

COMBINATION OF ULTRAFILTRATION (UF) AND POWDERED ACTIVATED CARBON (PAC) TO REMOVE MICROPOLLUTANTS, ANTIBIOTIC RESISTANT BACTERIA AND PHOSPHORUS FROM WASTEWATER

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ABSTRACT

Nowadays the high usage of pharmaceuticals like antibiotics, analgesics or X-ray contrast agents for human and veterinary treatment leads to increasing concentrations in municipal wastewater and in the aquatic environment. Apart from micropollutants, the global concern regarding the threat of antibiotic resistant bacteria (ARB) in the environment is growing. Due to a 40 % increase in global consumption of antibiotics over the last decade, combating antibiotic resistances represents a complex challenge. Municipal and industrial wastewater, which is collected and treated in wastewater treatment plants (WWTPs), harbours micropollutants and various bacteria from environmental, human and animal origins. Despite the removal of such substances during the biological treatment process of WWTPs, a significant amount still enters the aquatic environment by discharging treated wastewater into surface water bodies. Aside from being a source for the distribution of micropollutants and ARB to the aquatic environment, WWTPs represent a main source for the expulsion of nutrients, especially phosphorus, to surface water bodies. The accumulation of nutrients can lead to eutrophication and thus accelerated aquatic plant and algal growth, which causes a qualitative degradation of surface water bodies.

To achieve a sufficient removal of micropollutants, ARB and phosphorus by WWTPs, an additional treatment step is required. One example of this additional treatment step is the "BIO-CEL® Activated Carbon process", a combination of Powdered Activated Carbon (PAC) usage with submerged ultrafiltration membrane technology, developed by MICRODYN-NADIR. This hybrid process takes advantage of established methods (adsorption and filtration) and provides an option to meet higher effluent requirements of WWTPs. The BIO-CEL Activated Carbon process is located downstream the biological treatment step of conventional WWTPs and acts as an additional purification step before the discharge into receiving waterbodies.

Pilot plants with the BIO-CEL Activated Carbon process have been operational in North Rhine Westphalia (NRW) and Hesse, Germany since 2016. During this period a high removal efficiency of micropollutants (>80 %), especially for pharmaceuticals, was detected. Regarding the separation efficiency of germs and bacteria, a complete separation for E. Coli and a significant reduction of coliform bacteria (>5,2 log levels) was achieved. Due to extension of the BIO-CEL Activated Carbon process with post precipitation, dosage of a precipitant into filtration tank, effluent concentrations of <0,2 mg/l total phosphorus were monitored.

KEYWORDS

Ultrafiltration, Powdered Activated Carbon, submerged membranes, micropollutants, antibiotic resistant bacteria, nutrient removal (phosphorus)

Combination of Ultrafiltration (UF) and Powdered Activated Carbon (PAC) to remove micropollutants, antibiotic resistant bacteria and phosphorus from wastewater

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1. Introduction

Nowadays the high usage of pharmaceuticals like antibiotics, analgesics or X-ray contrast agents for human and veterinary treatment leads to increasing concentrations in municipal wastewater and in the aquatic environment. [1]

Apart from micropollutants, the global concern regarding the threat of antibiotic resistant bacteria (ARB) in the environment is growing. Due to a 40 % increase in global consumption of antibiotics over the last decade, combating antibiotic resistances represents a complex challenge. [2]

Municipal and industrial wastewater, which is collected and treated in wastewater treatment plants (WWTP), harbours various bacteria from environmental, human and animal origins. During the biological treatment process, a removal of micropollutants (up to 50 %) and ARB (up to 2-3 log levels) takes place. [3, 4] Despite the mentioned removal capability of conventional wastewater treatment plants, a significant amount of such substances still enters the aquatic environment by discharging treated wastewater into surface water bodies. Therefore, wastewater treatment plants play a major role regarding the removal, separation and distribution of micropollutants and ARB into receiving waterbodies.

