



# Laboratory test reports and fact sheets on water treatment technology and reuse

## Deliverable 3.2

WP3 Treatment and recovery technologies

Task 3.2: Laboratory experiments per wastewater treatment and reuse technologies

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Deliverable type		
R	Document, report	<b>X</b>
DEM	Demonstrator, pilot, prototype	
DEC	Web sites, patent fillings, videos, etc.	
OTHER	Software, technical diagram, etc.	
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## SUMMARY

The summary provides an overview of the most significant outcomes of the laboratory test reports and fact sheets on wastewater treatment technology and reuse for seven different technologies. Each chapter of Deliverable 3.2 provides more detail concerning lab-scale operations, process and design optimizations, and the interactions between the laboratory-scale and pilot-scale for water treatment. The deliverable shows how technology adaptations and optimisations have been carried out to meet Indian standards and Indian wastewater conditions at the laboratory scale and provides indications on what happens next in adjusting and / or evaluating the respective technologies going forward.

The **aerobic membrane bioreactor (MBR)** system is a lab-scale reactor with a capacity of 8 litres of working volume. The system was installed and operated at IIT Delhi waste treatment labs using Barapullah drain wastewater. The purpose of testing this technology is that it is a benchmark for innovative technologies to compare to, in addition the set up was to assess whether trace organics such as pharmaceutical residues and pesticides are being removed adequately by the technology. The MBR reactor, which had been ready for operation since the end of 2002, underwent continuous minor improvements, including the addition of a refrigerator to prevent biological degradation in the feeding tank before reaching the MBR tank. From March 22 to May 11, 2023, the reactor operated at a flowrate of 1.5 L/day, aiming to establish sufficient biomass without disrupting membrane filtration. Preliminary results indicated its performance, showcasing COD removal, pH maintenance within municipal wastewater standards, and stable operation at an elevated temperature of approximately 28°C. Notably, influent total COD from the Barapullah drain was relatively low, and the MBR effectively reduced it to around 29 mg/L due to the microfiltration membrane. Despite the low flowrate and non-continuous operation, the COD removal results were promising, suggesting potential for improved efficiency when operating at a higher flowrate. The remaining months of the project are used to assess whether trace organics are being removed by the Aerobic MBR.

**ANDICOS™** is a concept based on anaerobic digestion of concentrated wastewater and organic waste streams. The concentration of the wastewater is done with a BFM-membrane filtration process. Lab-scale experiments were conducted at IIT Kanpur using BFM membranes to filter Indian wastewater, including wastewater from IIT Kanpur Campus and more challenging sewage from the Jajmau Standard Treatment Plant (STP), contaminated with illegal tannery discharges. Critical flux experiments with the primary settled sewage showed an optimal filtration flux range of 15 - 25 L/h-m<sup>2</sup>. The permeate flow was around 95% of the wastewater flow with a COD concentration <120 mg/L. The concentrated wastewater represented around 5% of the initial wastewater flow with a COD concentration of 9 500 mg/L and a TSS concentration of 9 000 mg/L. The COD loss due to biodegradation in the air-scoured membrane tank was 32%. The change in the sewage characteristics observed after COVID-19 (2021-2022) had a major impact on both the composition of the concentrate stream and the unwished biodegradation that took place in the membrane reactor. Up to 70% of the COD was lost because of biodegradation indicating only limited concentration of the wastewater was possible. To reduce biodegradation, lab tests using nitrogen for scouring of membranes resulted in a reduced loss of COD from 70% (in air) to 40% (in nitrogen). During lab tests with reduced SRT from ~12 days to ~8 hours, the biodegradation losses were still high due to the deposition of biofilm on reactor walls. To optimize the membrane



cleaning process and frequency, different concentrations of sodium hypochlorite were tested using 100 - 900 ppm active chlorine solutions. The laboratory tests have been invaluable to assess different options to reduce biodegradation losses, which would be more costly to undertake at pilot scale. With the drastic improvements of the Jajmau STP influent occurring during the project, further laboratory scale work is investigating the treatment of different mixes of CETP and STP influent.

**Cleanblocks** are a mineral wool filtering material, that can be used for the treatment of polluted waters, like drain and wastewater. Lab-scale experiments were conducted to evaluate nutrient removal using mineral wool and biofilm in continuous operation. Mineral wool was characterized for its TSS removal capabilities and prone to clogging. Design modifications, including the use of mineral wool cubes, improved solids settling and run times, but it was concluded that Cleanblocks technology is not suitable for in-drain treatment due to clogging sensitivity. Nevertheless, Cleanblocks can be used as a polishing step to remove suspended solids after other treatment technology, such as the Photo Activated Sludge (PAS) system. Experiments were conducted at the Barapullah site, showed that while suspended solids were removed, total dissolved solids concentration and effluent conductivity increased. Finally, nitrification was observed, but denitrification did not occur due to high oxygen levels in the feed. Thus, no phosphorus or nitrogen removal was achieved. Further work is expected on evaluating the potential of biofilm modifications to Cleanblocks.

The **modified Constructed Wetland** technology combines vertical flow constructed wetlands with adsorptive elements such as granular activated carbon and specific sorbents for enhanced heavy metal removal. Lab-scale water treatment experiments were conducted at various institutions, to test the use of adsorbents (GAC and zeolites) and to assess their ability to remove heavy metals and dissolved organic carbon from synthetic wastewater. Batch tests indicated good removal rates for certain heavy metals (like Fe and Cr), while column tests showed variable removal rates for different contaminants over time. Overall, activated carbon performed better than zeolites. Rapid Small-Scale Column Tests (RSSCT) were used to predict breakthrough behaviour of pollutants more quickly and with less water volume. The column tests caused precipitation and clogging issues due to the enrichment of wastewater with heavy metals. Building upon the progress made thus far, further investigation will focus on optimizing the RSSCT tests. This includes refining parameters such as particle sizes and flow regime. The analysis of trace organic compounds is also included in future research.

The **photo-activated sludge (PAS)** uses the symbiotic relationship between microalgae and aerobic bacteria in an open bioreactor to maximize the oxidation of ammonium and organic carbon through photosynthesis. In the lab-scale bioreactors, the "survival of the fittest" strategy was used to increase lipid productivity by using microalgae-bacteria consortia in photo-sequencing batch reactors (PSBRs) settings. PSBR 1 was fed with synthetic brewery wastewater during the light period and synthetic liquid digestate during the dark period. PSBR 2 was operated under normal conditions, and it was fed with mixed synthetic brewery wastewater and liquid digestate during the light period. The PSBR 1 had higher COD, TOC, TIC,  $\text{NH}_4^+ - \text{N}$ , and TN removal rates of 14.0, 1.0, 1.2, 1.2, and 0.9 mg/L.h, compared to PSBR 2, that had removal rates of 2.0, 0.3, 0.7, 1.0, and 0.5 mg/L.h, respectively. To enhance the efficiency of the laboratory-scale PAS system in removing nutrients, COD, BOD, additional research should be conducted to determine the impacts of pond depth, light intensity, lipid yield, and other biochemical characteristics of the algal biomass. It is



recommended to integrate the PAS with the Clean Blocks in order to evaluate the overall treatment effectiveness of the hybrid system.

The **Self-forming Dynamic Membrane Bioreactor (SFD MBR)** is an integrated biological-filtration treatment technology where filtration nets are used as supports to favour the spontaneous accumulation of a sludge layer which becomes the main filtration medium. Lab-scale experiments were conducted to assess process performance in terms of effluent quality and operation sustainability. The results demonstrated excellent COD removal and effluents with low TSS levels and turbidity values suitable for UV-based disinfection and drip irrigation. Moreover, the technology enabled complete nitrification and maintained phosphorus levels. Furthermore, the results of process optimization tests indicated that mesh size and air scouring intensity play crucial roles in maintaining high effluent quality while minimizing manual cleaning requirements. Additionally, high SRT values were shown to improve the system's performance. The choice of maintenance strategies, such as periodic backwash and air mass load supply, also affected the sustainability of the operation. Finally, although there were differences in configurations between the bench-scale SFD MBR and the pilot plant (Xylem Taron®), comparable results were obtained in terms of effluent quality. Key findings indicated that higher suspended biomass concentrations and intermittent maintenance of filtering surfaces could offer advantages in terms of bioprocess stability, energy efficiency, and effluent quality maintenance. Further investigation is planned for optimizing the dynamic membrane maintenance through temporized air mass load and evaluate the effectiveness of this operating strategy with different mesh size supports. Once optimized, this approach can be transferred and tested at larger scale installations.

**Structured adsorbents** are based on the adsorption principle and is especially suited for effluents containing low amounts of contaminants. Lab-scale experiments focused on structured sorbents for wastewater treatment, with Sorbent 1, composed of 80% LDO and 20% bentonite, being identified as the optimal composition for adsorbing Cr<sup>3+</sup> and Cr<sup>6+</sup>. Batch tests revealed that higher solid-to-liquid (S/L) ratios improved Cr recovery efficiency, achieving removal efficiencies of 93-96% with a 2.5 g/100mL influent S/L ratio. An optimal desorption and regeneration solution using 2M NaCl with a neutral pH was found. Afterwards, the kinetic and isotherms for Cr sorption on self-structured adsorbents were studied, and it was determined that higher S/L ratios enhanced Cr recovery efficiency. Finally, columns designed based on feasibility tests and modelling ran for approximately 50 days. Regeneration of columns was achieved with a 2M NaCl solution at a neutral pH, but pH adjustment was required for the highly basic effluent before further use or disposal. As at this stage only limited experiments were performed under flow conditions, future work should include the optimization of operation parameters under flow conditions, validation of the regeneration procedure under dynamic conditions (to enable the multicycle use) and the investigation on the use/regeneration/disposal of the exhausted structured adsorbents. Finally, as this technology was demonstrated for various Cr concentrations, namely at small scale for effluents containing high Cr concentrations and at larger scale for low Cr concentrations, it will be important to test it under various Cr containing wastewater sources and investigate its applicability for wastewater treatments in different industrial sectors.



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## CHAPTER 1 TECHNOLOGY: AEROBIC MEMBRANE BIOREACTOR

### 1.1. ADMINISTRATIVE INFORMATION

Technology name	Aerobic MBR
Location of lab and/or pilot	Pilot – New Delhi, India
Organization(s)	IHE Delft, IITD
Contact information:	Lead: Hector Garcia, Anil, Shaikh Ziauddin
	Other:

### 1.2. BRIEF TECHNOLOGY DESCRIPTION

The aerobic membrane bioreactor (MBR) system is a lab-scale reactor with a capacity of 8 liters of working volume. The system was installed and operated at IIT Delhi waste treatment labs using Barapullah drain wastewater (Figure 1-1 and Figure 1-2). The system used Kubota membranes for filtration and works with a temperature-controlled feed supply. The system was automated using level sensors in both reactors and feed tanks to achieve a continuous steady state. The parameters were monitored to define the appropriate flow parameter values to maximize efficiency.

