Received: 12 March 2020 | Revised: 30 November 2020 | Accepted: 19 December 2020

DOI: 10.1002/ep.13587

REMEDIATION TREATMENT

ENVIRONMENTAL PROGRESS & SUSTAINABLE ENERGY

Removal of emerging micropollutants from wastewater by nanofiltration and biofilm reactor (*MicroStop*)

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Funding information
Hamburg Wasser: Company of municipal Wastewater Treatment; Rosa-Luxemburg Foundation

Abstract

Current wastewater treatment is not designed for the elimination of micropollutants (MP) and therefore these are released into the aquatic environment by means of the municipal wastewater. The *MicroStop* project investigates a combination of a fixed-bed reactor (FBR) and a nanofiltration plant (NF) for the elimination of organic MP. NF acts as a barrier for MP while biological degradation of the pollutants is to be induced in the FBR. A simultaneous treatment of the retentate should be realized by its return to the FBR. The NF is to be supplied with energy by a large separation of organic matter in the primary clarification. The experiments showed that NF can eliminate more than 95% of the selected and detected MP on average, and therefore, this method represents a suitable barrier for these pollutants. The retentate of the NF was used to test the potential of the biological degradation in the FBR. The test results showed that a significant biological degradation of different MP took place in the FBR. The degradability of the substances could be verified by not detecting significant adsorption on sludge and fixed-bed. For certain MP (sulfamethoxazole and carbamazepine) only little potential of biodegradation could be detected, which is confirmed by values from the literature. Apart from these two substances, the average degradation rate was 78%. The successful MP-retention in connection with the biological degradation rates demonstrated the potential of the concept.

Statement of Industrial Relevance: It is undisputed that the conventional wastewater treatment has to be upgraded, since it is not able to eliminate residual MP. The following describes how the *MicroStop*-process is able to address MP where competitive processes such as activated carbon or ozonation fail to do so:

- The process is able to eliminate MP comprehensively and to retain microplastics, pathogenic germs and metabolites to a large extent before they reach the environment.
- The retentate, which normally occurs as residue of the NF, is directly treated in the FBR. The only residue of the *MicroStop*-process would be the excess sludge, which could easily be used for sludge treatment.
- The effluent of the process is of high quality. This could be used in other regions of the world for irrigating plants, for industrial processes or even as tap water.

Abbreviations: FBR, fixed-bed (biofilm) reactor; MP, micropollutant(s); MWCO, molecular weight cut-off; NF, nanofiltration; TMP, transmembrane pressure.

- The potentially increased energy consumption (i.e., due to NF) is taken into account in our concept by the carbon extraction in the primary treatment.

Novelty or Significance: The combination of NF with biological degradation of MP in a FBR represents a process that has not yet been investigated in this form.
In addition, the combination with an elevated carbon extraction in the primary treatment (*Powerstep*-process) represents the idea of a complete redesign of conventional municipal wastewater treatment plants.

KEYWORDS
biodegradation, biodegradation micropollutants, bioreactor, innovation wastewater treatment plant, municipal wastewater, nanofiltration, retentate treatment, wastewater reuse, wastewater treatment

1 | INTRODUCTION

Various ecotoxicological effects are attributed to emerging micropollutants (such as active pharmaceutical ingredients, hormones, X-ray contrast media, pesticides, and microplastics) in surface waters. For example, the effect of antibiotics in surface waters is much discussed in terms of their potential role in the development of multi-resistant germs.[1,2]

Since conventional mechanical-biological wastewater treatment is not designed to effectively eliminate micropollutants (MP), municipal wastewater treatment plants are one of the most significant pathways of MP into surface waters.[3]

There are potential extended treatment stages of wastewater treatment plants for the elimination of MP (e.g., ozonation or activated carbon adsorption). These treatment stages have various disadvantages such as, the formation of dangerous transformation products (during ozonation) or the lack of elimination of multi-resistant germs and microplastics (during activated carbon adsorption).[2]

Intensive research is being carried out at our institute for more than 2 years in the field of MP-elimination from municipal wastewater (*MicroStop* research project). The experimental plant consists of a FBR (biofilm reactor) combined with a NF plant. Answers on our research question, to what extent MP are degraded in FBR, cannot be found in the literature so far. The NF acts as a barrier to protect the environment from MP and to increase the concentration of these substances in the FBR. This barrier is necessary to allow an all-encompassing degradation. The combination of these two methods represents a novel concept.