To optimize WWTP for the removal of micropollutants (>80 %), additional treatment processes (so called fourth treatment steps) have been implemented in several plants in Central Europe. [5, 6, 7] With currently established treatment technologies like ozonation with subsequent sand filtration, granulated activated carbon filtration and powdered activated carbon filtration followed by sand filtration, an efficient reduction of micropollutants can be achieved. [5, 8, 9] Nevertheless, a complete separation of ARB and PAC cannot be guaranteed with these technologies.

Although there are currently no legal obligations to eliminate micropollutants, events such as the German Stakeholder Dialog "Spurenstoffstrategie des Bundes" in March 2019 show that legal regulations can be expected shortly.

Aside from being a source for the distribution of micropollutants and ARB to the aquatic environment, WWTPs represent a main source for the expulsion of nutrients, especially phosphorus, to surface water bodies. The accumulation of nutrients can lead to eutrophication and thus accelerated aquatic plant and algal growth.

According to publications of the German Environment Agency (Umweltbundesamt) in 2017, excessive phosphorus concentrations were measured at over 65% of all monitoring sites. [10] Due to the national implementation of the European Water Framework Directive (EU Directive 2000/60/EC) and thereby the achievement of a

good ecological status of all surface waterbodies, existing regulations of the German wastewater directive (AbwV) for the discharge of phosphorus are being tightened significantly. [11]

In Germany, several states (e.g. Hesse) released a program of measures (2015 - 2021) to upgrade, extend and optimize municipal WWTPs to reduce the discharge of phosphorus. Depending on the category of the water body (surface water with phosphorus induced deficits) and the size of the WWTPs (10.000 - >100.000 p.e.), requirements of approx. 0,2 mg/l total phosphorus (P_{tot}) concentration in the effluent were set. [12]

Total phosphorus values of 1,0 - 0,5 mg/l in the effluent of conventional WWTPs can be achieved by combining treatment processes like biological phosphorus removal (BIO-P), increased precipitant dosage or two-point precipitant dosage (dosing of precipitant into primary sedimentation and biological treatment step). [13 ,14] For a further reduction of total phosphorus <0,5 mg/l, an additional treatment step, based on filtration and dosing of a precipitant must be implemented. [12]

One example of this additional treatment step is the “BIO-CEL[®] Activated Carbon process”, a combination of Powdered Activated Carbon (PAC) usage with submerged ultrafiltration membrane technology, developed by MICRODYN-NADIR. This hybrid process takes advantage of established methods (adsorption and filtration) and provides an option to meet higher effluent requirements of WWTPs, for example the removal of micropollutants, complete separation of bacteria and germs and post precipitation to reduce total phosphorus.

2. Material and method

Pilot plants with the BIO-CEL Activated Carbon process have been operational in North Rhine Westphalia (NRW) and Hesse, Germany since 2016. An important partner is the Emscher genossenschaft and Lippeverband (EGLV), the largest wastewater management company and operator of WWTPs in Germany, which provided sites for pilot testing.

The BIO-CEL Activated Carbon process is located downstream the biological treatment step of conventional WWTPs and acts as an additional purification step before the discharge into receiving waterbodies.