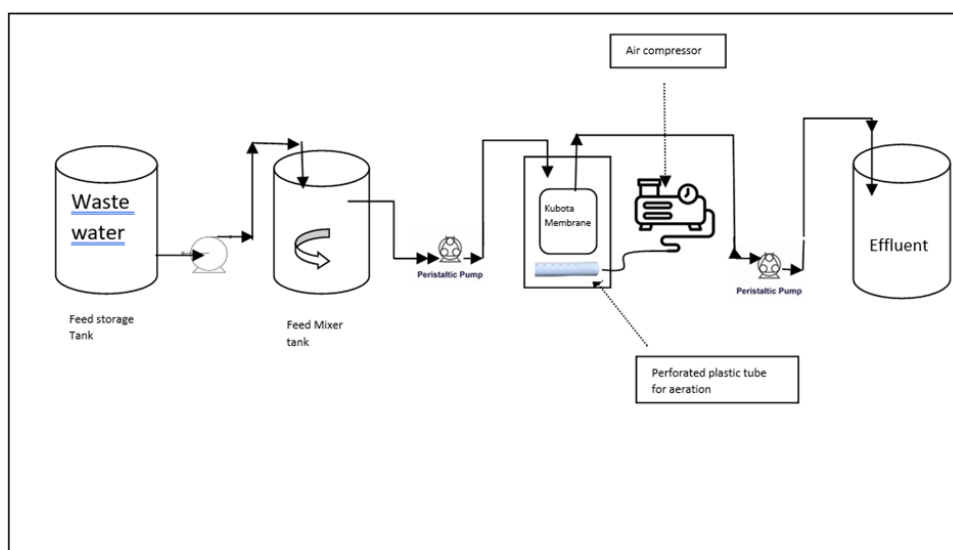


Figure 1-1: Schematic Diagram of MBR



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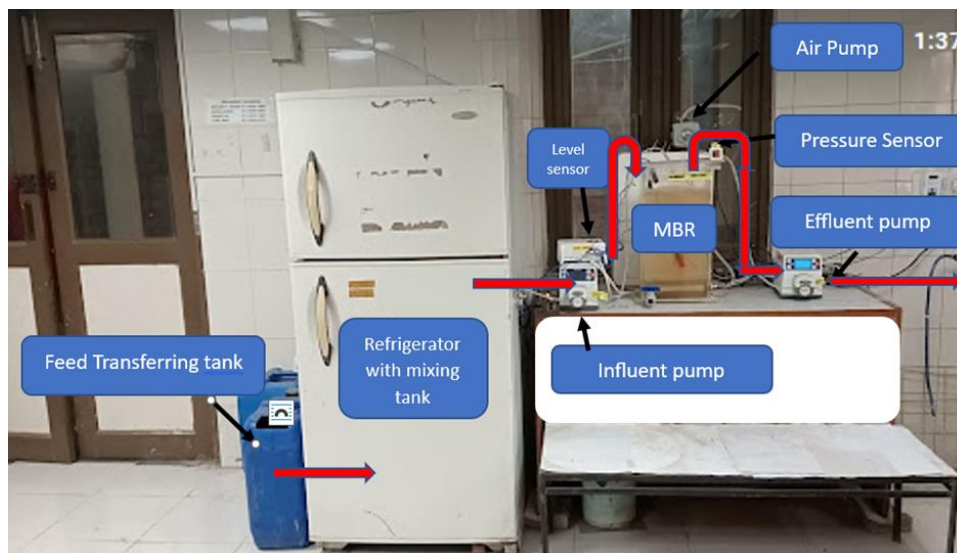


Figure 1-2: The image of MBR setup at IIT Delhi labs

Video link for the MBR setup in working state.

<https://photos.app.goo.gl/Lbretw1aHagWJHqL9>

### 1.2.1. RESEARCH OBJECTIVES

Two types of technologies were developed under the PAVITRA GANGA project as follows: (i) BN: Efficient, low energy bulk organic and nutrient removal technologies; and (ii) PS: Polishing technologies. The performance of the BNs and Ps technologies developed under the PAVITRA GANGA are compared with a more robust and well know technology such as the aerobic membrane bioreactor (MBR).

## 1.3. PROCESS OVERVIEW

### 1.3.1. PROCESS DESCRIPTION

MBRs combine an aerobic biological treatment with a membrane filtration process introducing the following advantages: (i) production of a clarified effluent (low turbidity, bacteria, TSS, and organic content); (ii) small footprint needs; (iii) bulking problems become less relevant; and (iv) possibility to operate the biological system at long solid retention times (SRTs) reducing the sludge production, among others.

### 1.3.2. DIMENSIONING

The schematic of the MBR system configuration is presented in Figure 1-3. The MBR system can operate as follows. The influent wastewater from the drain can be pumped daily or every two days to an influent feeding tank. Calcium carbonate can be added to increase the alkalinity (if required)



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to avoid acidification in the MBR reactor. An influent pump will introduce the influent wastewater to the MBR tank at a maximum flowrate of approximately 50 L/day (so the feeding tank will have a minimum capacity of approximately 50 L capacity). The influent pump on/off is controlled by a level sensor in the MBR. The effluent will be taken out of the system through a Kubota microfiltration membrane by a permeate pump. The permeate pump should work on cycles of approximately 20 minutes on and one minute off. For controlling the cycles, the effluent pump can be connected to a digital timer. Air will be introduced to the MBR to provide dissolved oxygen for the process, as well as to provide bubbles for scouring the membrane. A compressor with a minimum of 60 L/min capacity will be more than enough to provide aeration. The pressure of the membrane will be monitored by a pressure gauge. The pH, temperature, and dissolved oxygen will be also monitored as frequently as possible. The system as presented below is designed for carbon removal and nitrification (denitrification not included).

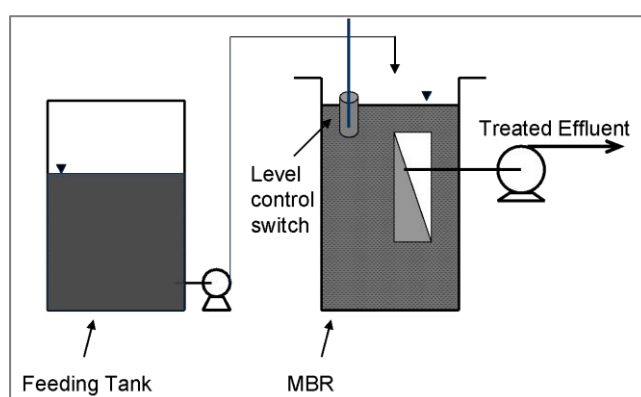


Figure 1-3: MBR Schematic

### 1.3.3. TECHNICAL SPECIFICATIONS

Components of the system

#### A - Plexiglas MBR basin/reactor

A Plexiglas (or similar material) rectangular reactor is shown in Figure 1-4.

Specifications:

- Plexiglas sheet thickness: 1.25 cm (approximately)
- Dimensions: Length = 22 cm; Width = 10 cm; Height = 47 cm (internal dimension)
- Total Volume: 10,340 cm<sup>3</sup> (10.34 L)
- Working volume: To operate at approximately 8.0 L.

Air will be introduced to the reactor from the bottom of the reactor. Figure 1-5 indicates the hole and thread that needs to be added at the bottom of the reactor to place the diffuser.





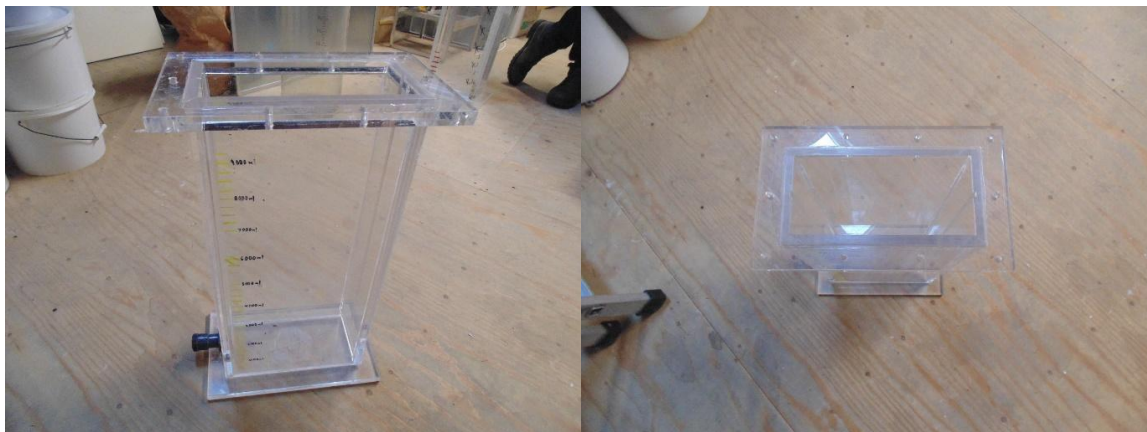
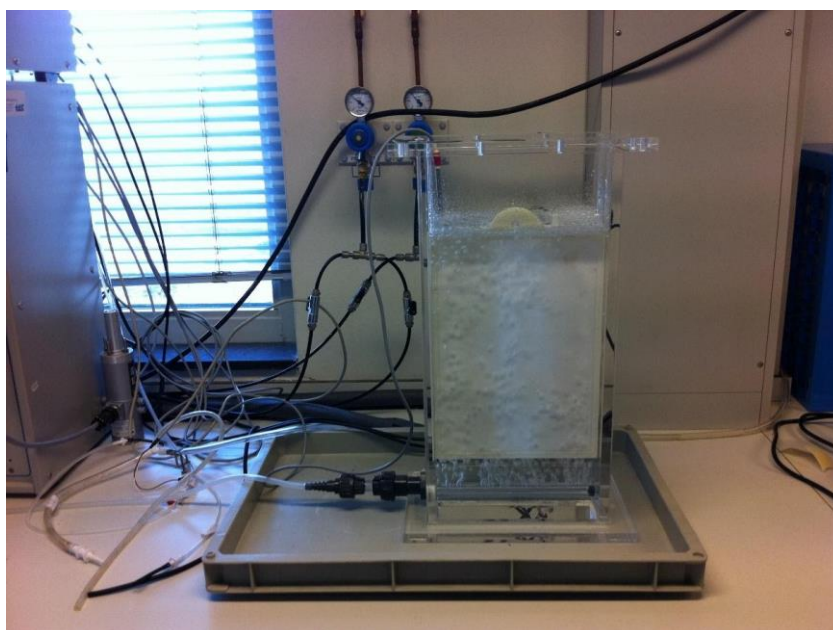


Figure 1-4: MBR aerobic reactor



Figure 1-5: Several views of the thread needed to introduce a plastic pipe for providing aeration to the reactor.



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Figure 1-6: Plastic tube introduced at the bottom of the reactor for providing aeration to the reactor.



Figure 1-7: Plastic tube showing the holes for forming coarse bubbles of 1 mm.

As observed in Figure 1-6, a flat sheet polymeric membrane will be submerged in the reactor. For placing that membrane in the reactor two grooves need to be cut at each side of the reactor as shown in Figure 1-8.



Figure 1-8: Grooves needed to be cut at both sides of the reactor for sliding the membrane.

#### B - Membrane

Kubota bench-scale membrane cartridge type 203. Membranes shown in Figure 1-9.

- Dimensions: Length 23 cm; Width 0.7 cm; Height 31.5 cm



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- Filtration area = 0.1364 m<sup>2</sup>
- Standard Flux = 20 L/m<sup>2</sup>-hr (LMH). Though we will operate below 10 LMH



Figure 1-9: Kubota A4 membranes

C - Air Compressor (in case compressed air/blower does not exist in the building where the bench MBR will be placed)

A compressor or blower needs to be provided for supplying air to the reactor.