This combined treatment process enables an all-encompassing MP elimination, which would not be possible with a conventional 4th treatment stage (ozonation or activated carbon). Thus, in addition to pharmaceuticals, multiresistant germs or microplastics are completely eliminated. The quality of the outlet of this treatment concept would allow a water reuse (i.e., infiltration, agriculture, industry, in arid and semi-arid regions also for direct or indirect reuse). At the same time, there would be no other residues apart from the sludge, which could be utilized in its classical treatment.

2 | OBJECTIVE AND CONCEPT OF THE OVERALL SYSTEM

2.1 | General Idea

The aim of the concept is not to add a fourth purification stage to a conventional wastewater treatment plant, but to reinvent it. The long-term project objective is to develop a wastewater treatment concept that purifies the wastewater at a reasonable cost in such a way that it is free from MP, microplastics, multi-resistant germs, and particles of any kind. Additionally, phosphates and nitrogen are to be largely eliminated. Through the combination of an elevated carbon extraction (chemical oxygen demand [COD]) in the primary treatment, a quantity of energy can be produced that recovers a part of the overall process. The flow chart of the system is shown in Figure 1.

2.2 | Description of Single Steps

2.2.1 | Primary treatment and carbon extraction

The wastewater reaches the primary treatment, where flocculation (with iron and/or cationic polymer) is carried out to reduce the COD content and to induce an increased carbon extraction. The aim is to produce significant amounts of energy through the sludge treatment/biogas production (together with the excess sludge of the bioreactor), to provide energy for the overall treatment process. This extra gain in energy could be used for the relatively energy intensive NF process. This concept has been used similarly in the newly designed treatment plant model *Powerstep* (powerstep.eu), whose potential has already been proven at various wastewater treatment plants in Europe.[4]

Furthermore, an aeration tank in our concept is not provided, in addition to the lack of elimination of MP, the appearance of larger amounts of nitrogen in the process is a problem. For this reason, the *MicroStop* project will also investigate the extent to which nitrogen decomposition (i.e., in the biofilm reactor) is possible or whether an extension of the process can be used to reduce nitrogen loads.[4,5]