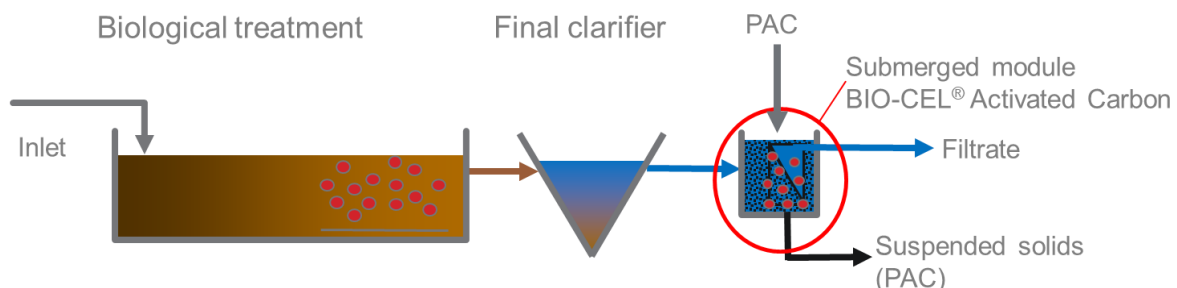


Figure 1: Schematic representation of the BIO-CEL[®] Activated Carbon process

The effluent of final clarifiers is used as feed for the process, in which the filtration tank comprises submerged BIO-CEL ultrafiltration membrane modules with a nominal pore size of 0.04 μm .

The main operation of the membrane modules involves repeated cycles of filtration followed by a short interval of relaxation and backwash where only aeration without filtration takes place. The membrane aeration is a critical part of the operation. The required turbulence on the surface of the membranes to remove accumulated solids is generated by air bubbles that arise from diffusers located underneath the membrane module.

Based on the feed volume, PAC is dosed directly into the filtration tank. By generating a suction with the permeate pump (negative pressure), treated wastewater flows through the membranes and is discharged as permeate. PAC with adsorbed micropollutants, particles, germs and ARB is retained by the ultrafiltration membrane. To guarantee a constant concentration of total suspended solids in the filtration tank, PAC is discontinuously deducted and pumped back into the biological treatment step.

Pilot plants can be operated with a net flux of 23 - 31 l/m²/h without a significant drop of permeability. In comparison to membrane bioreactor (MBR) applications, the applied concentration of suspended solids in the filtration tank could be significantly reduced. Chemical cleaning with sodium hypochlorite was executed in a two-week interval. Depending on the composition, it may be necessary to clean with citric acid.

Monitoring micropollutants

In addition to monitoring standard operational parameters, such as air flow, temperature, pH, permeability, permeate flow and transmembrane pressure, measurements regarding the elimination capacity of micropollutants were completed. PAC concentrations between 5 – 20 mg/l have been investigated to monitor the elimination capacity. The nominal grain size of commonly used PAC varies between 10 – 150 µm. [15] The PAC used for the pilot trials had a nominal grain size of the 15 µm.

The elimination performance of the BIO-CEL Activated Carbon process was assessed based on the following indicator substances:

- pharmaceuticals (diclofenac, carbamazepine, metoprolol, sulfamethoxazole, propranolol)
- X-ray contrast agents (amidotrizoate, lopamidol, lohexol)

Depending on the initial concentration of such substances, their relevance is not equal for each treatment plant. Further investigations for artificial sweeteners, corrosion inhibitors and other pharmaceuticals are still being measured in 2019/2020.

The measurements have been executed according to the recommendations of the German “Kompetenzzentrum Spurenstoffe Baden-Württemberg”, a cooperation between three independent partners: The University of Stuttgart and Biberach University of Applied Sciences and the Baden-Württemberg regional chapter of the DWA. To generate a 48-hours composite sample, two 24-hours composite samples were taken on two subsequent days. The samples were mixed in proportion to the daily amount of wastewater at the corresponding measurement site. The samples were taken from the feed (effluent of the final clarifier) and permeate (permeate tank). The list of substances to be investigated for the monitoring campaign were derived from the recommended range of substances to evaluate the removal efficiency of additional treatment processes. [16]

Apart from the analysis of micropollutants, the spectral absorbance coefficient at 254 nm (SAC_{254}) was considered a suitable parameter to obtain correlations between the attenuation of ultraviolet (UV) absorbance and the relative decrease of micropollutants by PAC. [17] With the UV absorbance measurement, the UV radiation that can pass through a sample of treated wastewater (for example) is detected. The absorbed amount of radiation gives a reference of the amount of substances in the sample. Dissolved organic matter and several organic micropollutants show aromatic compounds or double bonds, which absorb the UV radiation at 254 nm. [18, 19] Such aromatics can easily be oxidized by using ozone or adsorbed to activated carbon.

