

Treating water contaminated with PFAS (PFC) using functional precipitants in accordance with the PerfluorAd method

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Abstract

The use of the PerfluorAd functional precipitant is providing the market with a new water treatment method specializing in per- and polyfluoroalkyl compounds (PFAS) that can be used either alone or as a pre-treatment stage. This method is based on the principle of precipitation with liquid active ingredients, which are selected depending on the PFAS load and the water matrix and metered into the water flow. The precipitation process can be carried out in a traditional stirred tank. The resulting precipitate is separated using sedimentation and/or filtration techniques.

Key words: industrial wastewater, water treatment, perfluorinated compounds, polyfluorinated compounds, adsorption, precipitation.

1. Introduction

Perfluorinated and polyfluorinated chemicals, for which the acronym PFC is used in the national language area and the acronym PFAS in Stenert the international linguistic area as well as in the scientific literature, have gained increasing importance in the past 10 years. In the meantime, the relevance of this group of substances is sufficiently well-known to experts due to their relevance to human and ecotoxicology, their persistence and the ubiquitous spread. However, the PFAS continue to present challenges to science and practice in the field of human toxicological classification of many individual substances as well as the identification and classification of so-called Precursor substances whose concentrations can only be addressed by sum parameters. Depending on the PFAS composition and the concentration of the individual PFAS congeners as well as the organic and inorganic water matrix and the desired treatment objectives, the removal of the PFAS from aqueous media may still pose technical challenges and may be difficult. lead to high investment and operating costs.

This paper discusses the possibilities and limits that arise when using adsorption systems in connection with the treatment of PFAS-contaminated waters. Furthermore, the application of an innovative cleaning method, the precipitation with newly developed functional PerfluorAd active ingredients (hereinafter referred to as functional precipitants) for the optimization of the treatment process of PFAS contaminated waters explained. Based on case studies from different fields of application the range of possible applications for these functional precipitants should be presented.

Possibilities and limitations of other water purification processes in connection with PFAS have been highlighted in various publications in recent years. [1] [2] [3] [4] [5] [6] Apart from adsorption, membrane, and PerfluorAd

precipitation, marketable alternatives for large-scale applications and targeted removal of PFAS from water are unknown. In this Review, the processes of adsorption and precipitation as well as the combination of these two technologies are discussed in more detail. In Section 4.2 is among others, an application in the field of drinking water treatment, in which a membrane system is combined with an application of functional precipitants.

1.1 Instigation

Impurities from municipal and industrial wastewater, natural surface waters and groundwaters result from the decades-long use of PFAS in various applications. Due to their special material properties, PFAS have been and are used in numerous industrial and commercial applications. Examples are so-called.

Aqueous film forming foam (AFFF) fire extinguishing agents, applications of PFAS in galvanic processes (chrome plating), use in paper and textile manufacturing. From such applications, especially as a result of accidents, improper handling or inadequate treatment of wastewater, entries are made in soil and groundwater and, due to many years of emission via the sewage path, in the surface waters.

Thus, the introduction of PFAS into the environment is much more complex than is known from other trace substances that enter the surface waters through drains and sewage treatment plants.

1.2 Legal Requirements and Guidelines

In German law, binding legal provisions have so far only been established in two places for PFAS or for PFAS individual substances. In the Surface Waters Ordinance [7], the annual mean value of JD-UQN of 0.65 ng/L and the maximum value of ZHK-UQN of 36 µg/L for above-

ground waters - without transitional waters - has been established for the individual substance perfluorooctanesulfonic acid (PFOS), which is relevant for human toxicity. Furthermore, the Fertilizer Regulation [8] defines a limit of 0.1 mg / kg for the sum of perfluorooctanoic acid (PFOA) and perfluorooctane sulfonic acid (PFOS) as a minor component in fertilizers.