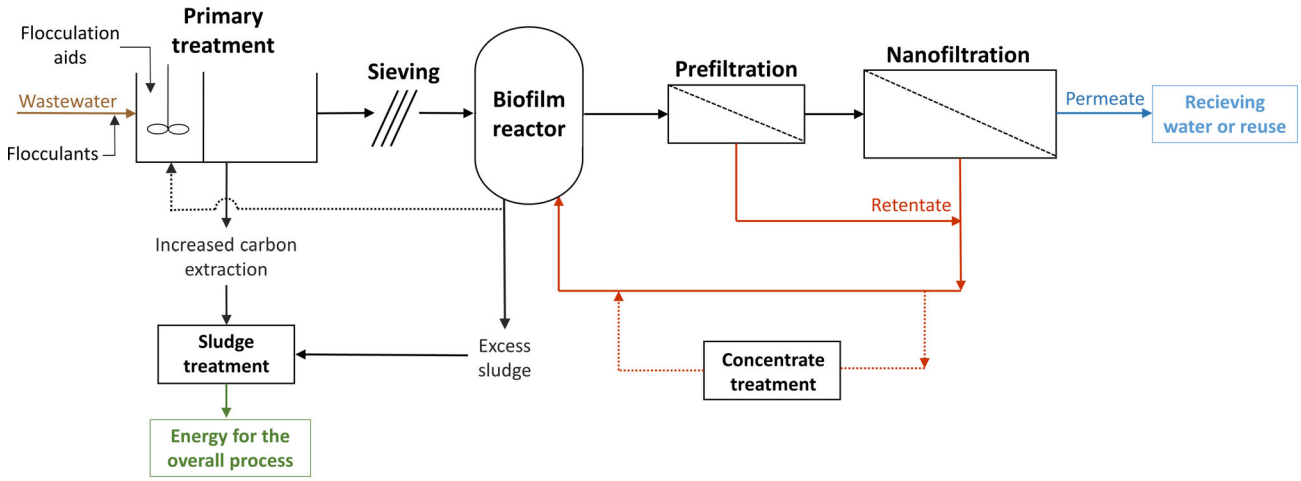

FIGURE 1 Flow chart for the technical implementation of the nanofiltration in combination with the biofilm reactor and preceding primary treatment (project *MicroStop*) [Color figure can be viewed at wileyonlinelibrary.com]

2.2.2 | Biofilm reactor (biodegradation) and NF

After the primary treatment and a secondary sieving (2 mm) the wastewater enters the biofilm reactor (in this case a FBR; see Section 3.1). Subsequently, the wastewater continues through the prefiltration to the NF (detailed description in Section 3.2). The recovered permeate can be introduced into the effluent of the treatment plant, while the retentate (concentrate) is returned to the biofilm reactor. By returning the retentate to the reactor, the concentration of MP is increased. By this measure, it is expected to have an achievement of the induction-concentration and, therefore, a transformation of the food spectrum of the bacteria. As a result, a large part of the MP is expected to be biodegraded.

2.2.3 | Excess sludge and concentrate treatment

The excess sludge of the reactor will be delivered to the sludge treatment/biogas production. Next, it is to be examined whether the sludge from the reactor has an increased adsorption potential, which could reduce the number of flocculants used in the preliminary clarification.

If necessary, the retentate flow can be extended by a concentrate treatment (e.g., ozonation) in order to eliminate substances which could not be biologically degraded in the reactor.

2.2.4 | Treatment of phosphorous and nitrogen

A membrane should be selected which acts as a barrier to phosphorus. This could also enable its recovery. Fixed-bed (biofilm) reactors are considered to have high potential for nitrogen elimination.[6,7] Methanol or biodegradable polymers, for example, could possibly adjust the C/N ratio in the reactor to achieve higher nitrogen degradation rates.[8,9]

3 | MATERIAL AND METHODS

The performance of the elementary components of the overall concept presented in Section 2 are examined in practical experiments: The NF (first operation in January 2018) and the FBR (biofilm reactor; continuous operation since June 2018). Currently, these plants are operated separately. The NF is fed with the effluent of a wastewater treatment plant, while its retentate is used as the influent of the FBR. The long-term aim is to combine the two plants with continuous operation.

3.1 | Fixed-bed reactor (biofilm reactor)

Two FBR (see Table 1 and Figure 2) are operated as batch experiments independently of each other. The identically operated FBR differ by their filling materials and their reactor volume (see Table 1). The difference in volume (liquid phase) depends primarily on the higher density of the clay material. The substrate consists of the retentate produced by NF, which is pumped into the sump of the FBR by means of a centrifugal pump. From the head of the FBR, the substrate flows back into the recirculation flask, which serves as a buffer. To start the plant, activated sludge from a wastewater treatment plant was added as inoculum (ratio 1:20). In addition to the NF retentate, the reactors were fed with raw wastewater in the first trial month.

3.2 | Nanofiltration (NF)

In the NF (see Table 2 and Figure 3), the medium from the feed container is fed to the cylindrical spiral winding module via a two-stage prefiltration consisting of two polypropylene candle filters (20 and 1 μm pore size).

The wastewater (effluent of a wastewater treatment plant with conventional structure) is introduced at one end of the module and flows axially through. The permeate passes through the membrane to the center of the module and discharges on the other side. The permeate is collected while the retentate is returned as feed.

3.3 | Methods of analysis

The elimination of MP is measured by the Central Laboratory of the TUHH using previously selected, representative, lead substances using HPLC-MS/MS (high-pressure liquid chromatography: Model *1260* from *Agilent*; triple-quad mass spectrometry: *5500 Q TRAP* from *Sciex*).[11] Table 3 shows the parameters of the HPLC method.