A reduction of SAC_{254} provides information about the existence of micropollutants in wastewater samples and is therefore used as a method of reference and detection. During the trials in NRW, SAC_{254} measurements were performed with samples from feed and permeate. To compare the removal efficiency of different WWTPs, the dissolved organic matter, which remains post biological treatment, related to the used PAC was quantified by measuring the dissolved organic carbon (DOC). Besides the SAC_{254} , the sum parameter DOC represents another parameter to evaluate the removal efficiency regarding micropollutants.

Monitoring of germs and bacteria

To determine the separation efficiency of the used ultrafiltration membrane, coliform bacteria and *E. coli* were measured with the IDEXX Colilert® - 18/Quanti-Tray® test. This method is accredited according to EN ISO 9308 - 2 by the Commission directive (EU) 2015/1787, which regulates the quality of water intended for human consumption. [20] The samples were taken with sterilized glass vessels (180 °C for ≥ 30 min and Ethanol ≥ 70 %). The withdrawal tap is connected to the permeate tank and was disinfected with Ethanol ≥ 70 % as well. Samples were taken 1 - 2 times per week.

Extended phosphorus removal

With the combination of ultrafiltration and post precipitation, dosage of a precipitant into the filtration tank of the BIO-CEL Activated Carbon process, particulate and dissolved phosphorus compounds can be removed from treated wastewater. Trials with the BIO-CEL Activated Carbon process and applied post precipitation were executed for more than 6 months with a pilot plant in Hesse. The applied precipitant for post precipitation was 40% iron (III) chloride. Additional trials with an aluminum-iron based precipitant are currently running in NRW. For the monitoring of orthophosphate ($PO_4\text{-P}$), a phosphorus compound that can be chemically precipitated, first laboratory analysis with vial tests were used. The limit of quantitation for $PO_4\text{-P}$ analysis is 0,05 mg/l. To obtain a wider spectrum of results, online analyzers were installed to take samples from feed and permeate.

Limitations that need to be achieved for the effluent concentration of total phosphorus were set to 0,4 mg/l P_{tot} for 2-hour composite samples and 0,2 mg/l P_{tot} for 24-hour composite samples. [10] The main design criteria for the post precipitation process are the average P_{tot} concentration in feed and P_{tot} target concentration in permeate. The input feed concentration was set by evaluating the P_{tot} concentration in the effluent of the final clarifier for one year, and then determining the arithmetic average. The basic design of the post precipitation was executed according to DWA-A 202 guideline of the German Association for Water, Wastewater and Waste (DWA). [21] The target P_{tot}

concentration to be achieved by applying post precipitation for the BIO-CEL Activated Carbon process was 0,1 mg/l P_{tot} . The calculated amount of precipitant was 5,7 g/l or 0,004 l/d.

3. Results

The results are derived from trials and experiments executed during the last two years in pilot plants in Hesse and NRW.

Removal Micropollutants

Results of the monitoring program to evaluate the removal efficiency for the BIO-CEL AC process regarding micropollutants are displayed in Figure 2 for concentrations of 10 and 20 mg/l PAC. The number of analyzed samples comprises 6 – 12 measurements, depending on the feed concentration.

For most of the investigated pharmaceuticals, except sulfamethoxazole, an elimination rate between 70 – 96 % could be achieved with the applied PAC concentration. An average elimination rate of 10 – 30 % confirmed the commonly known poor adsorption of X-ray contrast agents to PAC. Depending on the PAC concentration, significant fluctuations regarding the elimination of the antibiotic sulfamethoxazole have been detected.