Regulations, e.g. have not yet been published in laws or regulations, but only in decrees, guidelines, communications, etc. by individual authorities, commissions or consortia on provisions for drinking water and groundwater remediation or treatment. In addition, regulations of individual federal states differ in the selection and classification of the PFAS congeners, in the values assigned to the congeners, in the system used, etc. By way of example only, some publications will be referred to below, e.g. to the recommendation of the Federal Environmental Agency (01/2017) "Update of the preliminary evaluation of per- and polyfluorinated chemicals (PFC) in drinking water" [9], the decree of the Ministry of Environment, Climate and Energy Baden-Württemberg (06/2015) "Preliminary JRC Values PFC for Groundwater and Leachate from Harmful Soil Changes and Contaminated Sites" [10] and the Guidelines of the Bavarian State Office for the Environment (04/2017) "for the Preliminary Assessment of PFC Impurities in Water and Soil". [11]

The classifications of the currently analytically quantifiable PFAS congeners follow the trend that, based on the current state of knowledge, the longer-chain PFAS substances are assigned a higher toxicological relevance than the shorter-chain compounds. The shorter chain compounds have been given higher tolerance values in the recent past. Nevertheless, the Federal Environment Agency considers that the short-chain PFAS compounds (the so-called C4 to C6 structures) also have particularly worrying properties, such as longevity and mobility in soil and water. Furthermore, these structures may also be attributed toxic as well as accumulating effects in organisms. [12]

Since the discussion on the classification and target values for the PFAS substance group - including the short-chain congeners and the precursor substances that cannot yet be analytically determined as individual substances - has not yet been completed, but the "target value discussion" will continue with increasing knowledge, one appears Align water treatment technology to those structures that currently have less knowledge.

2. Water Treatment Process

2.1 Adsorption in a Fixed Bed

As currently most commonly used for the purification of PFAS contaminated water adsorption on activated carbon or ion exchanger in a fixed bed applies. [5] [6] Meanwhile, the limits of adsorption for this group of substances are well known. In particular, the polar short-chain PFAS congeners, such as perfluorobutanesulfonic acid (PFBS), perfluoropentanesulfonic acid (PFPeS) and in particular the short-chain perfluorocarboxylates perfluorobutanoic acid (PFBA) and perfluoropentanoic acid (PFPA) can be

classified as having very limited adsorbability. In recent studies [13] it was pointed out that in addition polyfluorinated precursor compounds, which can not yet be analytically detected as individual substances, can also be present in groundwater, which is used for drinking water. Furthermore, it can not be ruled out that such substances break through even earlier than the analytically detectable short-chain perfluorocarboxylic acids.

When using adsorbents, the water matrix can have a significant impact on the efficiency of the adsorption process. In this regard, in particular, competition effects due to an adsorbable organic background load of the water are mentioned.

Against the background of a possible presence of precursor substances and at low target values for the analytically detectable PFASs, the operation of a fixed bed adsorption demands high standards. The adsorbent requirement and thus also the operating costs of the treatment process can be very high as a result.

2.2 Use of Functional Precipitants

When using PFAS water purification technologies, it is important to apply procedures that are capable of ensuring low PFAS target values while still providing economically viable operating costs.

As it has repeatedly been observed in numerous applications over the past 10 years that PFAS contaminations of water from the use of fixed-bed adsorbents as well as the use of powder adsorbents often result in neither satisfactory cleaning results nor economically acceptable costs, Cornelsen Umwelttechnologie GmbH, Essen, in cooperation with the Fraunhofer Institute UMSICHT, Oberhausen, has developed functional precipitants specialized in PFAS. These precipitants are added to the PFAS-contaminated water as a liquid agent, causing precipitation of the dissolved PFAS compounds. The addition of the functional precipitant occurs in the flowing water stream, i. it is fed into a container or within a tube. After introduction of the active ingredient, depending on the contaminant inventory and the water matrix, the formation of microflocs takes place, which contain significant amounts of the previously dissolved PFAS molecules in undissolved form. These are separated from the water phase by conventional methods of solid-liquid separation, such as sedimentation and / or filtration, dehydrated and fed to a proper disposal. If necessary, powdered coal is added simultaneously or subsequently to the water pretreated with the functional precipitant, or the water is finally purified in a subsequent fixed bed adsorber (Figure 1).

The use of functional precipitants has some advantages that are important in removing difficult to adsorb PFAS compounds: Due to the fact that the active ingredients have been tailored to PFAS as specialized precipitants, there is hardly any negative influence from other organic water constituents.