The detected MP and their detection limits are listed in Table 4. Various circumstances were considered when selecting the MP (as lead substances): Insufficiency of conventional wastewater treatment plants, ozonation and activated carbon (substances that are not fully eliminated by these processes), ecotoxicity, rejection efficiencies of the NF in terms of molecular size and charge. The selection was also based on the attribution of problematic substances by German and Swiss institutions.[12,13] These following substances were measured, but could not be detected: Ibuprofen, Mecoprop, Tricolsan, Cyclamate, and Glyphosate.

TABLE 1 Operating parameters fixed-bed reactors

Parameter		Reactor 1 + 2
Inner diameter		0.1 m
Upstream-velocity		15 m/h
Flow rate inflow		100–200 L/h
pH-value		6–8
Aeration		10–20 L/h
Dissolved oxygen		>6 mg/L
Parameter	**Reactor 1**	**Reactor 2**
Height of the bed	1.15 m	1.35 m
Volume reactor (liquid phase)	2.8 L	4.7 L
Porosity	0.14	0.22
Filling material (type and appellation)	Swelling clay; *Liapor GmbH & Co. KG*; 8 4/8	Polyethylene carrier; *Stöhr GmbH & Co.KG*; Hel-X HXF12KLL
Filling material (shape and size)	Spherical, 4–8 mm	Hexagonal, 12 × 12 mm

TABLE 2 Operating parameters nanofiltration

Parameter	Value
Operating pressure (TMP)	4–6 bar
Operating temperature	18–32°C
pH-value	6–8
Feed volume flow	300–500 L/h
Permeability	10–12 L/(h × m^2 × bar)
Recovery (=permeate flow/feed flow[10])	10–40%
Type of membrane	NF270-2540 *DOW FILMTEC™*
Active membrane surface	2.6 m^2

Abbreviation: TMP, transmembrane pressure.

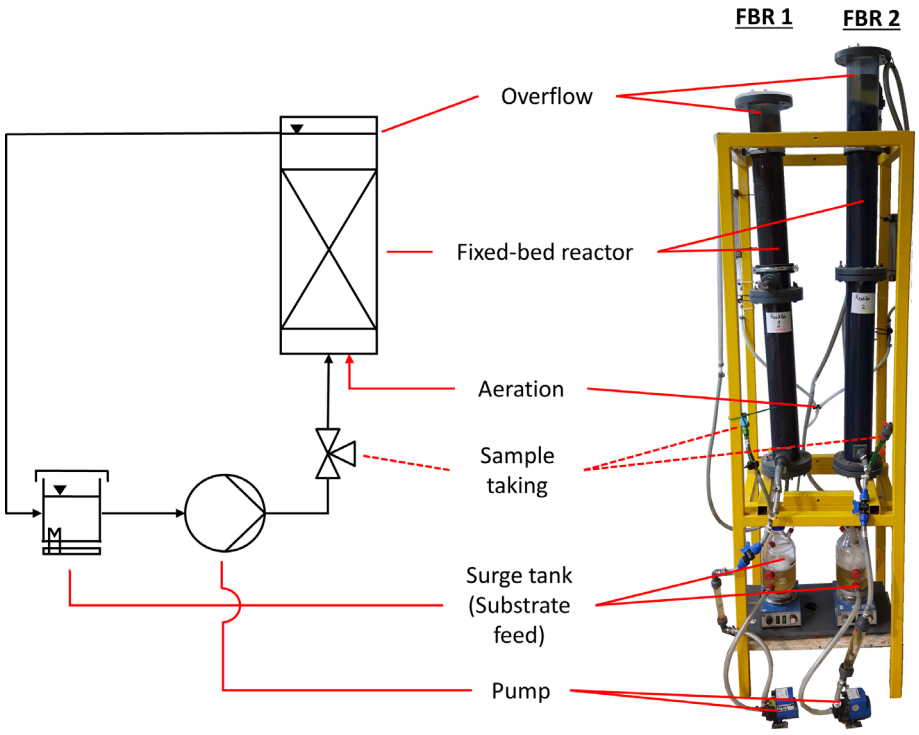

FIGURE 2 Flow chart and photo of fixed-bed reactors [Color figure can be viewed at wileyonlinelibrary.com]