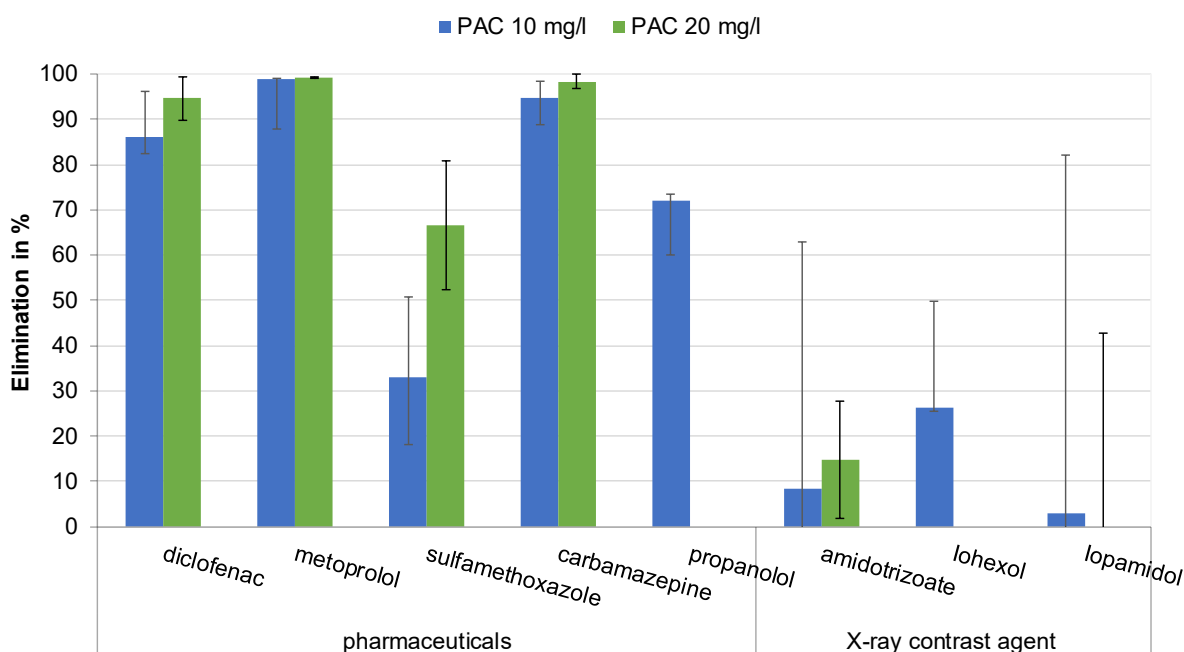


Figure 2: Elimination performance of investigated micropollutants

The required removal efficiency of WWTPs with implemented additional treatment to remove micropollutants is set to an annual average of 80 % for selected substances according to the DWA. [16] In Figure 2 displayed removal performance only refers to the BIO-CEL Activated Carbon process. To compare the process with the required removal efficiency of a WWTPs with implemented BIO-CEL Activated Carbon process the removal efficiency of the biological treatment step of WWTPs must be considered as well.

In addition to the analysis of micropollutants, the SAC₂₅₄ reduction and the DOC were monitored for the same period.

Depending on the investigated micropollutants, a difference in the correlation of ultraviolet (UV) absorbance attenuation and the reduction of the micropollutants was expected.

Full-scale implementations of the PAC process in combination with sand filtration and a sedimentation tank showed a reduction of the SAC₂₅₄ by 40 % and a correlated removal of the micropollutant diclofenac by 80 %. [22] With the BIO-CEL Activated Carbon process, similar results were achieved. Diclofenac showed a reduction of SAC₂₅₄ by 31 % and a removal efficiency between 83 – 94 % depending on the set PAC concentration in the filtration tank (see Figure 3).

Corresponding to a SAC₂₅₄ reduction of 30 %, a constant removal capacity for the pharmaceuticals, carbamazepine and metoprolol of approx. 90 % was detected.