D -Pumps

Pumps are needed both for the influent and for taking out the effluent permeate through the membrane. So at least two pumps are needed as follows:

- Precise peristaltic pump BT100 2J or similar with head YZ1515X or similar
  - Flowrate specification of that pump 0 to 380 mL/min
  - Flowrate needed approximately 20 L/d = 14 mL/min
  - At least two pumps are needed one for the effluent (connected to the membrane) and another for feeding the MBR reactor (from the feeding tank to the MBR)

E - Timer

The effluent pump connected to the membrane should be controlled (switch on/off) by a timer.

F - Level sensor

The influent pump should be controlled by a level control system.

G - Membrane Pressure meter

The pressure in the membrane should be monitored. The following digital pressure meter can be provided. Figure 1-9 describes one example of this sensor.



Figure 1-10: Digital pressure meter



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#### H - pH, T, and DO monitoring

These parameters can be monitored using portable meters.

### 1.4. TECHNOLOGY-SPECIFIC SUMMARY AND CONCLUSIONS

The reactor was ready to operate since the end of 2022; however, several minor improvements were continuously added including the addition of a refrigerator for the influent feeding to avoid biological degradation in the feeding tank before reaching the MBR tank.

The performance of the MBR reactor was monitored from March 22 to May 11, 2023. The reactor operated at a flowrate of 1.5 L/day approximately to build enough biomass in the system without affecting the membrane filtration process.

Some preliminary results indicating the performance of the MBR system in terms of COD removal, pH and Temperature performance are presented in Figure 1-11 to Figure 1-13, respectively.

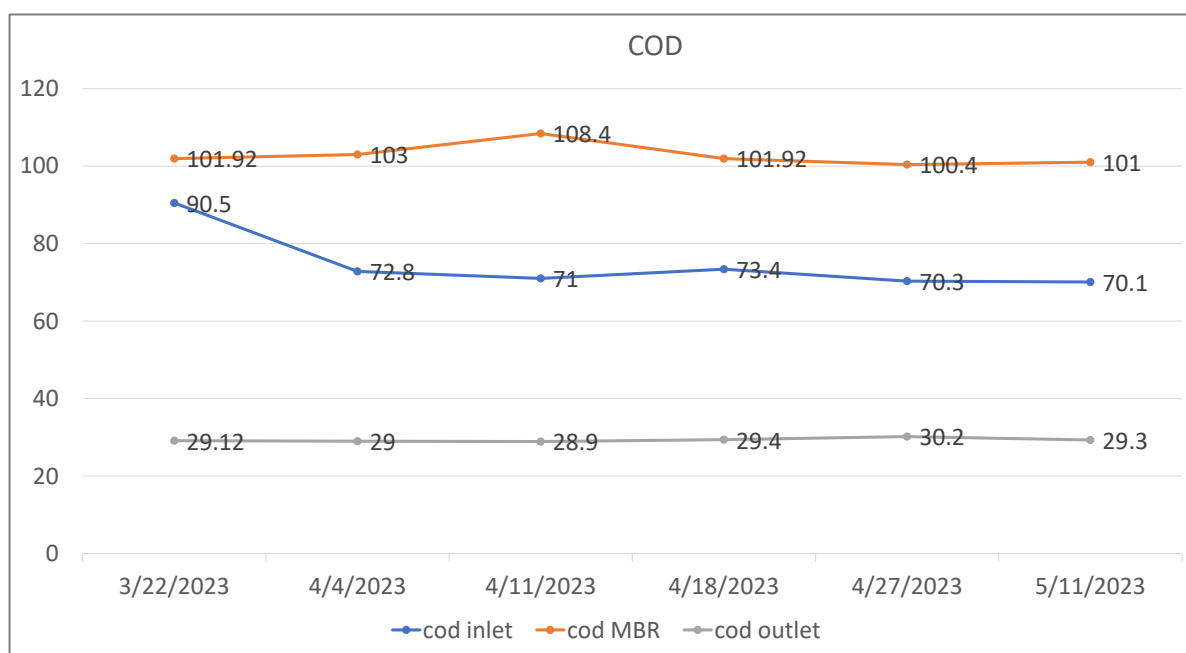


Figure 1-11: COD measurements

The influent total COD from the Barapullah drain fluctuated from approximately 90.5 to 70.1 mg/L. This can be considerable in the low range of total COD compared to municipal wastewater. The effluent COD obtained in the permeate of the MBR was much lower without showing many variations at approximately 29 mg/L. Since the MBR is equipped with a microfiltration membrane with a pore size exclusion of 0.45  $\mu\text{m}$ , both the biodegradable COD as well as the particulate COD could be removed explaining the COD removal in the reactor.

The total COD in the MBR is quite low, approximately 100 mg/L. Even though total suspended solids (TSS) or volatile suspended solids (VSS) were not determined, a conversion factor of 1.48 mg COD/mgVSS can be used to determine the amount of VSS in the reactor. So, applying that



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conversion factor yields a VSS concentration of approximately 70 mg/L which could indicate a TSS concentration lower than 100 mg/L being extremely low for operating an MBR. TSS concentration in a MBR can reach 10,000 mg/L. Therefore, it can be concluded that indeed the influent flowrate to the MBR was low (1.5 L/day) when it can go as high as 50 L/day. In addition, eventually, the reactor was not operated in a continuous mode during such an extended period. However, the results regarding the COD removal are quite promising.

The pH and temperature were also monitored during that period. The influent pH was observed in a standard range for municipal wastewater with an average value of approximately 7.3. Not major changes were observed in the influent. Slightly higher pHs were reported in the MBR. Not much biological activity was observed at the low influent flow rate. However, when the flowrate increases and if the presence of ammonia in the influent is considerable, some nitrification would occur reducing the pH. There should not be major changes between the effluent and MBR pH since the system works as a complete mixed tank.

The temperature values indicated that the influent was kept refrigerated and the system is being operated at an elevated temperature of approximately 28°C. The biological performance of the reactor should work fine at such a temperature.

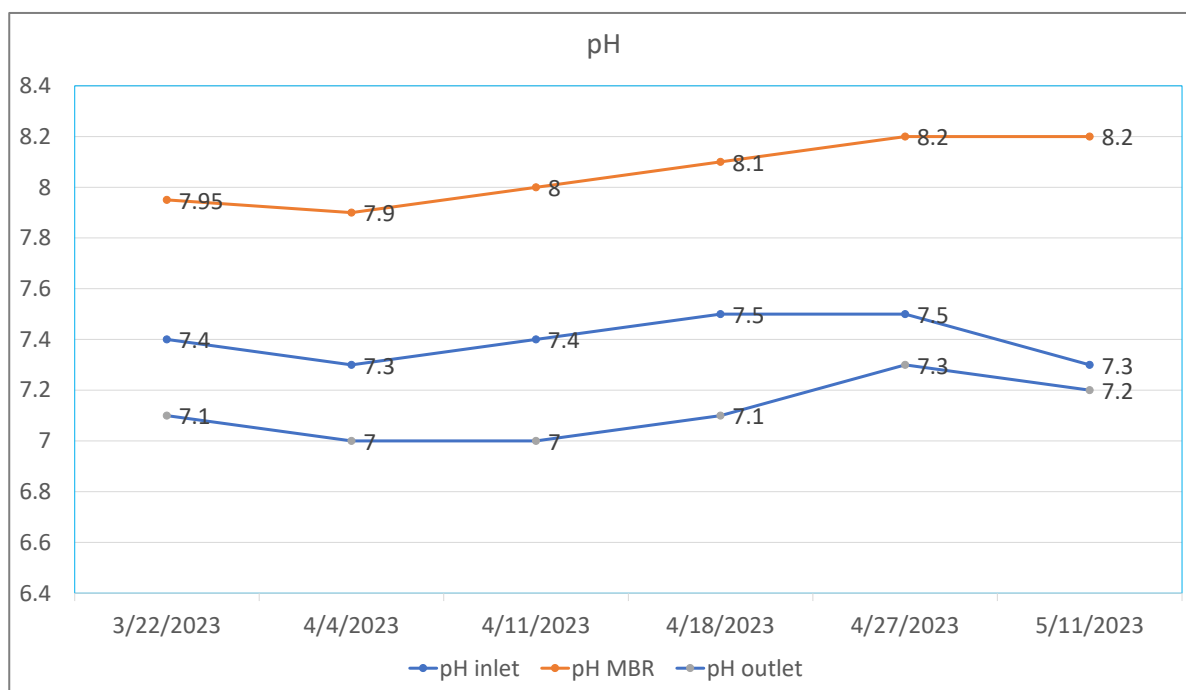


Figure 1-12: pH performance



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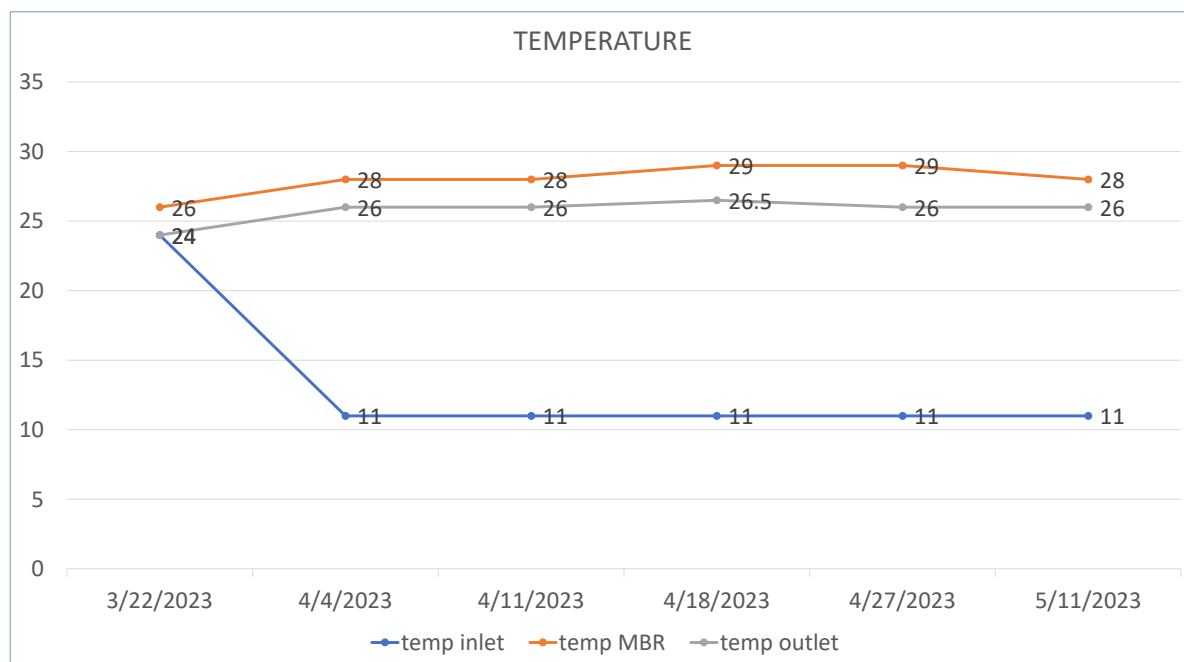


Figure 1-13: Temperature performance

#### 1.4.1. SAFETY MEASURES AND TROUBLESHOOTING

For the laboratory experiments, no extra measurements or troubleshooting issues have been encountered.