A significant negative influence of the PFAS cleaning process, as it must be assumed at higher dissolved

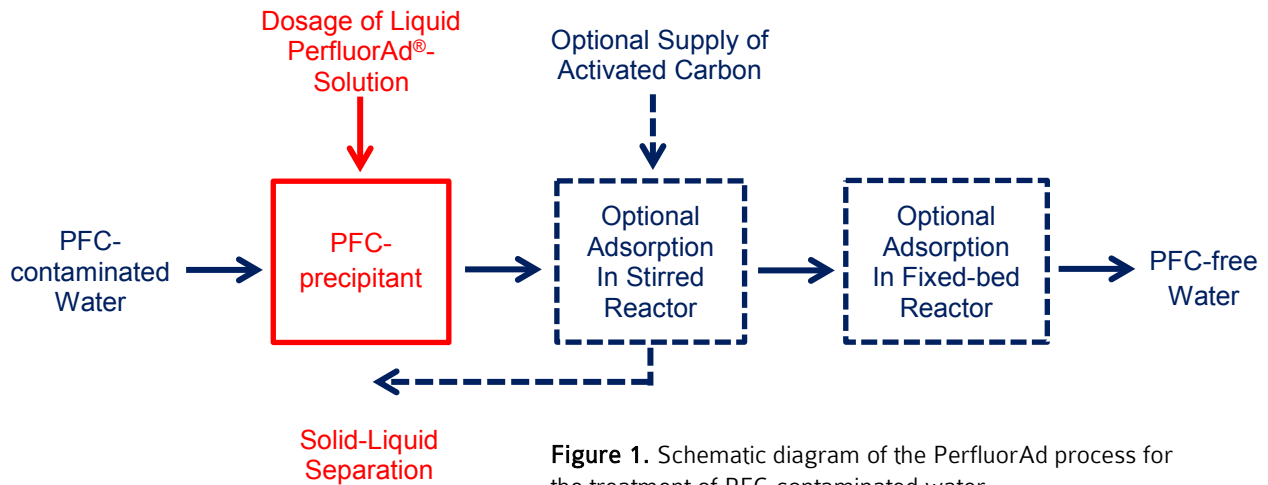


Figure 1. Schematic diagram of the PerfluorAd process for the treatment of PFC-contaminated water

organic carbon (DOC) concentrations in a classical adsorption, has not been observed so far.

The precipitation process in the stirred reactor is not a "static system", because this process can - in contrast to an adsorption in a fixed bed - be actively influenced at any time. Depending on the current flow rate, the PFAS concentration, the concentration of other impurities, etc., the functional precipitant can be varied and also its dosage can be adjusted to the respective boundary conditions.

It is therefore a flexible process, changing water conditions - as e.g. in the case of groundwater remediation measures, in the treatment of PFAS-contaminated extinguishing waters or other complex polluted wastewater - can be adapted at any time by modifying influencing variables. The apparatus technology required for the use of functional precipitants can be described as simple and at the same time robust and also offers the possibility of being able to use further active substances, for example in to modify the pH or conventional flocculants and flocculants, defoamers or powdered absorbents use.

The functional precipitants are a group of active substances from which the most suitable active substance or combination of active substances is selected, depending on the concentration of the individual PFAS substances and the target values targeted for the individual substances and on the pending water matrix. The active substances are biodegradable and not hazardous to water and thus follow the principle of sustainability.

The use of functional precipitants for the (pre-) treatment of PFAS-contaminated waters can significantly reduce the use of active ingredients. This applies not only to the consumption of the activated carbon required only for the final treatment, but also as a general rule, very low dosing required of the functional precipitant. The low use of active ingredients is not only ecologically and economically reasonable, but also optimizes the PFAS-contaminated residue quantities that are required for disposal.

3. Possibilities, limits and optimization potential for water purification processes

All of the results presented below are from tests carried out at Cornelsen Umwelttechnologie GmbH in Essen and on experimental plants. The PFAS concentrations were determined according to DIN 38407-F42: 2011-03 by a DAkkS accredited laboratory.

3.1 Breakthrough behavior and runtimes of adsorber plants

The breakthrough behavior of adsorber plants is influenced by several factors. The composition of the PFAS contamination of the water as well as the concentration of each congener are very important influencing factors. The background organic load of the water is also an extremely important influencing variable, at least when using activated carbons, which at higher contents can lead to complete inefficiency of the adsorption process with respect to the removal of individual PFAS components. The adsorber material as such is also of particular importance, since it has repeatedly been observed in the case of the difficultly adsorbable PFASs that there is not "a suitable activated carbon product" but that a project-specific identification of the presumably optimum adsorber material is to be recommended.

The desired treatment goal also has an influence on the achievable running times of the activated carbon adsorbers and thus on the cost development of the treatment process. In the figures 2a, 2b, 3a, 3b, 4a and 4b, breakthrough curves of activated carbon adsorbers are shown in various diagrams. Furthermore, it is shown how the breakthrough behavior of the activated carbon can change, provided that a pretreatment of the water is carried out with functional precipitants. The example is a groundwater, which is burdened on average with > 35 µg/L total PFAS. The DOC content of this water is still inconspicuous with <3 mg/L. The illustrations refer to a consideration after each 2.0 m height of dumping.