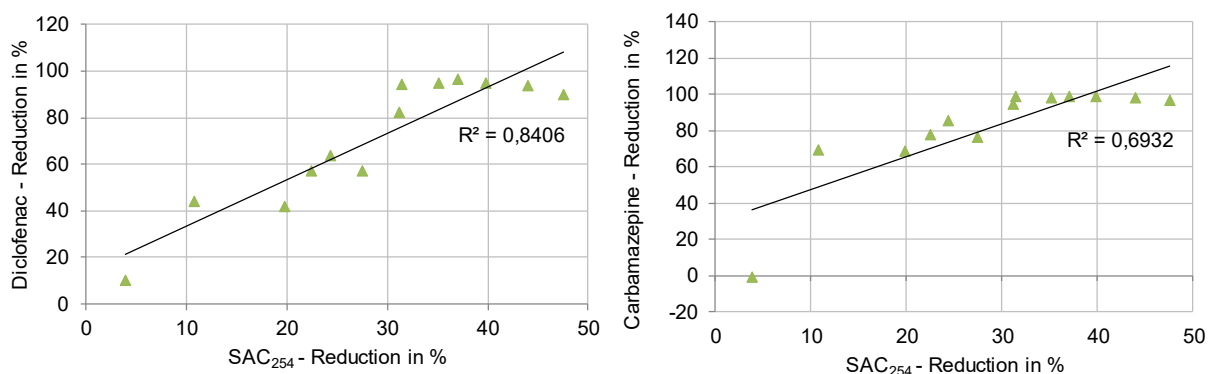


Figure 3: Correlation of SAC₂₅₄ reduction with removal capacity of diclofenac and carbamazepine

For X-ray contrast agents amidotrizoate, lopamidol and lohexol, no correlation with a SAC₂₅₄ reduction could be derived.

Based on the evaluation of the SAC₂₅₄ reduction and the removal efficiency of micropollutants, an indication for the application of the SAC₂₅₄ to regulate full-scale PAC process was derived. Due to the missing correlation of the SAC₂₅₄ reduction with the removal capacity of X-ray contrast agents, the SAC₂₅₄ should preferably be applied to control treatment processes with PAC concerning pharmaceuticals like diclofenac, carbamazepine, metoprolol and sulfamethoxazole.

Besides the evaluation of the SAC₂₅₄ reduction, the reduction of DOC was considered. Figure 4 shows a correlation between the reduction of DOC and SAC₂₅₄ depending on the applied PAC concentration. By increasing the PAC concentration from 10 mg/l to 20 mg/l, a reduction of DOC by 10 % and SAC₂₅₄ by 17 % was detected. The check plot sample represents the elimination capacity of the ultrafiltration membrane without dosing PAC.

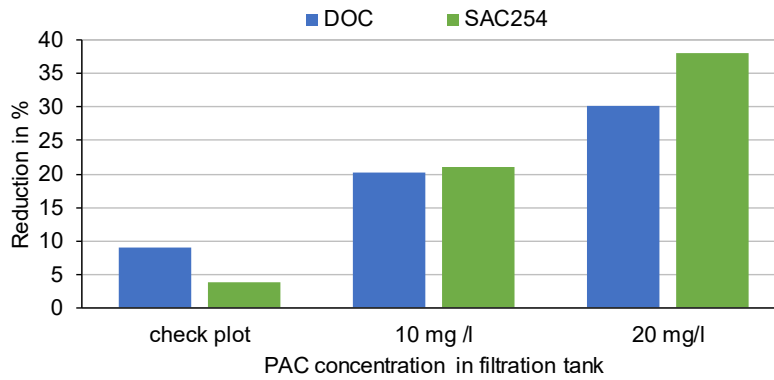


Figure 4: Reduction of DOC and SAC₂₅₄ for 10 and 20 mg/l PAC.

