

A new Procedure for the removal of perfluorinated and polyfluorinated chemicals from complex contaminated waters

Martin Cornelsen

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The polyfluorinated and perfluorinated chemicals substance group (for which the acronyms PFCs and PFASs are also used) has gained in significance since this form of soil and groundwater contamination first became known in Germany in Brilon-Scharfenberg (North Rhine-Westphalia) in 2006. Meanwhile, the importance of this substance group's persistence, its ubiquitous occurrence and its ecotoxicological relevance are recognized. PFCs represent major scientific and practical challenges, among other things in trace analysis, in the human toxicological classification of many individual substances, in the assessment of sediment transport in aquifers, and in many other fields. In particular, the removal of PFCs from aqueous media represents a major challenge.

In this scientific paper, a new procedural approach to the purification of water contaminated by PFCs is discussed, as the relevant parameters that influence the costs of processes for treating water contaminated by PFCs.

1. Introduction

1.1 Instigation

For the first time, contamination of soil, groundwater and surface water with PFC was recognized in 2006 when investigations by the Institute of Hygiene at the University of Bonn of drinking and surface waters revealed PFCs in Ruhr and Möhne rivers [1]. The contamination resulted from a PFC contaminated material that had been applied to arable land to improve soil.

However, this entry path of PFC into the environment is not a singular event but has been observed in subsequent years in many other places as well, due to various causes. In addition, sustainable contamination of surface waters and groundwater results from the decades-long use of PFC-containing fire extinguishing agents, the so-called Aqueous Film Forming Foam (AFFF). These foaming agents were not exclusively used only for extinguishing measures, but also for training purposes. Thus, it must be assumed that, especially where extinguishing exercises were carried out for long periods by factory fire brigades, components of the extinguishing agents were also introduced into the groundwater via the soil path. Furthermore, various major fires involving the use of AFFF have caused PFC to enter the groundwater. The EU has limited the use of the PFC single substance PFOS

(perfluorooctanesulfonic acid) since May 27, 2008 to up to 0.005% [2]. Evident is that since then polyfluorinated substitutes have found increasing use, whose structure is not always known and therefore cannot be detected analytically.

Due to their special material properties, PFCs have been and are being used in production processes as well as components of products in some industries. By way of example, applications in electroplating (chromium plating), papermaking and textile production and impregnation may be mentioned here. These applications, in particular as a result of accidents and mishandling, may have resulted in inputs into soil and groundwater and, due to long-term emission via the sewage path, also into surface waters.

Thus, the input of PFCs into the environment is much more complex than that of other trace substances known to enter the surface waters via sewerage and sewage treatment plants. Thus, as an entry pathway for PFCs, not only are they spread via the sewer / sewage treatment / surface waters (for industrial or industrial effluent emissions) but also via the ground / groundwater path (due to fire extinguishing agent applications, accidents, misuse, transfer or dumping) PFC-containing substances, etc. If the PFCs have been entered into an aquifer, it must be assumed that there is a high degree of mobility - at least for

individual compounds - of the PFC substance group, which can lead to a rapid spread in the aquifer and possibly also leads to the formation of extended pollutant plumes. Since no decomposition takes place due to the high persistence of individual substances under natural environmental conditions, it must be assumed that dilution only takes place as far as possible in the aquifer, but no degradation takes place, which would lead to a significant reduction in the substance potential once recorded.

1.2 State of Knowledge and Practice

This essay deals with the topic "cleaning of PFC-contaminated waters", so that other disciplines will not be discussed here. The survey of possibilities and limits of known water purification processes in connection with PFC has been highlighted in the past years in various publications [3] [4], but the topic still remains a challenge for science and practice. In studies, amongst other things, considering applications of evaporation processes and electrochemical processes for the treatment of electroplating effluents [5]. Pilot applications of oxidation processes in the treatment of PFC-contaminated groundwater are known [6]. Large-scale applications for a targeted removal of PFC from wastewater by oxidation processes or membrane processes are not yet known.