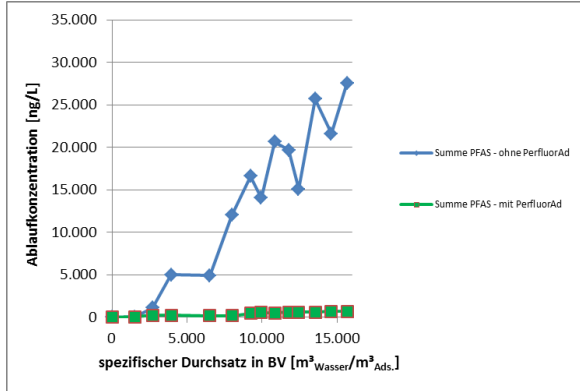


Figure 2a. Breakthrough behavior of activated carbon filters after 2 m bed height with and without PerfluorAd pre-treatment at a mean raw water concentration of 35,300 ng/L. Total PFAS.

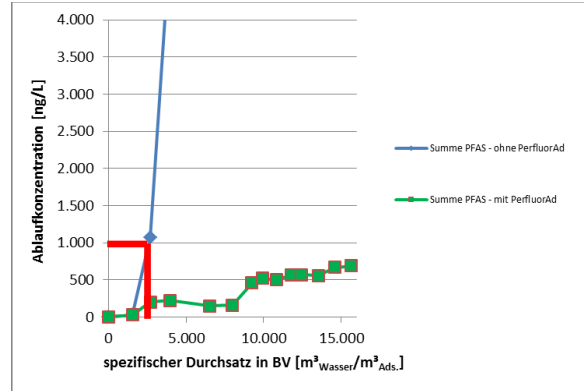


Figure 2b. Breakthrough behavior of activated carbon filters after 2 m bed height with and without PerfluorAd pre-treatment at a mean raw water concentration of 35,300 ng/L Total PFAS; with representation of the breakthrough criterion 1.000 ng/L sum PFAS.

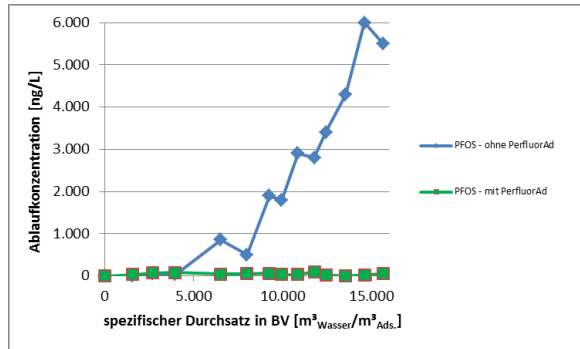


Figure 3a. Breakthrough behavior of activated carbon filters after 2 m bed height with and without PerfluorAd pre-treatment at a mean raw water concentration of 11,000 ng/L PFOS.

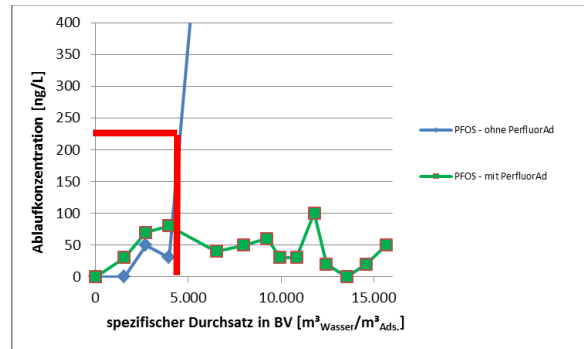


Figure 3b. Breakthrough behavior of activated carbon filters after 2 m bed height with and without PerfluorAd pre-treatment at a mean raw water concentration of 11,000 ng/L PFOS; showing the breakdown criterion 230 ng/L PFOS.

Figures 2a and 2b show the breakthrough behavior of the activated carbon filter after 2 m bed height for the sum of currently analyzable PFAS compounds. In Figure 2b, the sum of PFAS of 1.0 µg/L used in North Rhine-Westphalia/Germany for sewage discharges as an orientation value was assumed as the evaluation criterion. When using this evaluation criterion, the activated carbon filter without pretreatment only achieves a specific throughput of less than 2,500 BV [bed volume in m³ Water/m³ Ads.]. The activated carbon filter behind a pretreatment with the functional precipitant PerfluorAd, however, reaches up to this point run times of well over 15,000 BV.