The displayed results match several investigations with activated carbon in pilot and full-scale plants in Germany and Switzerland, where the SAC₂₅₄ was set as a suitable surrogate parameter for process monitoring. [23]

Separation of germ/bacterial

Considering the ongoing trials in NRW, the separation efficiency of the ultrafiltration membrane regarding coliform bacteria and E. coli was monitored. Depending on the feed concentration, the reduction of coliform bacteria of about 5,2 log levels was determined. For E. coli was a complete separation, depending on the feed concentration reduction >4,5 log levels, detected. The separation efficiency lies within the range of the recommendations for water reuse published by the WHO of min. 3 – 5 log levels reduction of indicator microorganism (as E. coli) [24]

Extended removal of phosphorus

The calculated amount of precipitant according to the DWA-A 202 guideline, 0,004 l/d, had to be increased by 25% during the period of trials. [21] Figure 5 shows the measured PO₄-P and P_{tot} concentrations that were detected by dosing precipitant for 6 months. The average concentrations in feed and permeate are displayed in Table 1. To achieve the target concentration of <0,2 mg/l P_{tot} it is important to reduce the PO₄-P concentration in permeate as much as possible. Relatively high P_{tot} concentrations >0,1 mg/l in permeate, e.g. in 23. March and 4. April resulted from an insufficient dosage of precipitant and the available, not precipitated, PO₄-P in permeate. With an automatized PLC (programmable logic controller) controlled dosing system such P_{tot} peaks could easily be avoided.

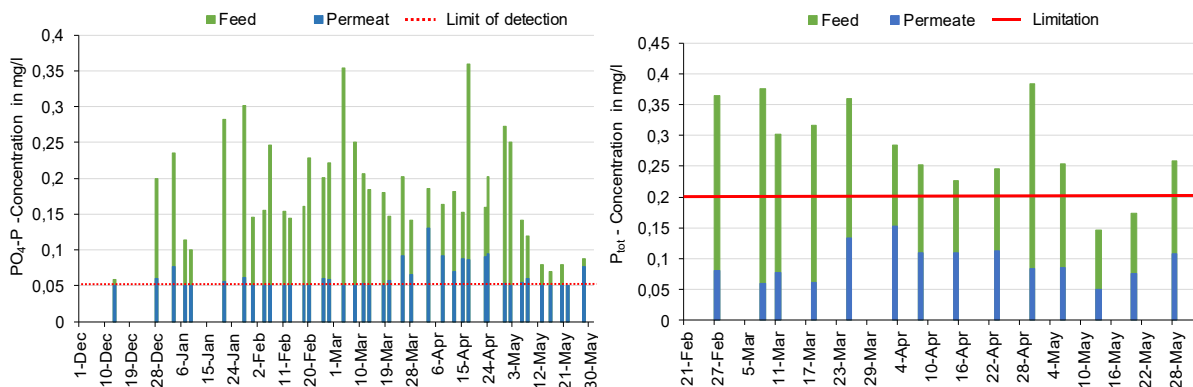


Figure 5: Measured P_{tot} and PO₄-P-concentration in feed and permeate

Table 1: Average P_{tot} and $PO_4\text{-P}$ concentrations

Parameter	Orthophosphate ($PO_4\text{-P}$) in mg/l	Total phosphorus (P_{tot}) in mg/l	Fractioning in %*	
			$PO_4\text{-P}$	Particulate P
Feed	0,18	0,30	59	41
Permeate	0,06	0,09	77	23

*Dissolved content, which is not available for precipitation (approx. 0,05 mg/l phosphonate) were not considered.

The average P_{tot} concentration in permeate shows that the limitations of $<0,2$ mg/l P_{tot} for WWTPs in a scale of 10.000 - >100.000 p.e. were permanently complied during the whole period of investigation. Furthermore Figure 6 and Table 1 present that due to the ultrafiltration membrane a significant amount of particulate phosphorus was retained.