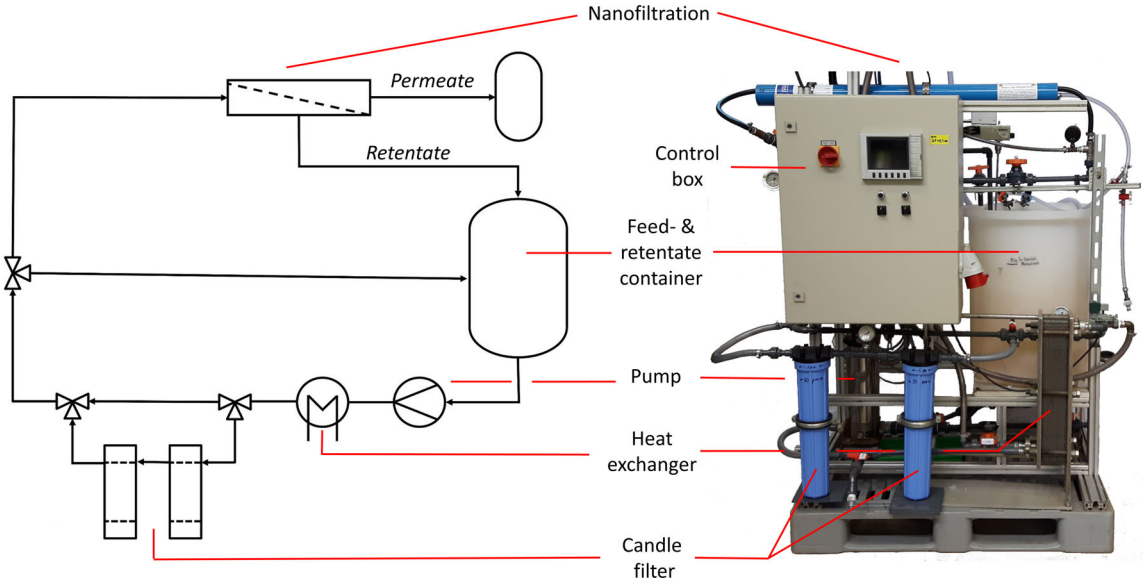

FIGURE 3 Flow chart and photo of nanofiltration [Color figure can be viewed at wileyonlinelibrary.com]

TABLE 3 Parameters of the HPLC method for seperating samples[11]

Type of method	Gradient method (15 min)
Column	Phenomenex Synergi Hydro RP, 150 × 3 mm, 4 µm
Eluent A	96% H_2O + 1 mmol ammonium format +0.05% formic acid
Eluent B	4% acetonitrile/methanol (50/50) + 0.05% formic acid
Eluent C	95% acetonitrile/methanol (50/50) + 0.05% formic acid
Flow	350 µL/min
Injected volume	50 µL
Column temperature	40°C

Abbreviation: HPLC, high-pressure liquid chromatography.

3.4 | Methods of evaluation

If the MP could not be detected in the samples, the detection limit (see Table 4) was assumed for the calculation. All values listed in the results are mean values while the standard deviation *N* is indicated by error bars.

3.4.1 | Fixed-bed reactor

The MP concentration in the reactors was determined after feeding (c_{start}). Thereupon, a sample was taken 14 days later (c_{end}) to calculate its removal through degradation during this time. The biological degradation was determined by the following equation[14]:

$$\text{Biological degradation}\,[\%] = \left(1 - \frac{c_{start}}{c_{end}}\right) \times 100\,[\%]$$

with

c_{start} = concentration MP at the start of the experiment [µg/L].
c_{end} = concentration MP at the end of the experiment [µg/L].

3.4.2 | Nanofiltration

The feed was measured at the beginning of the experiment (c_f) while the permeate was measured at the end of the concentration (c_p). Because the retentate was returned to the feed container, it can be assumed that the rejection at the end of the experiment was higher than shown in the experiment results (it could be declared a "worst-case scenario"). The rejection rates of the MP was determined by the following equation[15]:

$$\text{Rejection}\,[\%] = \left(1 - \frac{c_p}{c_f}\right) \times 100\%$$

with

c_p = concentration MP permeate [µg/L].
c_f = concentration MP feed [µg/L].