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## CHAPTER 2 TECHNOLOGY: ANDICOS

### 2.1. ADMINISTRATIVE INFORMATION

Technology name:	Andicos	
Location of lab and pilot:	IIT Kanpur, India	IIT Kanpur, India
Organization(s):	IIT Kanpur, VITO, IEX	IIT Kanpur, VITO, IEX
Contact information:	Lead: IIT Kanpur	Lead: IIT Kanpur
	Contact: Aditya Sharma, Prof Bose, Sachin Shah, Sofie Van Ermen, S. Sankararaman	Contact: Aditya Sharma, Prof Bose, Sachin Shah, Sofie Van Ermen

### 2.2. BRIEF TECHNOLOGY DESCRIPTION

ANDICOS™ is a concept based on anaerobic digestion of concentrated wastewater and organic waste streams. The principle is to increase the organic load from municipal sewage using filtration to allow a much more efficient anaerobic digestion. The increase in organic load can also be obtained by adding organic waste (e.g., kitchen waste, food waste, sewage sludge, industrial biowaste, etc.) The filtration is done with a BFM-membrane filtration process.

ANDICOS™ as a modular system can be installed as a stand-alone system or added to existing STPs to improve performance and/or extend capacity. Furthermore, it produces energy, which be used to off-set operational and maintenance costs within the treatment plant.

A general process overview of the ANDICOS process is presented in **Error! Reference source not found..**

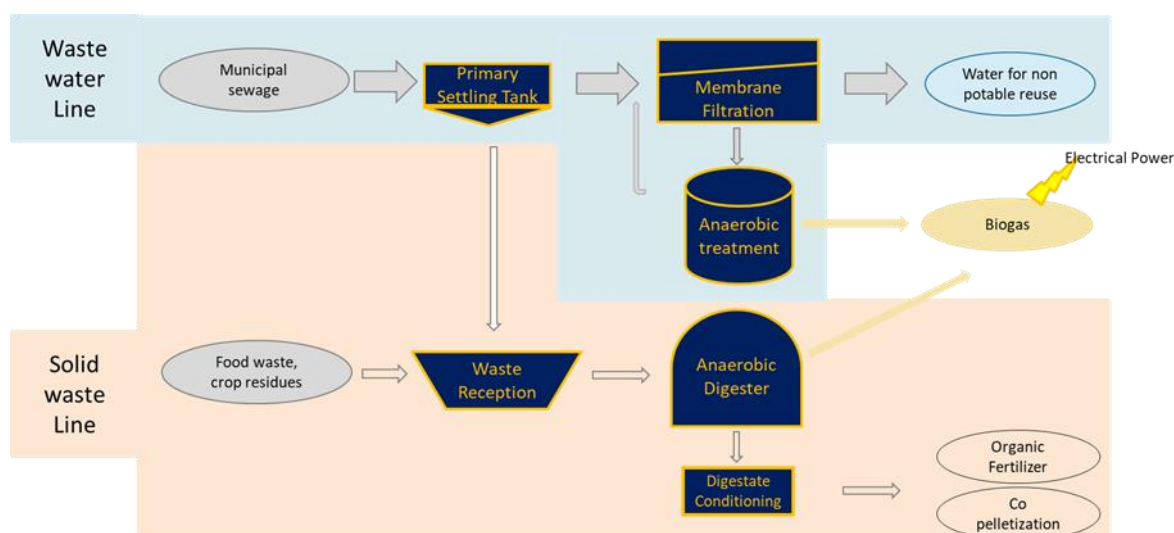


Figure 2-1: Andicos concept scheme and general process overview

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The sewage filtration uses BFM membranes. These IPC™ (Integrated Permeate Channel) membranes are the world's first thoroughly back washable flat sheet membranes. Thanks to the capability of back washing up to 2 bar(g), improved fouling control of the membranes can be achieved.

#### MEMBRANE SPECIFICATIONS

Material	PVDF
Pore-size	< 0,08 µm
Permeability	2000 l/m <sup>2</sup> .h.bar
Back wash pressure	< 2 bar

Figure 2-2: BFM membrane specifications (source [www.bluefootmembranes.com](http://www.bluefootmembranes.com))

Anaerobic digestion of the concentrated sewage, primary sludge and kitchen waste can be done using an anaerobic digester or a UASB reactor. During the project, anaerobic digestion of primary sludge was assessed on a lab-scale.

### 2.3. FACTSHEET: INFLUENT TYPES

Table 2-1: General factsheet: ANDICOS

Parameter		Units	Value	Comments
Influent type(s)	Sewage after primary sedimentation			Influent to BFM filtration
	Concentrated sewage (= concentrate from BFM filtration)			Influent to UASB or anaerobic digester
	Primary sludge			Influent to anaerobic digester
	Other organic waste streams			Influent to anaerobic digester
<b>BFM filtration</b>				
Influent quality	COD	mg/L	200 - 1000	
	TSS	mg/L	500 - 1200	
	Ntot	mg/L		
	Ptot	mg/L		
	Cr	mg/L	0 - 7	
Permeate quality	COD	mg/L	<100	
	TSS	mg/L	<1	
	Ntot	mg/L		



	Ptot	mg/L		
	Cr	mg/L	<0,1	
	Coliforms	CFU/ml	0	
Concentrate quality	COD	mg/L	500 - 7500	
	TSS	mg/L	2000 - 5700	
	Ntot	mg/L		
	Ptot	mg/L		
	Cr	mg/L		
Technology-defining Rates/ Ranges				
	Sewage concentration factor (volume based)		10 - 20	
	Membrane net flux	Lmh	11 - 20	
	Cleaning frequency		Every two weeks - every month	

## 2.4. TECHNOLOGY-SPECIFIC SUMMARY AND CONCLUSIONS

This chapter focuses on the BFM filtration step of the Andicos concept. Wastewater after primary sedimentation is filtrated producing two streams: a filtered water stream for non-potable reuse and a concentrated wastewater stream to feed an anaerobic digester (D3.3). The lab tests focus on the relation between the concentration factor and the membrane parameters on the filtered water quality and the concentrated wastewater quality.

### 2.4.1. LABORATORY BFM EXPERIMENTS TO PRODUCE CONCENTRATED WASTEWATER TO FEED AN ANAEROBIC DIGESTER AND TO PRODUCE FILTERED WASTEWATER FOR NON-POTABLE REUSE

Lab tests were done to evaluate the performance of Blue Foot Membrane (BFM) membranes for direct wastewater concentration in Indian conditions. The membrane flux was related to the cost of the treatment as this determines the number of membranes required. An optimum between high flux and minimal cleaning frequency was required. An optimal concentration factor combines both a minimal flow of concentrated water - resulting in a more compact anaerobic digester - and an acceptable quality of the filtered wastewater for non-potable reuse.

The IPC Filtration lab-scale comprised of a reactor (volume 32 L) with three submerged flat sheet A4 size BFM membranes (0.11 m<sup>2</sup> membrane surface/membrane) to filter and concentrate wastewater after primary sedimentation. The measurement and pumping devices were connected



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to a computer with the software program Mefias. Mefias co-ordinates filtration, backwash and relaxation by controlling parameters such as flow, flux, and airflow and registers the parameters such as TMP. A filtration cycle of 9 mins filtration, 40 seconds of backwash and 20 seconds of relaxation was used during the tests.

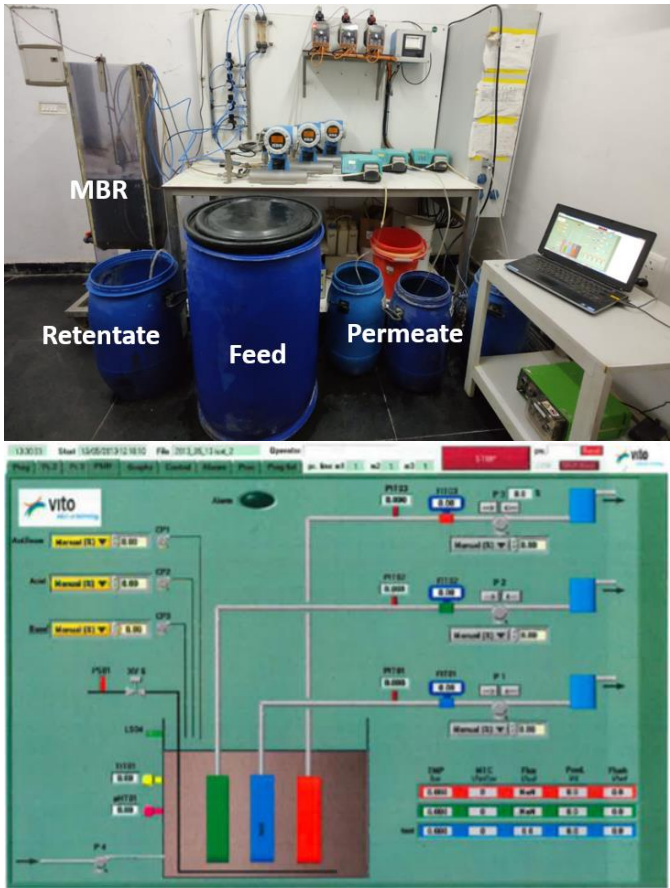


Figure 2-3: Laboratory-scale Andicos filtration set-up (left) and Mefias software (right)

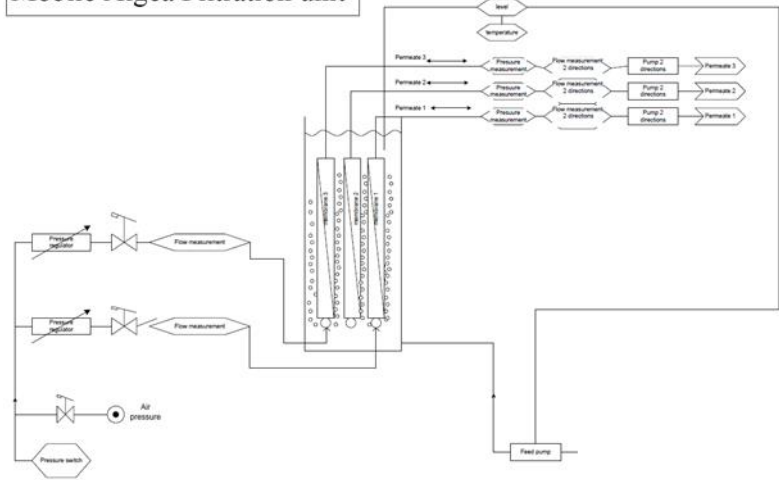


Figure 2-4: Laboratory-scale Andicos PFD.



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### → Filtration experiments during period Q1 2020 (high concentrated sewage)

The first tests with the IPC Filtration lab-scale unit were to prove that the IPC membranes were able to filter Indian wastewater to produce concentrated wastewater for high organic content feed to an anaerobic digester, and to produce filtered wastewater for non-potable reuse.