In Figures 3a and 3b, the same breakthrough is shown, but here shown for PFOS and a breakthrough score of 0.23 µg/L. This criterion is achieved when using activated carbon at less than 5,000 BV only. For the activated carbon filter after precipitation with PerfluorAd, run times of more than 15,000 BV are also achieved for this evaluation criterion.

In the figures 4a and 4b, the breakdown behavior for the sum of PFOS, PFOA and PFHxS applied, which was used in the past for groundwater remediation with 0.3 µg / L as an introduction criterion. The activated carbon filter

without pretreatment achieves this criterion after about 2,500 BV, whereas the activated carbon reaches a specific throughput of more than 11,000 BV after pretreatment with PerfluorAd®.

It can thus be stated that, depending on the pending boundary conditions and the desired treatment target, pretreatment by means of functional precipitants can lead to a significant increase in the specific throughput and thus to a significant increase in the service life of the adsorption type plants.

3.2 Filter Regime

The knowledge of so-called displacement effects in adsorption type plants is well-established in water purification technology, yet in practice there are rarely such significant effects as can be seen with the difficult adsorbable PFAS compounds.

In Figures 5, 6 and 7, the previously used example is continued and the breakdown behavior for the PFAS congeners PFBA (Fig. 5), PFPeA (Fig. 6) and PFHxA (Fig. 7) is plotted. A horizontal red line represents in each case the average raw water concentration of the congeners mentioned above. It can be seen that these compounds

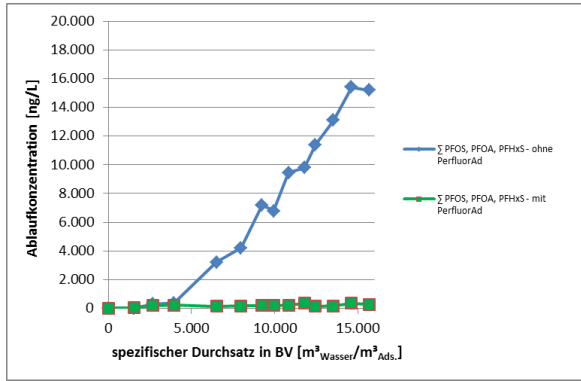


Figure 4a. Breakthrough behavior of activated carbon filters after 2 m bed height with and without PerfluorAd pre-treatment at a mean raw water concentration of 23,000 ng/L Total PFOS, PFOA, PFHxS.

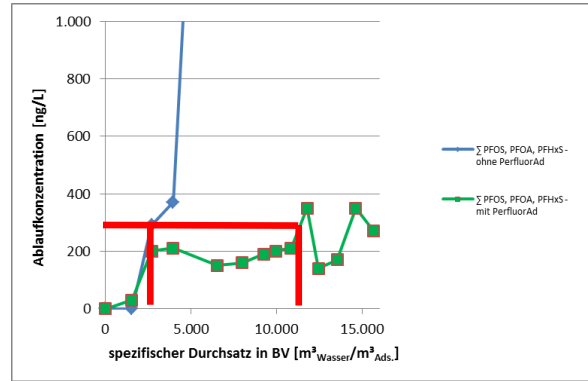


Figure 4b. Breakthrough behavior of activated carbon filters after 2 m bed height with and without PerfluorAd pre-treatment at a mean raw water concentration of 23,000 ng/L Total PFOS, PFOA, PFHxS; with representation of the breakthrough criterion 300 ng/L sum PFOS, PFOA, PFHxS.

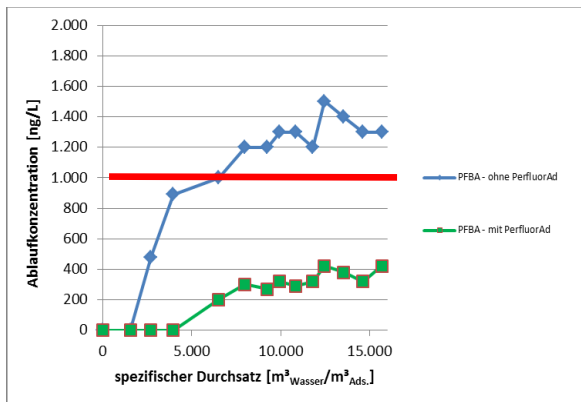


Figure 5. Breakthrough performance of activated carbon filters after 2 m bed height with and without PerfluorAd pre-treatment at a mean raw water concentration of 1100 ng/L PFBA (represented by the red line)