4 | RESULTS AND DISCUSSION

4.1 | Fixed-bed reactor (FBR)

The retentate from the NF experiments was collected and used as an influent for the FBR to investigate the biological degradation.

TABLE 4 Reference substances for the evaluation of micropollutant elimination with detection limits[11]

Micropollutant	Detection limit [μg/L]	Micropollutant	Detection limit [μg/L]
Bezafibrate	0.02	Iomeprol	0.1
Carbamazepine	0.01	Metoprolol	0.01
Clarithromycin	0.02	Sulfamethoxazol	0.01
Diclofenac	0.02	Terbutryn	0.002
Gabapentin	0.05		

TABLE 5 Ø Biological degradation [%] of MP in experiments with fixed-bed reactors (averages of 22 measurements in a period of 637 days; each value with standard deviation *N*) in comparison with literature data of the degree of elimination of MP in conventional wastewater treatment plants [%]

Source Micro-pollutant	Own results in FBR	2	30	28
Bezafibrate	92.4 ± 5.3	41.2 ± 21.9	68 ± 27	9.1–97
Carbamazepin	−5.9 ± 11.3	32.7 ± 17.9	0 ± 36	0–9.5
Clarithromycin	72.9 ± 29.3		28 ± 22	
Diclofenac	53.1 ± 23.7	35.8 ± 23	27 ± 34	2–51
Gabapentin	86.9 ± 25.8			
Iomeprol	75.4 ± 31.7			
Metoprolol	88.9 ± 13.5	37.6 ± 2.4		6.5–65
Sulfamethoxazol	21.6 ± 36.8	64.6 ± 20.4	47 ± 29	12–73.8

Abbreviations: FBR, fixed-bed reactor; MP, micropollutants.

Table 5 shows the average biodegradation rates of the investigated MP. Since there are no comprehensive literature values for specific micropollutants for biological degradation in biofilm reactors, the values are compared with the elimination in conventional wastewater treatment plants.

The table shows that most of the MP are eliminated to a higher degree in the FBR than in wastewater treatment plants. Apart from the two substances that stand out here with a low or even negative degree of degradation (sulfamethoxazole and carbamazepine), the average value was 78%. Tests conducted by other scientists have also proven the poor biodegradability of carbamazepine[16-21] (see also the low literature values in Table 5). This relationship is attributed to the polycyclic, mature structure and the functional amino group of the molecule.[22] Sulfamethoxazole is an antibiotic which is actively effective against bacteria. Various literature values prove a low potential of antibiotics (including sulfamethoxazole).[23,24]

Due to high degradation rates of different MP, it can be concluded that the method is suitable for elimination. Substances which are not degraded here would nevertheless be retained to a high degree in NF (see Section 4.2). These could then be completely eliminated by the adsorption in the sludge or by the "Concentrate treatment" (e.g., ozonation, see Figure 1).

Concerning the degradation rates, another question is to what extent these values result by adsorption of MP, by conversion into metabolites or by the influence of light. First measurements show that after 17 months of the test period, three times less MP were detectable on the fixed bed and sludge than can be found in the weekly feed. It can therefore be assumed that adsorption is a negligible component. Various measurements of the metabolite carbamazepine-10,-11-epoxide suggest that there are no mutual interactions with carbamazepine. There are a large number of other metabolites of the different MP, which concentrations have not been measured so far. In general, it is difficult to answer the question as to how far the metabolism has taken place completely. The influence of light does not seem to play a significant role in MP elimination. By darkening reactor 2, no relevant changes in MP-degradation were detected in a five-month test period using six measurements.

4.2 | Nanofiltration (NF)

4.2.1 | Rejection micropollutants

Table 6 shows the high rejection rates (which can only be specified as a minimum due to the detection limits of the MP) of these MP. The average minimum rejection rate was 95%. Even at high concentrations in the feed/retentate of the NF (factor of concentration up to 96), elimination rates of at least 85% for every single MP could be achieved. Thus, this method represents a suitable barrier for these pollutants.