Tests were conducted with two types of wastewaters: 1) the easily available, but not particularly harsh IIT Kanpur Campus wastewater; and 2) the more distant, and more challenging wastewater from the Jajmau STP, which is sewage from Kanpur city but contaminated with illegal discharges from nearby tanneries. Three different tests were done: a short-term test for determination of critical flux, a 4-day (96h) concentration test, and a 25-day long-term concentration test.

Table 2-2: Water quality parameters testing with primary settled Jajmau STP sewage - IPC filtration unit period Q1 2020 (average  $\pm$  standard deviation).

	influent	Retentate	Effluent
<b>COD (mg O<sub>2</sub>/L)</b>	1138.8 $\pm$ 164	7240.1 $\pm$ 2182.1	96.9 $\pm$ 26.5
<b>sCOD (mg(O<sub>2</sub>/L)</b>	433.6 $\pm$ 61.5	1707.4 $\pm$ 584.3	96.9 $\pm$ 26.5
<b>TSS (mgSS/L)</b>	979.4 $\pm$ 134.5	7515.7 $\pm$ 1781.3	4.3 $\pm$ 2.7
<b>N-TKN (mgN/L)</b>	52.85 $\pm$ 2.2	373.1 $\pm$ 10.7	20.2 $\pm$ 0.7
<b>N-NH<sub>4</sub><sup>+</sup> (mgN/L)</b>	17.7 $\pm$ 1.5	17.8 $\pm$ 1.3	14.8 $\pm$ 1.1
<b>SO<sub>4</sub><sup>2-</sup> (mg/L)</b>	116 $\pm$ 12	128 $\pm$ 19	114 $\pm$ 9
<b>Total Cr (mg/L)</b>	6.6 $\pm$ 0.3	31.1 $\pm$ 11.8	<0.1

### Conclusions:

- A 17 to 20-fold volumetric concentration of Jajmau wastewater after primary sedimentation is possible using a filtration flux of 20 – 25 L.h<sup>-1</sup>.m<sup>-2</sup> and a backwash flux of 30 – 37 L.h<sup>-1</sup>.m<sup>-2</sup>. Stable TMP values are reached during periods of one week and more without cleaning. The permeate flow is around 95% of the wastewater flow with a COD concentration <120 mg/L. The concentrated wastewater represents around 5% of the initial wastewater flow with a COD concentration of 9 500 mg/L and a TSS concentration of 9 000 mg/L. The COD loss due to biodegradation in the air-scoured membrane tank is 32%.
- The outcome of the tests was promising as this indicates that primary settled sewage can be filtered with stable membrane performance to produce a permeate (95 % of the flow) with low organic content and a concentrate (5 % of the flow) with high organic content that can be treated anaerobically to produce biogas.
- Based on the results and discussions between VITO, IIT Kanpur and Ion Exchange, it was decided that not only should be focused on the use of an anaerobic digester, but it would also be very worthwhile to assess the approach for a UASB reactor. UASB reactors are installed throughout India, after being heavily promoted as a wastewater technology during the 1990s – but performances have been poor (compared to Europe). It is therefore proposed to evaluate and validate the approach of feeding the concentrated wastewater rich in solids from the IPC Filtration Module to a UASB reactor at a lab-scale at IIT Kanpur. In addition, treatment of 5 % of the initial primary settled sewage flow as concentrate, when upscaled to an operational system, would require an extremely large anaerobic digester to deal with the feed, so tests on thickening the concentrated wastewater using flocculation and sedimentation are planned in the next phase.



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**References: measuring report references:** Master Thesis Ruben Vingerhoets UA, Andicos: a new approach to treat wastewater and waste in the Ganga River basin, case study in Kanpur, India (2020)<sup>1</sup>.

#### 2.4.2. FILTRATION EXPERIMENTS PERIOD 2021-2022 (LOW CONCENTRATION SEWAGE)

Due to the COVID-19 pandemic, the lab tests were halted for several months. In the period 2021-2022, tests were restarted at similar conditions as in subtask 3.2.1. It was observed that the COD concentration of the primary settled sewage was drastically reduced compared to the 2020 tests (reduction from a COD average of 1,139 mg/L to 300 mg/L). Critical flux experiments with the primary settled sewage showed an optimal filtration flux range of 15 - 25 L/h-m<sup>2</sup>, which is comparable with the results from the previous subtask. The long-term lab experiments were done using a slightly lower membrane flux to ensure stable membrane operation to be able to focus on concentration factor and water quality.

Table 2-3: Operational parameters of IPC filtration.

	<b>Filtration</b>	<b>Backwash</b>	<b>Relaxation</b>	<b>Net / Total</b>
<b>Flux</b>	15 Lmh	22.5 Lmh	0	11.36 Lmh
<b>Duration</b>	9 min	40 s	20 s	10 min

#### Conclusions:

- The performance of lab-scale IPC membranes was assessed with sewage collected after the primary settling tank of STP Jajmau, Kanpur. Aeration, being a necessary part of the membrane process, was done at 0.5 m<sup>3</sup>/h for each membrane. In 35 days of operation, COD reduced from 250 mg/L in the influent to 40 mg/L in the permeate. Total Suspended Solids were found to be below the detection limit in the effluent. Ammoniacal nitrogen was reduced from 34 mg/L in the influent to < 5 mg/L in the effluent. Due to continuous aeration for the scouring of membranes and high (Sludge) Retention Time (~12 days), ~70% of the COD was lost because of biodegradation. The TSS of the concentrated sewage increased from 250 to 2600 mg/L in 20 days (~10-fold increment). During this phase, the membranes could safely run continuously for 10-15 days without any type of physical/chemical cleaning.
- The change in the sewage characteristics had a major impact on both the composition of the concentrate stream and the unwished biodegradation that takes place in the membrane reactor. This will result in lower biogas production in the anaerobic treatment step, having a negative impact on the Andicos business model.

**References:** Integrated Permeate Channel membranes: Evaluating longevity and establishing chemical cleaning protocols (Research article in draft - By IIT Kanpur and VITO)

<sup>1</sup>[https://www.scriptiebank.be/sites/default/files/thesis/2020-10/Thesis.Ruben\\_.Vingerhoets%20%281%29%20%282%29.pdf](https://www.scriptiebank.be/sites/default/files/thesis/2020-10/Thesis.Ruben_.Vingerhoets%20%281%29%20%282%29.pdf)



### 2.4.3. MEMBRANE CLEANING EXPERIMENTS

Cleaning experiments were done to collect information on membrane cleaning procedures to be used during pilot testing. To optimize the membrane cleaning process and frequency, different concentrations of sodium hypochlorite were used. 100 - 900 ppm active chlorine solutions were used for chemical cleaning.

#### Conclusions:

- Based on the extent of membrane fouling, the concentration of chlorine was chosen for checking the efficiency of chemical cleaning. Physical cleaning with tap water is suggested when transmembrane pressure increases to 0.5 bar for the first time. For the next cycle, 300 ppm chlorine was suitable for achieving a notable reduction in TMP. 900 ppm dose was used when 3-4 physical rinsing operations have been performed consecutively.

**References:** Integrated Permeate Channel membranes: Evaluating longevity and establishing chemical cleaning protocols (Research article in draft - By IIT Kanpur and VITO)

### 2.4.4. EXPERIMENTS AT DECREASED SRT FOR REDUCING BIODEGRADATION

To reduce the undesired biodegradation of the primary settled sewage in the membrane tank (see subtask 3.2.2) during the filtration step, a set of experiments were performed at low SRT (~ 8 h) by continuous removal of concentrate from the bioreactor.

#### Conclusions:

- After reducing the SRT from ~12 days to ~8 hours, the biodegradation losses were still high due to the deposition of biofilm on reactor walls. For reducing the loss of organics due to biodegradation, low SRTs are recommended (2-4 h). Such low SRT is possible when compact reactors are used where most of the volume is occupied by the membranes and accessories.

**References: measuring report references:** Critical comparison of the performance of IPC membranes in different dissolved oxygen concentrations and evaluating its effect on biodegradation (Research article to be prepared by IIT Kanpur and VITO)

### 2.4.5. EXPERIMENTS USING NITROGEN GAS FOR AERATION OF MEMBRANES FOR REDUCING BIODEGRADATION

In the next series of experiments, the effect of the absence of oxygen on biodegradation in the membrane tank was studied by replacing air for the membrane with nitrogen gas.

#### Conclusions:

- The loss of organics reduced from 70% (in air) to 40% (in nitrogen). Nitrification was completely inhibited and the increase of suspended solids concentration in the retentate



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was found to be much higher due to reduced losses. The average COD of permeate was 40 mg/L.

**References: measuring report references:** Critical comparison of performance of IPC membranes in different dissolved oxygen concentrations and evaluating its effect on biodegradation (Research article to be prepared by IIT Kanpur and VITO).

## 2.5. SAFETY MEASURES AND TROUBLESHOOTING

For the laboratory experiments, no extra measurements or troubleshooting issues have been encountered.



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## CHAPTER 3 TECHNOLOGY: AQUA-Q OZONATION

### 3.1. ADMINISTRATIVE INFORMATION

Technology name:	Aqua-Q
Location of lab and pilot:	Sweden
Organization(s):	Aqua-Q
Contact information:	Lead: Sudhir Chowdhury, Ulla Chowdhury Contact: Sudhir Chowdhury, Ulla Chowdhury

### 3.2. BRIEF TECHNOLOGY DESCRIPTION

AQUA-Q is a Cleantech Swedish SME with extensive applied research experience from Municipal and industrial water & wastewater treatment plants and has developed a novel ozone polishing technology to remove pharmaceutical residues and pathogens in water.

Under this project, the AQUA-Q technology was evaluated directly as a pilot-scale system. Thus, results from the testing are presented in Work Package 5. For further information regarding this technology, see Deliverables 5.3, 5.4 and 5.5.



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## CHAPTER 4 TECHNOLOGY: CLEANBLOCKS

### 4.1. ADMINISTRATIVE INFORMATION

Technology name:	Cleanblocks	
Location of lab and pilot:	Delft, The Netherlands	New Delhi, India
Organization(s):	TU Delft	
Contact information:	Lead: Merle de Kreuk	Lead:
	Contact: Steef de Valk, Antonella Piaggio	Contact: Shaikh Ahammad

### 4.2. BRIEF TECHNOLOGY DESCRIPTION

Cleanblocks are a mineral wool filtering material. According to Wedge R. and Abt E. mostly the slag that is obtained during the reduction of Iron ore to pig iron is used to produce mineral wool fibres. Chemically the mineral wool is composed of aluminium silicates, magnesium, and calcium along with lesser amounts of other oxides.

Different mineral-based wools such as glass wool, rock/stone wool, and alkaline earth silicate (AES) wool exist. The mineral wools used with water are mostly rock wools, which are chemically composed of roughly 38-46% SiO<sub>2</sub>, 15-20% CaO, 15-19% Al<sub>2</sub>O<sub>3</sub> and 6-9% Fe<sub>2</sub>O<sub>3</sub> (Campopiano *et al.*, 2014). Although mineral wool is thought to be inert, a study on the bio solubility of mineral wools showed the dissolution of silicon (Si) and calcium (Ca) at different pH values, inducing calcium-phosphate (Ca-P) precipitates (Campopiano *et al.*, 2014). According to an assessment of mineral wool as a support material for on-site sanitation, the mineral composition will influence the interaction between the material and the wastewater content: suggesting interaction between the mineral wool and the wastewater (Wanko *et al.*, 2016).