The most frequently piloted and industrially applied process currently used for the purification of PFC-contaminated water is the adsorption on activated carbon and ion exchangers in a fixed bed [7], [8]. Nevertheless, the limits of adsorption are evident for this substance group. In particular, the polar short-chain individual substances, such as perfluorobutanesulfonic acid (PFBS), perfluorhexanesulfonic acid (PFHxS) and in



Figure 1. Column Test Facility for Comparative Assessment of Adsorbents

particular the short-chain perfluorocarboxylates perfluorobutanoic acid (PFBA) and perfluoropentanoic acid (PFPeA) must be classified as hardly adsorbable on currently known commercially available adsorbents [9]. In addition to the substance characteristics of the individual PFC substances as well as the concentration of individual PFC substances and their concentration ratios, the water matrix can have a significant influence on the efficiency of the adsorption process. In particular, it is pointed out that there are competition effects which occur due to an adsorbable organic background load of the water and by the presence of long and short-chain PFCs and which often lead to low loading values, as well as premature breakthrough of heavy-adsorbable - mostly short-chain - fluorine compounds. The basics of adsorption as well as the knowledge of negative influences have been known for decades [10] [11] and are confirmed once more in this new group of substances. Nevertheless, it has to be stated that sufficient empirical data are lacking in the assessment of the adsorption behavior of individual PFC substances and their behavior in the case of competing adsorption in order to be able to make reliable forecasts of achievable loadings and thus service life and operating costs. In the course of studies on the adsorption of activated carbon PFC, which were carried out by the Fraunhofer.

Institute UMSICHT and Cornelsen Umwelttechnologie GmbH, standardized column test systems have been designed and built for semi-technical scale, which make it possible to pilot several adsorbent materials in parallel to thus being able to carry out a comparative assessment of products under real conditions (**Figure 1**). Installed in mobile containers, the columns can be used for pilot applications at the location of the water attack and thus be used under practical conditions for an assessment of adsorbent materials, which is aligned to the upcoming PFC load and the matrix situation of the water.

¹ Perfluorinated surfactants (PFT) belong to the group of fluorocarbons and have an exclusively anthropogenic origin. PFTs are organic surface-active compounds in which the hydrogen atoms of the alkyl group have been completely replaced by fluorine atoms.

² Perfluorinated and polyfluorinated chemicals (PFCs) include the group of PFTs as well as the group of polyfluorinated chemicals (polyFT), in which a large part of the molecule is completely fluorinated, but part of the molecule is fluorine-free.

2. Development of a PFC-specialized Water Treatment Process

2.2 Problem and Objective Targets

The type and concentration of individual PFC compounds to be treated in the water, the organic and inorganic water matrix, and in particular the treatment targets to be achieved, are important criteria for process selection and have an impact on the operating costs resulting from the water treatment process.

Depending on the particular case of application, the processing targets may be highly different due to the current legal basis. By decree of 16.06.2014 ("reevaluation of PFT substances") applies e.g. in NRW for discharges into surface waters an orientation value of 0.3 µg/L for the sum PFOA + PFOS and an orientation value of 1.0 µg/L for the sum of all measured PFCs. In this context, it should be pointed out that approximately 12 to 24 individual PFC parameters are determined with currently standard laboratory analysis. Studies by the DVGW Technology Center for Water (TZW), which are based on comparative measurements with the newly developed AOF sum parameter, give clear indications that the currently determinable amount of individual PFC substances often does not cover all organofluorine constituents [12]. As an extreme requirement, compliance with the value given in the so-called environmental quality standard [13] for the perfluorooctane sulfonic acid (PFOS) of 0.65 ng/L can be considered, which is even far below the current quantification limit, given the current possibilities of PFC trace substance analysis. In the case of discharges of PFOS-containing waters into surface waters, where proportionate, ensure that the requirements of the EU Directive on the environmental quality standard are observed. The maximum permissible discharge value required for this results from the ratios of the volume flows of the discharge point and the watercourse. With small differences in the volume flows, a low treatment target value is required, which equates to a "zero emission", as detection of PFOS - currently a limit of quantification of 10 ng/L for this compound is often indicated - already significantly exceeds this value in the process plant would attest.

When using such a low target value, the treatment plant would thus have to be operated at any time in a state that ensures a zero emission. The adsorbent requirement, and thus the operating costs of the treatment process, would be correspondingly high.

The use of water purification technologies to remove PFCs therefore requires the adoption of reliable

procedures that are capable of ensuring low PFC target values, while still providing economically viable operating costs.