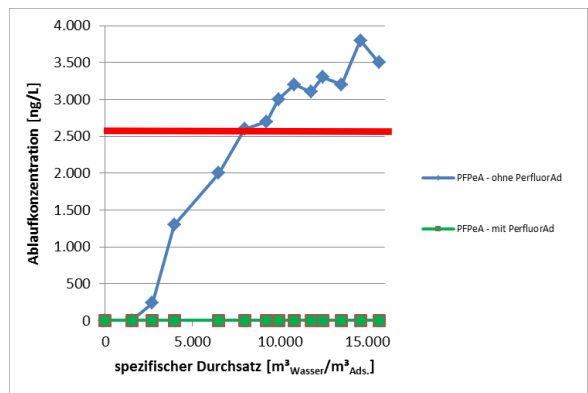


Figure 6. Breakthrough behavior of activated carbon filters after 2 m bed height with and without PerfluorAd pre-treatment at a mean raw water concentration of 2,700 ng/L PFPeA (represented by the red line)

are discharged from the activated carbon filter after a certain filter run time with a not insignificant concentration overshoot - and if they are present in practice - are transferred to the subsequent filter. These heavy adsorbable congeners can lead to a preload of the filter and its achievable life under these circumstances is noticeably restricted. It is therefore necessary to operate a filter management oriented to the breakdown behavior of the critical individual connections.

In a pre-purification of the water with PerfluorAd as a functional precipitant, the risks of an early breakthrough and a pollutant discharge, which rises above the feed concentration, significantly reduced, or - as shown in the examples above - even completely excluded.

4. Application examples for the use of functional precipitants

After more than 10 years ago it became apparent that the exclusive use of activated carbon adsorption in complex groundwater damage cases can lead to short filter runtimes and high operating costs, the functional precipitants shown here have been developed for these applications (see also Figures 2 to 7). However, the PFAS

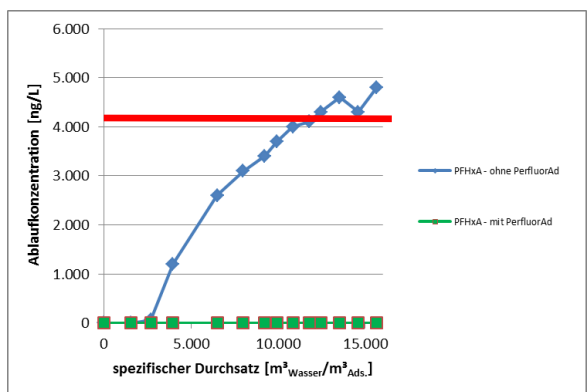


Figure 7. Breakthrough behavior of activated carbon filters after 2 m bed height with and without PerfluorAd pretreatment at a mean raw water concentration of 4,100 ng/L PFHxA (represented by the red line)

issue has long ceased to be confined to groundwater damage cases, but is now used in a large number of projects, such as the cleaning of industrial/commercial wastewater, PFAS contaminated fire water, temporary measures such as construction water retention, groundwater sampling, well drilling and cleaning up to



Figure 8. Mobile PerfluorAd system - built into 20 'containers - for temporary use for the treatment of PFAS contaminated water

applications in the field of drinking water treatment, namely the retreatment of retentate from RO systems.

The use of functional precipitants can be used both as a mobile plant solution, in the context of temporary missions, applied (Figure 8) as well as in the form of stationary solutions permanently used. Examples are given below by way of example in which an exclusive use of adsorber plants has clear limits or can be completely ruled out for cost reasons. It should be noted that the dosing quantities of the liquid agent added depends on the PFAS concentrations found in the treated waters.

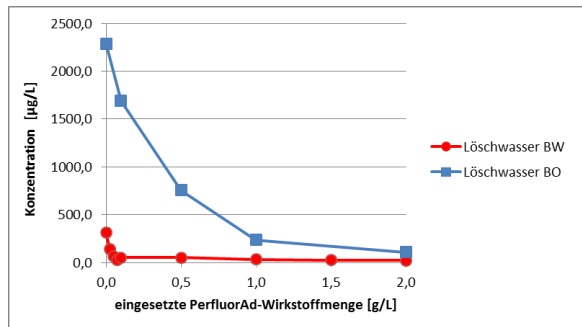


Figure 9a. Residual Concentrations Total PFAS after treatment of two extinguishing waters with different amounts of PerfluorAd active substance.