The rejection rates can also be regarded as high, particularly in view of the "worst-case scenario" used in the evaluation (the permeate at the end of the concentration is set in relation to the feed from the start, see Section 3.4.2). As the retentate repeatedly passes the NF as feed during the concentration process, it is similar to the overall concept shown in Figure 1. In this overall concept, the retentate is returned to the biofilm reactor and then functions again as NF-feed. Thus, this test setup can be regarded as proof of concept with regard to the MP retention. Figure 4 in Section 4.2.2 also confirms this connection, in that despite the increasing MP concentration in the feed/retentate, the concentration in the permeate increases only slowly.

TABLE 6 Ø Rejection rates [%] and experimental setup of experiments with NF270 (average values of 11 experiments; each value with standard deviation *N*) in comparison with literature data; values of molecular weight by National Center for Biotechnology Information[25]

Source Experimental setup/ MP pollutant	Own results	29	31	32	Molecular weight [g/mol]
Feed	Effluent wastewater treatment plant	Artificial feed with 20 µg MP per liter	Artificial feed with 100 µg MP per liter	Artificial feed with 200 µg MP per liter	
Operating mode	Recirculation retentate; collection permeate	Recirculaton Retentate + Permeate	Recirculaton Retentate + Permeate	Recirculaton Retentate + Permeate	
Operating temperature	18–32 °C	25 ± 1°C	20–40°C	25 ± 0.5°C	
Membrane area	2,6 m^2	42 cm^2	40 cm^2	32 cm^2	
TMP	4–6 bar	5 bar	Not specified	5 bar	
Bezafibrate	93.1 ± 6.0		95	80	361.8
Carbamazepine	91.8 ± 4.7	87	74	80	236.3
Clarithromycin	98.8 ± 0.8	99			748.0
Diclofenac	98.0 ± 1.3		95	85	296.1
Iomeprol	99.1 ± 0.7				777.1
Metoprolol	90.8 ± 5.2	99			267.4
Sulfamethoxazol	97.1 ± 2.0	93	95	60	253.3
Terbutryn	96.0 ± 3.3				241.4

Abbreviations: MP, micropollutants; TMP, transmembrane pressure.

Table 6 also shows the different experimental setups. It can be seen that the tests of this project were carried out with real wastewater, a permeate collection and a larger membrane area, in contrast to the comparative values. These circumstances indicate a test setup closer to real MP-elimination. Permeate collection could lead to an increasing feed concentration and thus to poorer rejection efficiencies. Table 6 shows that our results have at least similar or often even higher rejection rates.

The MWCO of the NF 270, which plays a decisive role in MP-rejection, varies between 150 and 400 g/mol depending on the source.[22-24,26] In these experiments, even the smallest molecules like carbamazepine (236.6 g/mol) were highly retained, which supports a MWCO below 300 g/mol. Although all MP are retained to a high degree, a dependency between molecule size and respective deposition efficiency can be established. For example, the retention rates of proportionately larger molecules such as iomeprol or clarithromycin are higher than those of smaller ones like carbamazepine or metoprolol.

The NF 270 is usually negatively charged in the pH range between 6 and 8.[27] According to Besha et al.[28] and Zhao et al.,[29] negatively charged substances such as diclofenac and sulfamethoxazole are retained better than neutral (e.g., carbamazepine) or positive (e.g. metoprolol) substances, which could be confirmed in the experiments.

4.2.2 | Correlation concentration retentate/permeate

Figure 4 shows the relationship between the summarized concentration of MP in the retentate and the concentration in the permeate. It is clearly apparent that the concentration in the permeate increases with the concentration in the retentate. Since the retentate also represents the feed in the NF, this relationship corresponds to the expectations. The trendline has a low slope rate of 0.02 indicating that the MP concentration in the permeate increases very slowly relative to the concentration in the retentate. Based on this trendline, it can be concluded, that the MP are retained to a high degree even at high concentrations in the feed/retentate.