It can be assumed that the scope of the analytically determinable individual PFC substances will increase permanently. Keep in mind that as a result of the use of substitutes, more and more new PFC compounds are being placed on the market, which - if they occur in water and can be analyzed - must be included in the "total PFC". When choosing a process for the purification of PFC-contaminated water, therefore, it should not be limited exclusively to the content that can currently be determined as "total PFC", but should also take into account that boundary conditions - as described above - could change in perspective.

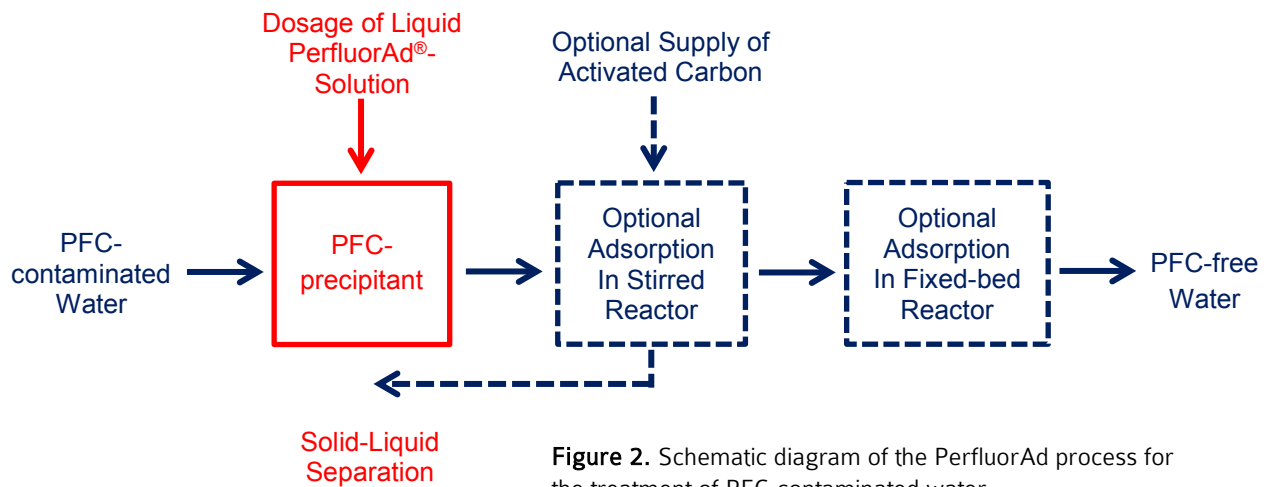
2.2 Process Description

Since it has repeatedly been observed in studies as well as in numerous applications that not always satisfactory cleaning results result in economically acceptable costs in the case of PFC contaminations of the water from the use of fixed-bed adsorbers and also from the use of powder adsorbents, Cornelsen Umwelttechnologie GmbH, Essen in cooperation with the Fraunhofer Institute UMSICHT, Oberhausen develops the PFC-specific PerfluorAd process.

This process is not an adsorption process but is based on the principle of forming adducts by adding a liquid agent to the PFC-contaminated water. The aim is thereby a formation of adducts of liquid active and PFC to precipitate the previously dissolved PFC compounds. The addition of the liquid active substance called PerfluorAd® takes place in the flowing water stream, in other words, it is fed into a container or within a tube.

After introduction of the active ingredient is carried out depending on the pollutant inventory and the water matrix in a stirred tank within 10 to max. 30 minutes flocculation of the PFC is complete. These are separated by conventional methods of solid-liquid separation, such as sedimentation and / or filtration from the water phase, dewatered and fed to proper disposal. If necessary, powdered activated carbon is added simultaneously to the water thus treated or it is post-treated in a subsequent fixed bed adsorber (**Figure 2**).

The PerfluorAd process and the PerfluorAd active ingredients used for it have been tested in recent years in the laboratory as well as several times as pilot scale. The first large-scale permanent applications have been running since 2015.