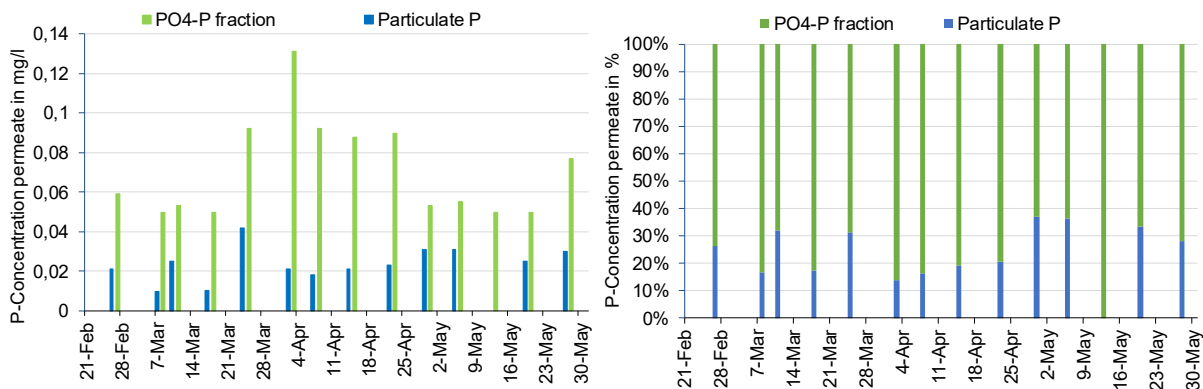


Figure 6: Fractioning of phosphorus components in permeate

With the combination of post precipitation, ultrafiltration and a controlled dosing system, P_{tot} concentration $< 0,1$ mg/l in the effluent of WWTPs could be attained and therefore the threshold according to the German wastewater charges act (AbwAG). [25] By discharging such low P_{tot} concentration to surface waterbodies a reduction of sewerage charge can be obtained.

Further trials to verify the yet obtained results and to test different types of precipitants are still running and scheduled for 2020.

4. Conclusion

Based on the results of a three-year pilot scale operation of the BIO-CEL Activated Carbon process, ultrafiltration membrane treatment in combination with PAC and post precipitation represents a promising technology for the removal of micropollutants, germs/bacteria and phosphorus from treated wastewater effluents. The expected implementation of significant stricter phosphorus discharge limitations of WWTPs, to improve the overall quality of surface waterbodies, bears another opportunity for the application of the BIO-CEL Activated Carbon process.

The following knowledge was gathered with the pilot scale trials:

- High removal efficiency of micropollutants (>80 %), especially for pharmaceuticals
- SAK_{254} sum parameter is suitable to control and monitor treatment processes with PAC

- Due to the dosage of precipitant into the filtration tank P_{tot} concentrations $<0,2$ mg/l were achieved. By implementing a controlled dosing system effluent concentration of 0,1 mg/l can be realized.
- With the used ultrafiltration membrane, a complete separation of E. coli was detected. Coliform bacteria were, depending on the feed concentration, reduced by 5,2 log levels.

Regarding the retention efficiency of PAC (15 μm nominal grain size), with adsorbed micropollutants, a complete separation due to the ultrafiltration membrane (pore size 0,04 μm) is expected. In comparison to the retention efficiency of established treatment processes, like ozonation with subsequent sand filtration, granulated activated carbon filtration and powdered activated carbon filtration followed by sand filtration, the complete separation of used activated carbon with the BIO-CEL Activated Carbon process presents a mentionable advantage. [15]

An economical evaluation of the BIO-CEL Activated Carbon process based on annual costs in relation to the amount of treated wastewater showed specific costs between 0.09 – 0.12 €/m³ for WWTPs in scale of 10.000 - >100.000 p.e. The extension of the BIO-CEL Activated Carbon process with the process component post precipitation results in an increase of specific costs by approx. 3 %. In scientific literature, specific costs of established processes for the removal of micropollutants are indicated with 0.10 – 0.25 €/m³ depending on the considered treatment process. [26]

In summary, the BIO-CEL Activated Carbon process represents an alternative solution for advanced wastewater treatment, with which not only micropollutants and germs can reliably be removed from treated wastewater, but also a significant reduction of phosphorus can be achieved.

From an economical point of view, the BIO-CEL Activated Carbon process offers a cost-effective and competitive solution.

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