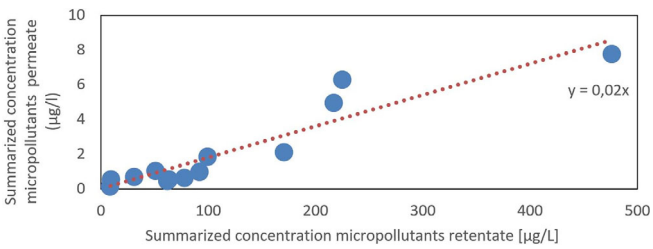

FIGURE 4 Behavior of concentration of micropollutants in the permeate related to the concentration in the retentate [Color figure can be viewed at wileyonlinelibrary.com]

5 | CONCLUSION

The experimental results show that a significant biodegradation of various MP takes place in the fixed-bed (biofilm) reactors (using the retentate of the NF as the effluent). Six of the eight detected MP had a degradation rate of more than 78%. The degradability of the

substances was proven by testing the adsorption on sludge and fixed bed and the influence of light. The antibiotic sulfamethoxazole was degraded to a smaller extent (22%) while the antiepileptic carbamazepine even had negative degradation rates. The poor degradation potential of these two substances is confirmed by experimental results from the literature.

NF has demonstrated its effectiveness as a barrier to MP with an average separation efficiency of more than 95%. Substances which are not biodegradable in the FBR can still be retained by the NF and eliminated by adsorption in the sludge or by a concentrate treatment in the overall concept. As expected, large molecules are better retained by the NF system than smaller ones. The assumption regarding the charge was also confirmed: negatively charged MP can be better retained by the negatively charged NF 270 than positively or neutrally charged MP.

The successful biological MP degradation of the retentate concentrated by NF in the FBR demonstrated the potential of the concept. A combination plant of the two processes has been put into operation, although its optimization and evaluation of results is still pending.

In the long term, a pilot plant with the overall concept is to be set up so that the *MicroStop*-project will achieve a uniform process for the elimination of MP including X-ray contrast agents, antibiotics, microplastics, and pathogenic germs.

ACKNOWLEDGMENTS

We would like to express our special thanks to the following persons for their reliable support: Susanne Eggers (internal laboratory affairs), Dipl.-Ing. Alina Stahl/Dr. rer. nat. Heike Frerichs (Central Laboratory of the TUHH), Jessica Rumpca/Tatjana Grimm/Jon Wullenweber (Student Assistants) and all the students, who carried out their student works on the project. Special thanks also go to Sarah Löhn, who provided Figure 3 from her master thesis. We would also like to thank our cooperation partner *Hamburg Wasser* for the exchange of information, the provision of wastewater and the financial support. Additionally, we would like to thank the *Rosa-Luxemburg Stiftung* for the provision of a doctoral scholarship. Open access funding enabled and organized by Projekt DEAL. WOA Institution: TECHNISCHE UNIVERSITAET HAMBURG Blended DEAL: ProjektDEAL.

AUTHOR CONTRIBUTIONS

Bastian Buening: Conceptualization; data curation; formal analysis; funding acquisition; investigation; methodology; resources; software; validation; visualization; writing-original draft; writing-review and editing. **Dorothea Rechtenbach:** Conceptualization; data curation; formal analysis; funding acquisition; investigation; methodology; project administration; resources; supervision; validation; visualization. **Joachim Behrendt:** Conceptualization; data curation; formal analysis; funding acquisition; methodology; project administration; resources; supervision; validation. **Ralf Otterpohl:** Conceptualization; funding acquisition; project administration; resources.

DATA AVAILABILITY STATEMENT

The data that supports the findings of this study are available in the supplementary material of this article. Since it is not possible to upload Excel-FIles on ScholarOne, I have sent the Data to Mr. Houston via Mail. When "Data openly available in a public repository that issues datasets with DOIs" is preferred, this is also possible.

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SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of this article.

How to cite this article: Büning B, Rechtenbach D, Behrendt J, Otterpohl R. Removal of emerging micropollutants from wastewater by nanofiltration and biofilm reactor (*MicroStop*). *Environ Prog Sustainable Energy*. 2021;40:e13587. https://doi.org/10.1002/ep.13587