2.3 Process Optimization

The PerfluorAd precipitation process has several advantages that are important in removing difficult to adsorb PFC compounds:

1. Due to the fact that the liquid PerfluorAd active ingredients (also referred to as catchers or catcher substances) are directed to a specific interaction with PFC, there is hardly any negative influence from other organic water constituents. A significant negative impact of the PFC purification process, as it must be assumed at higher DOC concentrations in a classical adsorption, therefore does not occur in the specialized PerfluorAd active ingredients.
2. In the PFC precipitation process, other organic as well as inorganic accompanying parameters can be treated. Thus, e.g. precipitable DOC structures - such as humic acid compounds - and iron or turbidity can be addressed simultaneously, without the need for separate steps should be arranged.
3. The PerfluorAd process or the precipitation process in the stirred reactor is not a "static system", this process can - in contrast to an adsorption in a fixed bed - are actively influenced at any time. Depending on the current flow rate, the PFC concentration, the concentration of other impurities, etc., the PerfluorAd active ingredient can be varied and also its dosage can be adjusted to the respective boundary conditions. It is therefore a flexible process designed to deal with changing water conditions, such as e.g. in the case of groundwater remediation measures, in the treatment of PFC-contaminated

extinguishing waters or other complex polluted effluents - can be adapted at any time by modifying influencing variables.

4. The apparatus technique required for the PerfluorAd technology can be said to be simple yet robust, and also provides the opportunity to use other active ingredients, e.g. to modify the pH or the process flocculants, flocculants, defoamers or powdered absorbents supply.
5. The liquid active ingredient PerfluorAd® is classified as biodegradable and thus follows the principle of sustainability.

2.4 Minimization of Auxiliary Use and Residue Formation

There are several PerfluorAd active ingredients available, which are selected depending on the concentration of individual PFCs and the target levels targeted for the individual substances as well as the upcoming matrix of the water to be treated. Also, combinations of the PerfluorAd active ingredients can be used.

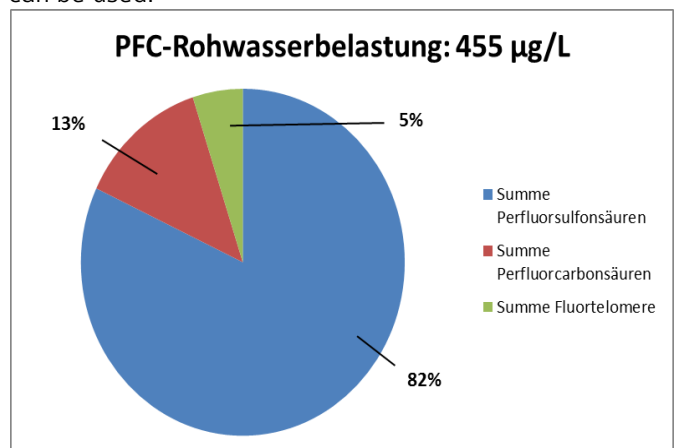


Figure 3. PFC Raw Water Pollution

The achievable for the PFC removal efficiencies are thus dependent on the active ingredients used and their amount used. **Figure 3** shows the initial situation of a groundwater polluted by the use of a PFC-containing extinguishing agent (starting concentrations: sum PFC 455 µg/L, DOC 15 mg/L).

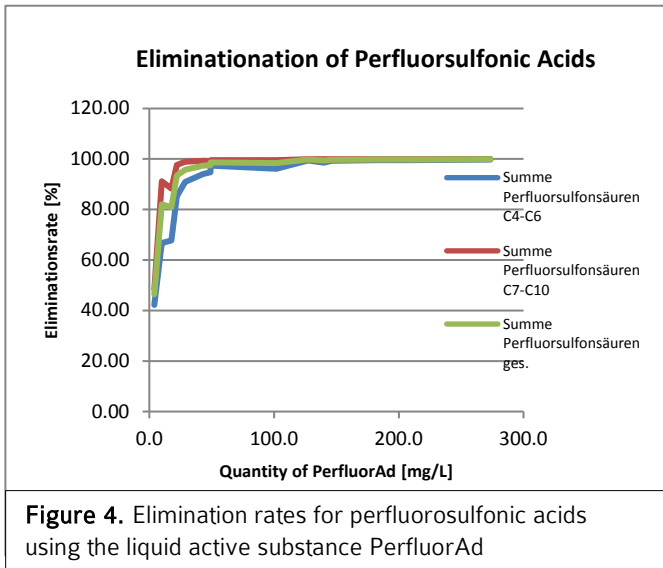


Figure 4. Elimination rates for perfluorosulfonic acids using the liquid active substance PerfluorAd

Figure 4 shows typical efficiencies of the active ingredient flow using PerfluorAd® as an example of this groundwater. Perfluorosulfonic acids with different chain lengths (C4-C6, C7-C10, total) are used as parameters.