Mineral wool shows a lacunar structure where the matter was structured as scattered balls and crossed fibres. This means that the contact area of the wastewater with the biofilm is high, enhancing the removal efficiency and rate is high. Furthermore, it was proven that the saturated water content of mineral wool was almost twice as high as that from the natural granular media such as silt, clay, loam, and sand. Due to this the water retention capacity is high, which directly influences the hydraulic residence time in the filter positively. Finally, due to the mineral composition of the mineral wool, its degradation rate is slow.



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Figure 4-1: Cleanblocks material, made from mineral wool. The cubes shown in this image are around 1 cm<sup>3</sup>.

### 4.3. TECHNOLOGY-SPECIFIC SUMMARY AND CONCLUSIONS

Based on the material properties of mineral wool an initial design of the Cleanblocks filter was assessed and evaluated on a lab-scale. Initial results excellent TSS removal with short (<1h) run times. Due to the high porosity, the system is prone to clogging and therefore not suited for in-drain applications. The design was improved and evaluated further with drain water in Delhi to further improve run times and TSS removal. The final design has been made and a blueprint is available. Estimated run times are based on the initial design and extrapolated. Run times are between 10 and 15 days with TSS concentrations at 100 to 250 mg/L.

### 4.4. PROCESS OVERVIEW

The Cleanblocks system (figures below) was designed to remove solids from the photo-activated sludge (PAS) effluent.



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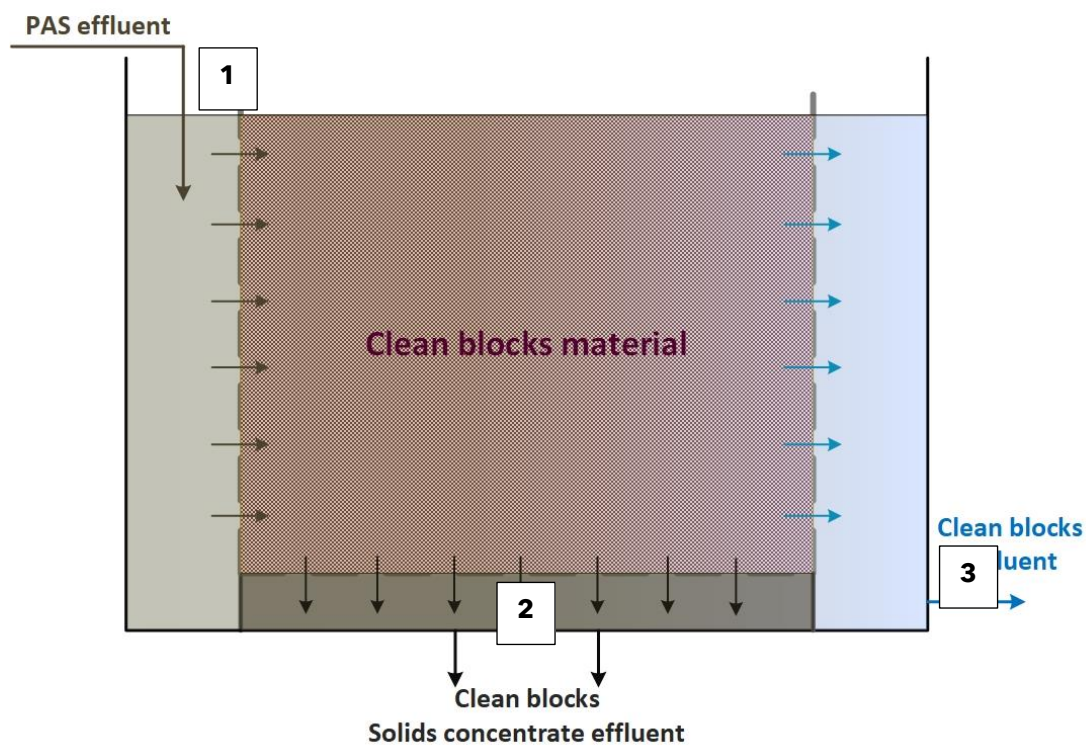


Figure 4-2: Process overview. The arrows indicate the water flows: Influent (1), effluent (3) and drainage points (2) are shown. The red mesh indicates clean block cubes.



Figure 4-3: Cleanblocks system assessed at TU-Delft lab facilities. The system contains a slab sheet of mineral wool, used for evaluating the filtration capacity of the system.



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Figure 4-4: Cleanblocks system evaluated at the Barapullah site (New Delhi). The influent tested was effluent from the PAS system, at different flow rates. This system was to investigate the connections between the lab-scale system to the pilot-scale system (deliverable 3.2-3).

The influent to the Cleanblocks system passes through the Cleanblocks material, either slab or in the form of blocks, as seen in both figures above. This material filters the suspended particles present in the influent allowing it to have a better-quality effluent. The particles retained by the mineral wool material can either stay attached to it or settle down. Thus, drainage points are located at the bottom of the system to remove solids surplus from it.

- TUD Influent was stored in a 200 L, continuously mixed storage tank and fed using a peristaltic pump to the setup. At a flow of 150 L/d.
- TUD setup had a total volume of 30 L and was evaluated for inflow of up to 150 L/d (100 ml/min)
- Barapullah drain influent (PAS effluent) was stored in a 100 L tank, and continuously mixed.
- The bench-scale setup had a total volume of 60 L, it was directly fed by the effluent of the PAS system, at flows between 50 to 150 ml/min.



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#### 4.5. FACTSHEET: CORE PARAMETERS ON WATER TREATMENT AND RESOURCE RECOVERY

Table 4-1: General factsheet: Cleanblocks

Parameter		Units	Value	Comments
Influent type(s)	Tested at TUD: Diluted activated sludge /digestate. Drain water			
Influent quality range	TSS	mg/L	<150	TSS is the key design aspect
	Turbidity	NTU	<100	
	pH	-	6 – 7	
Effluent quality range	TSS	mg/L	1 – 10	
	Turbidity	NTU	10 – 20	
	pH	-	7 – 7.5	pH increases due to the buffering capacity of mineral wool
Technology characteristics	Turbidity	NTU		
	pH	-		
	Temperature range	°C	15 - 30	
	System operational volume	L	30	Delft lab
Technology-defining Rates/ Ranges	Flow	L/d	150	
	Filtration velocity	m/h	0.3	Based on Wegelin et al. [1]
	Hydraulic retention time	h	3	At 1m filter length



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	Solids retention time		$\infty$	TSS is filtered out
Maintenance	Cleaning frequency	weeks	6	Estimation based extrapolated lab performance
Resource recovery				No phosphate recovery is possible.

Influent type(s)	Tested at the Barapullah drain: PAS effluent from monsoon time			
Influent quality range	TSS	mg/L	<150	TSS is the key design aspect
	Turbidity	NTU	10	
	pH	-	7	
	TS	mg/L	600	
	TDS	mg/L	500	Most of the solids are dissolved
	COD	mg/L	80-115	
	NO <sub>3</sub> <sup>-</sup>	mg/L	4	
	PO <sub>4</sub> <sup>2-</sup>	mg/L	3	
	T	°C	30-32	
Effluent quality range	TSS	mg/L	1 - 10	
	Turbidity	NTU	< 2	
	pH	-	7	
	TS	mg/L	400	



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	TDS	mg/L	400	Most of the solids are dissolved
	COD	mg/L	100-60	
	NO <sub>3</sub> <sup>-</sup>	mg/L	4	No N removal was observed
	PO <sub>4</sub> <sup>2-</sup>	mg/L	3	No P removal was observed.
Technology characteristics	Turbidity	NTU		
	pH	-		
	Temperature range	°C	15 - 30	
	System operational volume	L	60	Barapullah drain
Technology-defining Rates/ Ranges	Flow	L/d	50-150	
	Filtration velocity	m/h	0.3	Based on Wegelin et al. [1]
	Hydraulic retention time	h	0.8 - 1.5	At a water table height of around 4 cm
	Solids retention time		∞	TSS is filtered out
Maintenance	Cleaning frequency	weeks	6	Estimation based extrapolated lab performance
Resource recovery				No phosphate recovery is possible.



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## 4.6. LABORATORY EXPERIMENTS PER WASTEWATER TREATMENT AND REUSE TECHNOLOGIES

### 4.6.1. SUMMARY AND CONCLUSIONS OF THE TASK-SPECIFIC RESULTS

#### → Lab-scale operation for water treatment

##### Summary:

- Nutrient removal:
  - The nutrient removal capabilities of mineral wool and biofilm were evaluated using vertical 30 L PVC columns and mineral wool cubes in continuous operation. Nitrification and denitrification were assessed. The results showed that nitrification was observed while denitrification did not occur due to the high oxygen concentration in the feed. COD removal was observed.
  - Improved tests with fully matured biofilm are planned and will be assessed in different flow-through modes.

##### Conclusions:

- Further research is needed to adequately determine the nitrogen removal potential of the Cleanblocks system.

**References:** Practical lab reports WTR course 2019.

#### → Process and design optimization

##### Summary:

- Mineral wool characterisation:
  - The initial hydrolytic conductivity and filtration capabilities of several mineral wool densities, fibre orientations and filter orientation (vertical or horizontal) were determined for TSS concentrations between 130 and 450 mg/L. The constant head test was used for the assessment. The result showed that mineral wool slabs are highly efficient in TSS removal (>90%) and prone to clogging. A maximum run time of 1 hour was observed irrespective of mineral wool thickness.
  - Elemental analysis (XRF and ICPOES) of the mineral wool showed that the main constituents of mineral wool were composed of silicon (34%), calcium (33%), aluminium (14%), magnesium 9% and iron (8%).
  - Mineral wool acts as a buffer in acidic or basic environments.
- Design:
  - The initial lab design in Delft, (filter length of 30 cm) using mineral wool cubes instead of slab showed improved solids settling and thus removal and run times.
  - An optimised and larger design (filter length of 60 cm) was assessed with Barapullah drain water. Based on the initial results with TSS concentrations of 100 to 400 mg/L, the head increase rate (cm/h) was determined and used as input for the final design. It was further shown that run time is directly related to TSS concentrations.



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**Conclusions:**

- Cleanblocks have a high filtration rate and hydraulic conductivity.
- Cleanblocks technology is not suited for in-drain treatment due to the sensitivity of clogging.
- The potential of a final design must be determined in practice.

**References:** BSc report, Anupam Sani, "Primary wastewater treatment using mineral wool filters" 2019

→ **Research interactions between pilot and lab-scale for water treatment****Summary:**

Tests were conducted for 21 days at the Barapullah site. These tests aimed to assess the filtration capacity of the unit, and mainly the removal of solids. Three flows were chosen for the assessment: 50, 70 and 100 ml/min (75-150 L/d). Experiments were run in triplicates, and samples were also taken in triplicates. Final TSS removal for each flow can be observed in the table below.

Table 4-2. Influent (PAS effluent) and effluent total suspended solids (TSS) concentration, from the Cleanblocks system located at the Barapullah drain, when influent flow was set at 150 L/d.