4.1 Treatment of complex contaminated fire water barrels with different PFAS inventory

Fire-fighting waters usually have a very complex water matrix as a result of the fire event and the use of potentially differently composed extinguishing agents. They are not to be cleaned with the use of adsorber alone or only at a considerable cost. In contrast, they can be treated inexpensively and in an environmentally friendly manner with the unobtrusive system technology for a precipitation reaction. In Figures 9a and 9b shows the cleaning of two different firing barrels with functional precipitants. At starting concentrations of about 0.4 and 2.3 mg/L total PFAS elimination rates of > 80% can be achieved even with a moderate organic background load of >1.0 g/L DOC even with moderate dosing amounts. If a post-treatment would have to take place, the upstream precipitation would lead to a significant under-consumption of activated carbon. Depending on the regulatory approval and the possibly existing on-site plant technical inventory, it is conceivable at high efficiencies that no further treatment by adsorption would have to take place.

4.2 Retentate supplement treatment in membrane processes for drinking water treatment

Against the background of the stringent regulation for PFOS with regard to the environmental quality standards for surface waters, an introduction of weakly PFOS contaminated water streams is already hardly possible

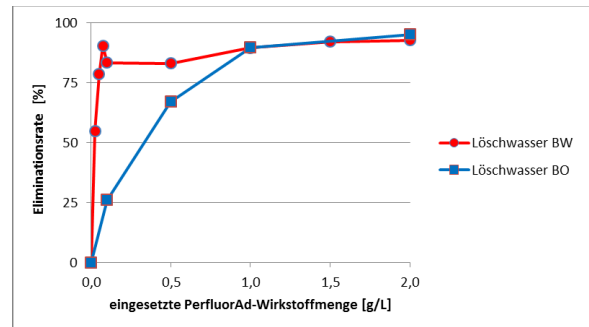


Figure 9b. Elimination rates for sum PFAS after treatment of two firing barrels with different PerfluorAd active substance amounts.

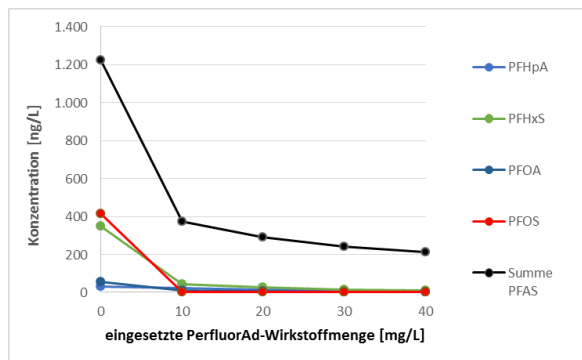


Figure 10a. Residual Concentrations Total PFAS after treatment of two extinguishing waters with different amounts of PerfluorAd active substance.

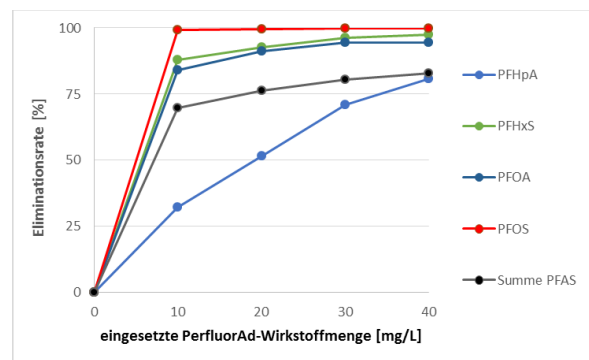


Figure 10b. Elimination rates for individual PFAS substances after treatment of a RO concentrate with different PerfluorAd active substance amounts

today. As an example of the purification of PFAS-contaminated waters by means of functional precipitants, the following is the treatment of a retentate from a reverse osmosis (RO) plant, which is used for drinking water production.

The retentate shows a total PFAS load of about 1,200 ng/L. The individual parameter PFOS, which is critical for the discharge into the surface water, has a share of 415 ng/L.

In Figures 10 a and 10b shows that even with the use of small amounts of functional precipitants (between 10 to 40 mg/L) high elimination rates can be achieved. For PFOS, starting at 415 ng/L PFOS, PFZ achieves purification efficiencies of <1 ng/L with these small amounts of active ingredient, and thus even falls below the strict specification of surface water ordinance for the annual mean value.