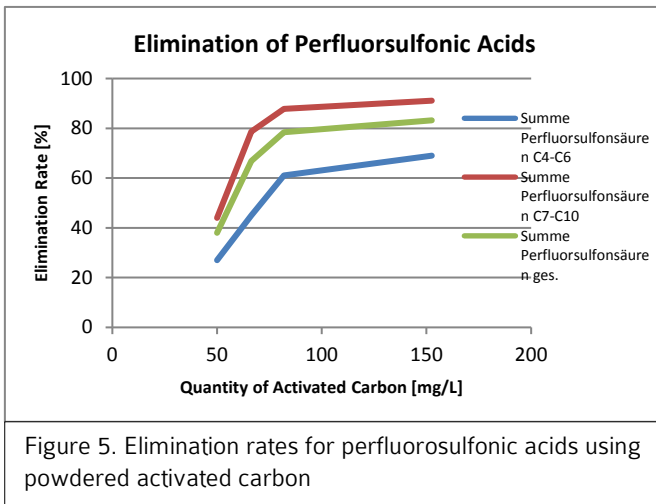


Figure 5. Elimination rates for perfluorosulfonic acids using powdered activated carbon

Figure 5 shows the values obtained for the same water when using the powder activated carbon most suitably determined for this application. Efficiencies comparable with the PerfluorAd material are achieved with the use of powdered activated carbon only at a much higher dosage.

Project-related, it is important to use a suitable active ingredient at its optimal dosage, in order to set the desired efficiency. As already mentioned, in this context it is important to note that the PerfluorAd cleaning process can be influenced at any time and can therefore be "adjusted" to the preparation target. If, due to the desired target values, no high degree of efficiency is required, it is possible to achieve the cleaning goal by feeding in small quantities of PerfluorAd. As a result, a minimization of the use of active ingredients and at the same time an optimization of the costs resulting from the water purification process total costs are possible.

Figure 6 shows the relationship between dosing quantity and efficiency for the PFC single compound PFOS - the substance that has so far been assigned the highest toxicological significance. It is clear that for PFOS a very high efficiency is already achieved at lower PerfluorAd dosing than this is the case with the use of powdered activated carbon. If only one reprocessing target value should be set for PFOS or PFOS is the main contaminant, the targeted goal can already be achieved by minimizing the use of active ingredients by deliberately discontinuing the reprocessing process for this substance.

Such a possibility for influencing the treatment process does not exist in the application of adsorption in a fixed bed. In this method, the time at which the activated carbon needs to be replaced, determined solely by reaching the relevant limit in the effluent of the activated carbon filter - regardless of whether the activated carbon for other substances at this time might still have had adsorption capacity.

Scientific studies [6] and various practical applications [14] have shown that the use of activated carbon often only achieves 0.01% to a maximum 0.1% for cleaning PFC-contaminated water. Depending on the respective boundary conditions, load values for short-chain PFC substances may even be significantly lower.

On the other hand, considering the amount of active ingredient used in relation to the amount of pollutant removed in the PerfluorAd process, capacities of up to 1.0% or more can be achieved (i.e., 1g PFC per 100g of active ingredient). Project-related values of >4.0% have already been achieved with the PerfluorAd process. In the case of complexly contaminated waters, a factor ≥10 improves efficiency compared to the use of conventional activated carbon and/or iodine Ion-exchange materials.

Figure 7 shows a comparison of the pollutant active ingredient ratio in relation to the respectively

achieved elimination rate for PFCs, when using the liquid agent PerfluorAd® and powdered activated carbon.

It can therefore be concluded that the amount of active substance required is significantly more favorable when using the PerfluorAd process at higher PFC concentrations than in activated carbon applications. Accordingly, the PFC contaminated amount of waste to be disposed of in the PerfluorAd process is also lower than in adsorption processes.

The current scientific knowledge is that PFCs are fully mineralized only at a temperature level of >1,100 °C [15]. Only from this temperature level can it be assumed with a high degree of probability that no fluorinated transformation products will form in the thermal process. Any residuals that occur in a PFC water purification process, such as iron hydroxide sludge, activated carbon, etc., would therefore be supplied to a suitable high-temperature combustion. For these reasons, groundwater remediation, which typically involves reactivating the loaded activated carbon, would instead require the combustion of PFC-loaded activated carbon in high-temperature incineration at a temperature level of >1,100 °C. Consequently, after removal of the PFC-loaded activated carbon from the filter vessels fresh activated carbon would have to be installed. These requirements result in significant costs that significantly negatively impact the overall cost of a PFC water purification process.