Flow	Influent TSS (g/L)	Effluent TSS (g/L)	Removal %
50ml/min	0.066	0.038	42.42
70ml/min	0.066	0.036	45.45
100ml/min	0.012	0.004	66.66

No changes in nutrient content ( $\text{NO}_3$  and  $\text{PO}_4$ ) were observed for all assessed flows. Furthermore, while suspended solids were removed, Total dissolved solids (TDS) concentration and effluent conductivity increased. This is because the mineral wool is not able to retain dissolved matter. The retention of suspended solids and water has the counter effect of increasing the overall concentration of dissolved matter in the effluent (while the total load might be considered stable). Results from the runs at 150 L/d can be observed in the figures below, depicting changes in conductivity and pH (Figure 4-5), solids content (Figure 4-6) and nutrient content (Figure 4-7).



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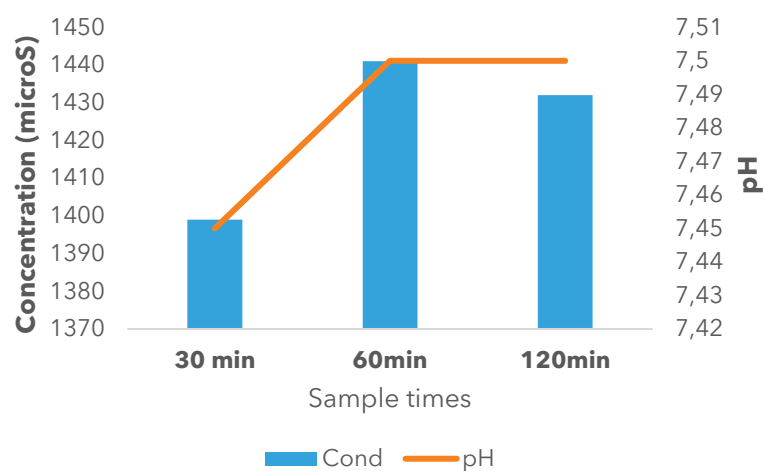


Figure 4-5: Conductivity and pH of the effluent of the Cleanblocks system at the Barapullah site, at different sample times and under an influent flow of 150 L/h.

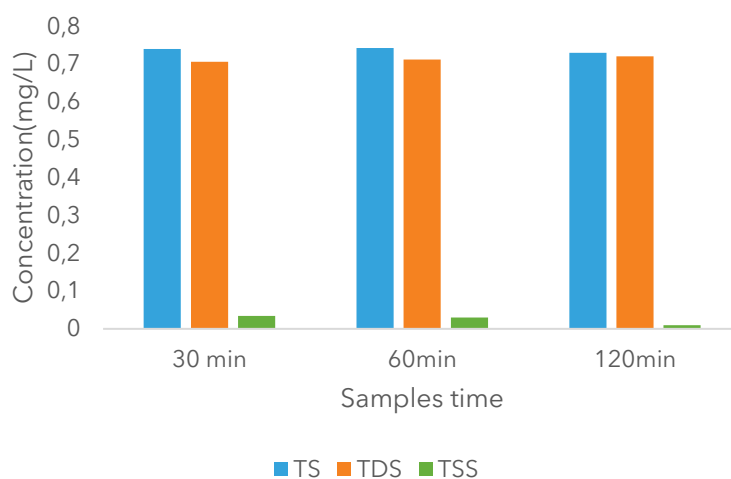


Figure 4-6: Total solids (TS), total dissolved solids (TDS) and total suspended solids (TSS) concentrations in the effluent of the Cleanblocks system at the Barapullah site, at different sample times and under an influent flow of 150 L/h.



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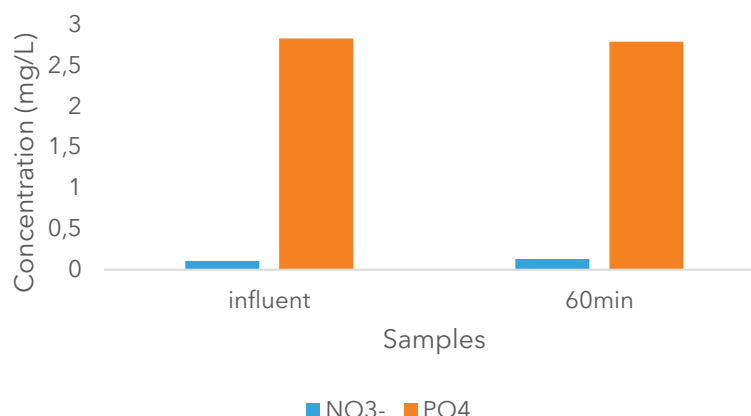


Figure 4-7: Nitrate and phosphate concentration in the influent and effluent of the Cleanblocks system at the Barapullah site, under an influent flow of 150 L/h, and a sampling moment after 60 minutes of running the system.

#### Conclusions:

- Throughout the running hours, an increase in the water level was not observed. This also indicated that there was no clogging, and a breakthrough was not achieved.
- The TSS removal improved with an increase in flow rate and filtration time.
- In the current study 100ml/min (150L/d) was the maximum flow studied. However, a breakthrough was not achieved. It also points towards the fact that flow rates higher than 100ml/min should be evaluated.
- At the end of the study, biofilm growth was observed in a handful of blocks. Nevertheless, the integrity of the blocks was maintained.
- No P or N removal was achieved. This is aligned with results from the lab-scale system. Thus, no P recovery can be achieved with this system.
- Due to expenses of operation and maintenance of the Cleanblocks, mainly conditioned by the needed change of the filter material due to clogging, and the lack of P recovery, this system is not recommended to upscale to pilot or full scale. Further research should be considered on the 60 L bench-scale system at the Barapullah site to establish the breakthrough point.

**References:** Barapullah July 2023 tests report. R. Srinivas & A. Piaggio.

## 4.7. TROUBLE SHOOTING AND PROCESS SAFETY

This section contains information on safety, operational and troubleshooting aspects that have been encountered or are foreseen during the lab-scale tests.

### 4.7.1. SAFETY MEASURES

- Fibres: Wear gloves and a mouth cap when handling dry mineral wool.
- Filter clogging: leak tray or feedback loop to influent pump to prevent flooding.



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#### **4.7.2. TROUBLESHOOTING**

- The Cleanblocks system should be positioned above the ground level to avoid issues due to flooding (as expected at the Barapullah site). All electric materials (such as pumps) should be located safely, far away from water.
- In case of overflow, a buffer tank should be located after the system.



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## CHAPTER 5 TECHNOLOGY: CONSTRUCTED WETLANDS PLUS

### 5.1. ADMINISTRATIVE INFORMATION

Technology name:	Constructed Wetland Plus	
Location of lab and pilot:	Bochum, Germany	STP Jajmau Kanpur, India
Organization(s):	HBO	IIT Kanpur
Contact information:	Lead: Christian Kazner	Lead: Purnendu Bose
	Contact: Luca Ofiera	Contact: Auchitya Verma

### 5.2. BRIEF TECHNOLOGY DESCRIPTION

The modified Constructed Wetland (CW) technology combines vertical flow constructed wetlands with adsorptive elements such as granular activated carbon and specific sorbents for enhanced heavy metal removal. The GAC serves as an adsorbent, particularly of recalcitrant organic compounds, in particular trace organic compounds, and supports the growth of specialised bacteria to improve their biodegradation. Zeolite is particularly suitable for the removal of inorganic compounds such as HMs. Biochar enhances plant growth, water retention, microbial activities and complexation reactions. Limestone will further be added to control the pH and minimize the remobilization of HMs. The vertical flow constructed wetland (VFCW) will be composed of several layers consisting of gravel, sand, and sorbents planted with *Cana indica*. The effluents from the ANDICOS system and the SFD-MBR will serve as the influent of the CW+ units. Perforated tubes distribute the influent evenly onto the wetland which then flows vertically through the various substrate layers to retain and degrade the different pollutants.

### 5.3. TECHNOLOGY-SPECIFIC SUMMARY AND CONCLUSIONS

To identify the most promising adsorbents, the adsorption capacities for organic and inorganic compounds of various natural zeolites and granular activated carbons (GAC) were investigated in batch tests. Since the solubility of most heavy metals is strongly dependent on the prevailing pH value, precipitation tests were conducted at different pH values. To evaluate these results, the speciation of the HMs as a function of pH was simulated by using the software Hydra/Spana. Based on these pre-tests the best adsorbents were selected for the subsequent rapid small-scale tests (RSSCT).



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## 5.4. PROCESS OVERVIEW

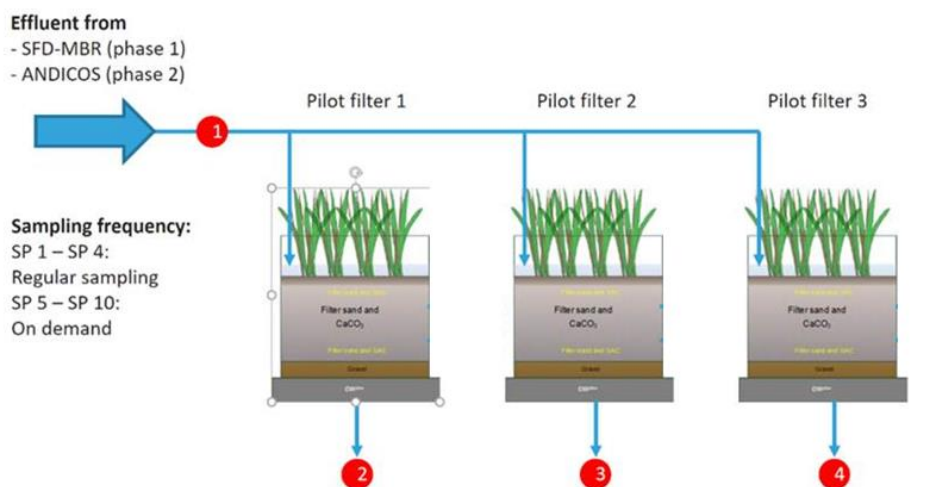


Figure 5-1: Process flow diagram of the Constructed Wetlands Plus the Constructed Wetlands Plus.

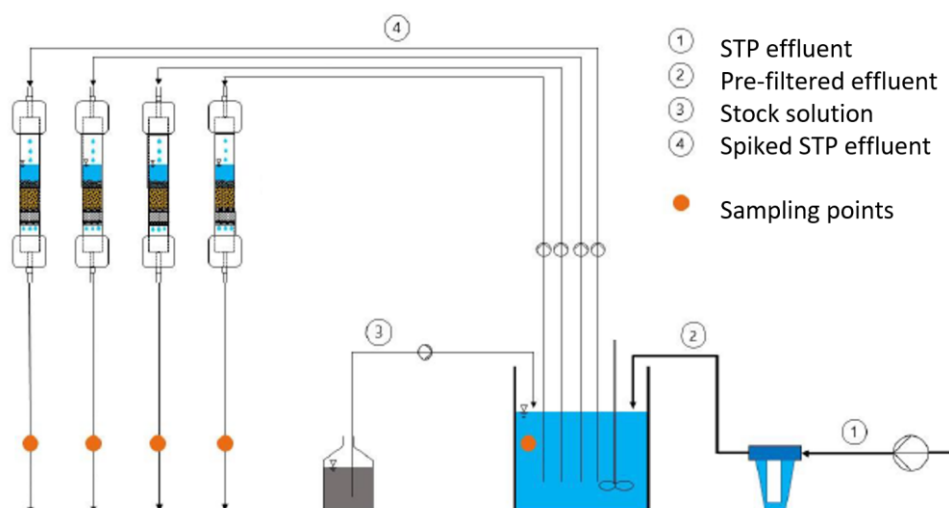


Figure 5-2: Experimental setup of column tests.