In the drinking water sector, there is public interest in the costs of water treatment due to the fees. Thus, a consideration of the running costs or the investment costs for a retentate supplementary treatment with the functional precipitating agent offers here. However, this can only be an estimate for the specific, individual case described above.

On the basis of an active substance use of functional precipitants for retentate supplement treatment amounting to 10 mg/L, delivery costs for this equipment of approx. 0.065 Euro/m³ of treated retentate are calculated. In addition, there are the costs listed below for sludge disposal. Cost of capital, personnel and energy are not included. On the basis of the assumption, which is very unfavorable for a residual substance determination, that the amount of functional precipitant used as liquid active ingredient is completely present as solid after the water treatment process, a maximum of 10 mg/L residue (100% dry matter) would be expected at 10 mg/L feed. Depending on the technology used for the precipitation process for solid-liquid separation (for example sedimentation, sand filtration, bag filtration, membrane processes or the like), the dry matter (TS) content of the resulting residue can be different. Based on the use of conventional sand filtration, a TS content of between 3 and 6% is to be expected in the backwash water. Through devices for sludge thickening including the return of the clear water supernatant of the TS content of the residue phase can be further reduced without major investment outlay. Any residue loaded with PFAS should be disposed of or processed to ensure a temperature level of $\geq 1,100$ °C. On the basis of a dosage of 10 mg/L, specific costs for

the disposal of PFAS-loaded media in high-temperature incinerators are estimated to be 350.00 Euro/ton, after the sludge thickening reached TS content of 20%, calculated in the use of functional Precipitant-specific costs for the disposal of residues amounting to approximately 0.018 Euro/m³ treated water. In the case of higher PFAS concentrations and high matrix effects, an increased use of active ingredients and, consequently, a higher sludge accumulation are to be expected.

For plant engineering equipment, there are costs for the additional metering station to be arranged and the required stirred reactor in which the functional precipitant is introduced and the adduct of precipitant and PFAS forms as a microflake. These are dependent on the project-specific conditions and in particular on the throughput of the system. Due to low residence times in the stirred reactor, often 5 to max. 10 min., I.d.R. be arranged only small containers, so that often result in additional investment costs of less than 50,000 euros from a supplementary arrangement of the technology required for the precipitation technology.

5. Summary and Outlook

With the use of the PerfluorAd functional precipitant, a new PFAS-specific water purification process is provided to the market, either in its sole application or as a pre-purification stage - e.g. before a conventional fixed bed adsorption - can be used.

The method is based on the principle of precipitation with liquid active substances (functional precipitants), which are selected depending on the upcoming PFAS load and the water matrix and dosed into the water flow. The precipitation process can be carried out in a classic stirred tank. The resulting precipitation products (microflocs) are separated by conventional sedimentation and/or filtration systems.

In contrast to fixed-bed adsorption, especially in complex-loaded waters, the process shows advantages due to the low use of active ingredients and the resulting low accumulation of PFAS-contaminated residues. Even with low PFAS raw water pollution, the process can be used for pretreatment or as a sole method to achieve even low target values.

In particular, with regard to the expected in the future in the introduction of PFAS (in particular PFOS) -loaded waters in surface waters strict regulatory practice, the use of functional precipitants can bring technical and especially economic benefits.

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Abbreviations

PFAS	Per- und polyfluorierte Alkylsubstanzen
PFC	Per- und polyfluorierte Chemikalien
PFT	Perfluorierte Tenside
AFFF-Schaum	Aqueous Film Forming Foam (wasserfilmbildendes Schaummittel)
PFOS	Perfluoroktansulfonsäure
JD-UQN	Jahresmittel- bzw. Jahresdurchschnittswert gemäß Umweltqualitätsnorm
ZHK-UQN	Zulässige Höchstkonzentration gemäß Umweltqualitätsnorm
PFOA	Perfluoroktansäure
GFS-Wert	Geringfügigkeitsschwellenwert
PFBS	Perfluorbutansulfonsäure
PFPeS	Perfluorpentansulfonsäure
PFBA	Perfluorbutansäure
DOC	Dissolved Organic Carbon (gelöster organischer Kohlenstoff)
BV	Bettvolumen [$\text{m}^3_{\text{Wasser}}/\text{m}^3_{\text{Ads.}}$]
PFHxS	Perfluorhexansulfonsäure

PFPeA	Perfluorpentansäure
PFHxA	Perfluorhexansäure
TS	Trockensubstanzgehalt

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