The PerfluorAd method, on the other hand, is designed to minimize the amount of active ingredient used and thus the amount of waste generated, thereby enabling the best possible life cycle assessment while minimizing the costs associated with properly performing high temperature incineration of PFC-contaminated residues arise. In particular, from a combination of conventional adsorption processes (powdered activated carbon or granular activated carbon) with the PerfluorAd process, possibilities for optimizing the water purification process, the required amount of active substances, the amount of waste produced, and the resulting overall costs are opened up.

Figure 7 shows by way of example that the pollutant-active substance ratios obtainable with the PerfluorAd process (generally referred to as loading in the case of the use of adsorbents) are more favorable than is known from the use of conventional adsorbents. As has already been stated, the amount of active ingredient used thereby falls significantly lower. A generally binding specification of specific amounts of active substance is not possible because the demand for active ingredients depends

significantly on the pollutant compositions and their concentrations as well as the organic water matrix and above all on the cleaning objectives to be achieved.

The resulting in the desired precipitation process residues initially incurred in the form of a thin sludge with low dry matter content of about 1-2%, by means of gravity drainage to about 4-6% and with a possibly adjoining technical apparatus for drainage optimization to a dry matter content of up to > 30%.

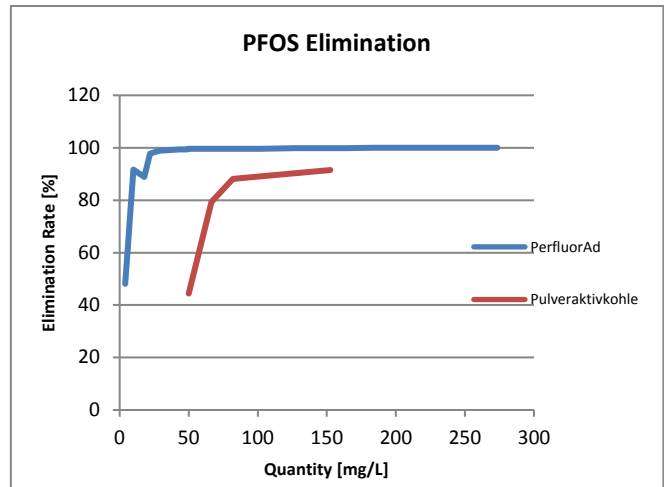


Figure 6. Comparison of the elimination rates for the individual PFC substance (perfluorooctane sulfonic acid) when using PerfluorAd and powdered activated carbon

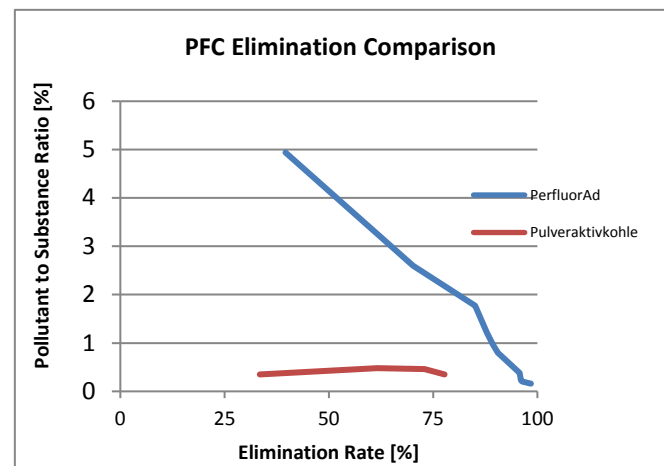


Figure 7. Comparison of the substance-to-substance ratio with the achievable PFC total elimination rate using PerfluorAd and powdered activated carbon.

3. Cost Considerations

Before selecting a water purification process, a process assessment of the technically potentially suitable process shall be carried out. In connection with the treatment of PFC-contaminated waters, a further assessment based on orienting pilot plant tests as well as semi-technical pilot tests is recommended.

Furthermore, a business assessment of the candidate procedures is to be made. For this, the future user of the technology should use identical evaluation criteria for each method to be considered. Information from the technology providers and / or empirical values from comparable applications can be used as a guide. However, these should not be unconditionally adopted without project-related validation.

