by matrix competition or rapid breakthrough. From an engineering and economic perspective, these differences translate into distinct capital and operational cost profiles. The larger adsorbent inventories required for GAC, particularly under stringent PFAS discharge limits, imply greater contactor volumes and solid handling requirements, potentially increasing capital expenditures (CAPEX). In contrast, AER systems require substantially smaller bed volumes but typically involve chemical regeneration with saline or alkaline solutions, requiring dosing systems and management of secondary waste streams, with implications for operational expenditures (OPEX).

Importantly, the CAPEX/OPEX balance may vary depending on the regulatory target. If compliance is based on total PFAS concentration, system sizing will depend on the behavior of the overall PFAS mixture. However, if a specific congener (e.g., PFOS) drives compliance, or if total PFAS is dominated by short-chain compounds (e.g., PFBA or PFBS), the relative performance of the adsorbents may differ. As observed in this study, GAC showed lower affinity for short-chain PFAS than AER, potentially requiring larger adsorbent inventories to achieve the same target concentration. Under these conditions, the infrastructure and operational burden associated with GAC may increase disproportionately, shifting the economic balance between the two technologies.

It should be emphasized that these calculations are based on batch equilibrium data and do not account for breakthrough behavior, adsorption kinetics, competition from dissolved organic matter or inorganic ions, or long-term fouling under continuous-flow conditions. Therefore, the estimates should be regarded as order-of-magnitude values intended to compare the adsorbent inventories and infrastructure footprints of the two technologies rather than as design criteria.

Pilot-scale fixed-bed column studies are required to determine breakthrough curves, regeneration or replacement frequency, and realistic life-cycle costs before full-scale implementation.

4. Conclusions

This study demonstrated that AD represents a viable strategy for the energy valorization of tannery sludge, ensuring stable performance under both mesophilic and thermophilic conditions. PFAS were consistently detected in CD, with total concentrations exceeding 10,000 ng/L and a profile dominated by short-chain compounds such as PFBS and PFHxA. No significant differences were observed between mesophilic and thermophilic digestates, suggesting that the operating temperature had a little influence on PFAS occurrence in the liquid fraction. However, as a complete PFAS mass balance was not performed, conclusions regarding degradation, phase redistribution, or precursor transformation should be interpreted cautiously. Adsorption tests revealed that AER exhibited higher adsorption capacity than GAC for both total PFAS and individual congeners. AER also displayed more uniform adsorption across chain lengths, whereas GAC showed lower affinity for short-chain compounds. These results are consistent with the different adsorption mechanisms governing the two materials.

A preliminary scaling analysis indicated that stringent discharge targets may result in substantial differences in adsorbent inventory and infrastructure footprint between AER and GAC, although pilot-scale fixed-bed studies are required to validate these estimates under continuous-flow conditions. Future regulations focusing on short-chain PFAS may further amplify these differences, potentially altering the relative competitiveness of adsorption media.

Overall, integrating AD with targeted PFAS post-treatment appears a promising approach for sustainable tannery sludge management. Nevertheless, pilot-scale studies remain necessary to confirm long-term adsorption performance and define realistic operational and economic parameters for full-scale implementation.

CRediT authorship contribution statement

Marco Gottardo: Conceptualization, Data curation, Investigation, Methodology, Supervision, Writing – original draft, Writing – review & editing. **Giulia Adele Tuci:** Conceptualization, Data curation, Formal analysis, Visualization. **Sara Danieli:** Data curation, Formal analysis, Investigation, Methodology. **Federico Battista:** Conceptualization, Investigation, Visualization. **Paolo Pavan:** Funding acquisition, Resources, Visualization. **Francesco Valentino:** Conceptualization, Project administration, Supervision, Validation, Writing – original draft.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jenvman.2026.130412.

Data availability

Data will be made available on request.

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