At this point, a complete discussion of all influencing variables cannot be carried out. The following explanations are therefore not a definitive catalog of all the cost parameters to be taken into consideration, but an enumeration of essential criteria. In addition to the costs for the purchase or use of the equipment (for example in the form of rental or leasing models or other services), the operating costs in particular should be considered in the context of a decision-making process. These are (among others) cost parameters to consider:

- Procedural support and operation of the system
- Maintenance
- Resources (delivery costs for activated carbon, flocculants, flocculants, oxidizers, etc.)
- Energy and other media
- Other auxiliaries (filter sand, catalyst masses, media, etc.)
- Take back, recovery or disposal of residues
- Fees and other fees (sewer discharge introduction, land use, etc.)

The most commonly used fixed-bed adsorption processes currently used for the purification of PFC-contaminated waters are, in particular, the costs arising from the supply and disposal of the adsorbent material. In addition, there are also other residue fractions that could be PFC contaminated - such as e.g. iron hydroxide sludge, etc. - for proper disposal. Since perfluorinated compounds are considered to be persistent under natural conditions, the handling of PFC-contaminated residues is being handled increasingly stricter (see paragraph 2.4). As a currently acceptable disposal method for PFC-contaminated residues combustion at a high temperature level applies. The disposal costs incurred in a high-temperature combustion affect the

operating costs of the water purification process to a considerable extent. Specific disposal costs of ≥ 500 Euro/ ton plus handling and transport have to be estimated. Furthermore, the re-procurement of burnt adsorption materials must be included in the operating costs.

From the use of containers for the storage of PFC-containing extinguishing agent is known that even after diligent cleaning of the container impurities may remain. It was repeatedly observed in vessels that were previously used by fluorine-containing extinguishing agent, showed PFC loads. In order to avoid similar effects, some authorities expect a "carryover-free shipment" without PFC contaminated residues", e.g. by co-incineration of the transport containers (e.g. IBCs - Intermediate Bulk Containers) in high temperature incineration. Relevant requirements and the resulting costs must be taken into account in the collection of operating costs and also be included in the decision-making for the treatment technology.

Taking into account the growing demands with regard to the careful handling of waste contaminated with PFC, it is advisable to place the greatest emphasis on minimizing the use of auxiliary agents and the resulting formation of residues in the process selection process.

4. Summary and Outlook

Perfluorinated and polyfluorinated chemicals (PFCs) are often cited in the context of trace substances, but their routes to environmental release are much more diverse. Furthermore, concentrations of groundwater contaminants are found to be much higher than those of the trace substance level. In connection with PFC, science and practice have many questions, such as: in the field of trace substance analysis, the human toxicological classification of many individual substances, the assessment of the mass transport in aquifers and much more. In particular, the removal of PFC from aqueous media presents a major challenge.

With the PerfluorAd process, a new PFC-specialized 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 PerfluorAd process is based on the principle of precipitation and uses liquid active ingredients, which are selected depending on the PFC load and the water matrix and dosed into the water flow. The precipitation process is carried out in a classic stirred tank. The resulting precipitation products are

separated by sedimentation and/or filtration techniques.

In contrast to fixed-bed adsorption - especially in highly polluted waters - the process offers advantages through a low use of active ingredients and a resulting low accumulation of PFC-

contaminated residues. In the PerfluorAd process, both organic and inorganic background pollution of the water can be addressed simultaneously. Thus, the process shows a significantly lower sensitivity for complex impurities than is known in the application of conventional adsorbents.

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Abbreviations (in the order of appearance)

PFT	Perfluorinated surfactants
PFC	Perfluorinated and polyfluorinated chemicals
PFAS	Perfluorinated and polyfluorinated alkyl substances
AFFF-Foam	Aqueous Film Forming Foam (water-film-forming foaming agent)
PFBS	Perfluorobutane sulfonate
PFHxS	Perfluorohexane sulfonate
PFBA	Perfluorobutanoic acid
PFPeA	Perfluoro-n-Octanoic acid
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctanesulfonic acid
AOF	Adsorbable organically bound fluorine
DOC	Dissolved Organic Carbon
PAK	Powdered activated carbon
IBC	Intermediate Bulk Container (Plastic containers with up to one cubic meter content)

Author:

Dipl.-Ing. M.Sc. Martin Cornelsen

Email: cornelsen@cornelsen-umwelt.de

Cornelsen Umwelttechnologie GmbH

Graf-Beust-Allee 33

45141 Essen