The laboratory tests aimed to identify suitable adsorbents and to anticipate the adsorption processes that take place in the constructed wetland pilots by downscaling in a significantly shorter time. For this purpose, Sewage treatment plant (STP) effluent was collected and prefiltered to avoid subsequent clogging. The tests conducted at FHNW were conducted with heavy metal spiked STP effluent, and the tests at IITK were conducted with STP effluent without spiking heavy metals. The water to be treated is pumped via peristaltic pumps into the glass columns filled with the respective adsorbents and then passes through them from top to bottom. The treated effluent is then collected and analysed.



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## 5.5. FACTSHEET: CORE PARAMETERS ON WATER TREATMENT AND RESOURCE RECOVERY

Table 5-1: General factsheet: Constructed Wetlands Plus

Parameter		Units	Value
Influent type(s)	Filtered STP Jajmau effluent		
Influent quality	pH	-	$8.03 \pm 0.2$
	TDS	mg/L	$1516 \pm 285$
	Conductivity	$\mu\text{S}/\text{cm}$	$2097 \pm 399$
	DOC	mg/L	$20.77 \pm 7.89$
	SAC	1/m	$0.554 \pm 0.259$
	NH <sub>4</sub> -N	mg/L	$70 \pm 10$
	Cd	mg/L	$0.006 \pm 0.002$
	Cu	mg/L	$0.145 \pm 0.076$
	Cr total	mg/L	$0.041 \pm 0.016$
	Fe	mg/L	$0.446 \pm 0.152$
	Mn	mg/L	$0.027 \pm 0.010$
	Ni	mg/L	$0.008 \pm 0.005$
	Pb	mg/L	$0.001 \pm 0.001$



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	Zn	mg/L	0.034 ± 0.017
Effluent quality	pH	-	7.44 - 8.31
	TDS	mg/L	1137 - 14575
	Conductivity	µS/cm	1696 - 2217
	DOC	mg/L	17.79 - 35.2
	SAC	1/m	0.279 - 0.782
	Cd	mg/L	0.003 - 0003
	Cu	mg/L	0.05 - 0.28
	Cr total	mg/L	0.02 - 0.05
	Fe	mg/L	0.55 - 0.87
	Mn	mg/L	0.01 - 0.04
	Ni	mg/L	0.005 - 0.006
	Pb	mg/L	< LOQ
	Zn	mg/L	0.01 - 0.02
Technology-defining Rates/ Ranges	Column diameter	cm	1
	Filter bed height	cm	4.38
	Flow volume	ml/min	7.48



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	Empty bed contact time (EBCT)	s	27.6
	Filtration velocity	m/h	5.7
Influent type(s)	Synthetic STP ARA BIRS effluent		
Influent quality	pH	-	8.42 ± 2
	COD	mg/L	21
	DOC	mg/L	7.23
	N <sub>tot</sub>	mg/L	7.6
	TSS	mg/L	6.1
	P <sub>tot</sub>	mg/L	0.62
	Cd	mg/L	0.006
	Cu	mg/L	0.029
	Cr	mg/L	0.020
	Fe	mg/L	0.048
	Mn	mg/L	0.068
	Ni	mg/L	0.046
	Zn	mg/L	0.061
Technology-defining Rates/ Ranges	Column diameter	cm	1
	Filter bed height	cm	10



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Flow volume	ml/min	15.67
EBCT	s	30.08
Filtration velocity	m/h	11.97



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## 5.6. LABORATORY EXPERIMENTS PER WASTEWATER TREATMENT AND REUSE TECHNOLOGIES

### 5.6.1. SUMMARY AND CONCLUSIONS

#### → Lab-scale operation for water treatment

##### Lab tests at FHNW:

For the lab tests, STP ARA BIRS (Switzerland) effluent was synthetically modified to obtain water contaminants similar to the effluent of the STP Jajmau. 500 ml of the synthetic wastewater was filled into different Schott bottles with different concentrations of adsorbents (GAC and zeolites). The flasks were shaken at 150 rpm for exactly 7 days. Room temperature was constant at  $21^{\circ}\text{C} \pm 1$ . Samples were then filtered with  $0.4\ \mu\text{m}$  glass fibre filters to remove particulate matter and analytical measurements were taken. Samples were stored at  $4^{\circ}\text{C}$  in steamed Schott bottles when measurements could not be performed immediately.

To perform the RSSCT, a set-up had to be established to ensure that the flow rates were sufficient and continuous and that the quality of the feed water remained the same during all experimental phases. To overcome the back pressure created by the suspended solids forming an impenetrable cake layer in the column during the experimental phase, a pre-filtration stage was added, and sufficient pressure had to be built up. The water was pre-filtered with a  $10\ \mu\text{m}$  winding filter before entering the RSSCT to avoid clogging the column. The RCCST unit was also equipped with a  $\text{CaCO}_3$  cartridge filter to retain excess HM in the effluent. This ensured that once the adsorbents had reached a breakthrough, the water was still treated before it was discharged into the sewerage system.

Thus, the conclusions of the lab tests at FHNW are the following:

##### Batch tests:

- GAC loading rates of up to 30 mg/g for DOC were reached.
- Good adsorption of Mn, Ni and Zn
- Adsorption for Cu, Cd, Fe and Cr generally does not exceed 50%

##### RSSCT:

- Removal of up to 99% for Fe, 90% for Cr, 50 % for Cd & Zn but only about 20% for Cu over the whole treatment period of 20,000 bed volumes at an EBCT of 0.5 minutes
- Ni removal was only observed at the beginning of the experiment followed by a fast stagnation of removal, which replaced the initial bad removal of Mn to a removal above 95 % after reaching 10,000 bed-volumes.

##### Lab tests at HBO and IITK:

Since some heavy metals tend to precipitate even at lower pH values, precipitation experiments were conducted with sewage plant effluent of the STP Emschermündung in Dinslaken, to which nitrate salts of the heavy metals Cd, Cu, Cr, Ni, Pb and Zn have been added at a concentration of 5 mg/L each. The pH was adjusted with sodium hydroxide ( $\text{NaOH}$ ) and nitric acid ( $\text{HNO}_3$ ) to four



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different values ranging from acidic to neutral (4, 5, 6, 7). The HM concentrations were adjusted to 5 mg/L. In addition to a blank sample, 250 mg of zeolite < 45 µm was added to the solution.

To evaluate the most promising adsorbents for the column tests, batch tests were conducted. The adsorption capacity for heavy metals and dissolved organic carbon (DOC) was determined using the same HM-spiked STP effluent as in the precipitation pre-tests. Adsorbent concentration was set to 50, 100, 250, 500 and 1000 mg/L with a particle diameter < 45 µm.

Based on the results of the adsorption batch assesses, two zeolites and two GACs were selected. Different mixing ratios of the selected adsorbents were used to conduct the experiments. To avoid clogging in the glass columns, the used primary effluent from the STP Jajmau in Kanpur, India, was pre-filtered by a hollow fibre membrane (pore size: 0.1 µm). The 10 mm diameter columns were filled with different adsorbents and backing support layers, consisting of two filter plates (100 µm), glass wool, and glass beads to create a proper flow regime and prevent an outwash of the adsorbents. Feed was continuously with a flow rate of 7.48 mL/min.

Thus, the conclusions of the lab tests at HBO and IITK are the following:

Batch tests:

- DOC removal rates with increasing adsorbent doses ranged between 85.3% - 96.2% for the different tested activated carbons.
- DOC removal by zeolite does not exceed 31.8% at an adsorbent dose of 100 mg/L, even at higher concentrations.
- Selectivity in batch tests for zeolite was: Pb > Cr > Cu > Cd = Ni > Zn
- Negative loading rates for Zn occurred for all zeolites below adsorbent doses of 250 mg/L.

RSSCT:

- The highest mean removal was 52.4% for Cu, 2.1% for Fe, 29.2% for Mn, 44.6% for Zn, 17.2% for Ni, 56.6% for Cd, 35.2% for Pb and 14.1% for Cr
- A higher zeolite ratio had no positive effect on the removal of most HMs.
- Lower removal rates compared to the batch test findings can be explained by a shorter contact time and a different wastewater composition, especially by higher ammonium concentrations in the STP Jajmau effluent.

**References: measuring report references:** M.Sc. thesis, Martin Huspeka "Assessment of adsorbents for advanced removal of trace organics and trace metals from sewage treatment plant effluents in India."

M.Sc. thesis, Andreas Bauer "Adsorption of heavy metals on zeolites and activated carbon for the treatment of sewage plant effluents."

Conference Paper, Ofiera et al. (2023) Removal of heavy metals in modified constructed wetlands using activated carbon and zeolite. 13th IWA International Conference on Water Reclamation and Reuse, 15-19 January 2023, Chennai, India

### → Process and design optimization

The selection of the most suitable adsorbents was the focus of the laboratory tests. the tests had no direct influence on the general design or the process itself. The aim was to predict the long-term adsorption behaviour in the soil filter in a brief time by scaling down the pilot to anticipate the breakthrough behaviour.



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**→ Research interactions between pilot and lab-scale for water treatment**

By using RSSCT, a prediction can be made about the breakthrough behaviour of the various pollutants in a significantly shorter time and with a significantly lower total water volume. However, it must be considered that the column tests cause precipitation and thus clogging of the columns due to the enrichment of the wastewater with heavy metals, which leads to additional problems due to the small diameter of 10 mm. In the future, the focus will also be on the elimination of organic micropollutants.

**5.7. TROUBLE SHOOTING AND PROCESS SAFETY****5.7.1. SAFETY MEASURES**

*Brief description of critical safety aspects and possibilities for mitigation*

- Generally, all experimental set-ups and handling of any substance were pre-discussed with the laboratory chief of security.
- For safety reasons, the infrastructure usage was only permitted when a second person was in the same laboratory.
- In all laboratories, the wastewater treatment plant, and the process technology centre, as well as in all the other labelled lab zones, personal protective equipment (i.e., laboratory coat, protective glasses) has been used appropriately.
- During the handling of any hazardous substance disposable gloves for laboratory use have been worn
- During the development of the synthetic wastewater and while handling MP's, HM's and other potentially hazardous substances, their toxicity, solubility, evaporation point, and corresponding hazard and precautionary statements of the substances were taken into account.
- Efforts have been taken to minimize waste generation, and potential substitution of hazardous substances has been undertaken whenever possible. After working with those substances, surfaces were cleaned, and if necessary personal protective equipment was changed.

**5.7.2. TROUBLESHOOTING**

- Clogging issues during the lab-scale column tests are a problem due to the precipitation of heavy metals even at lower pH values.
- pH values need to be adapted when working with synthetic solutions to prevent precipitation.
- A proper pre-filtration unit is necessary to avoid clogging.



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## CHAPTER 6 TECHNOLOGY: PHOTO ACTIVATED SLUDGE

### 6.1. ADMINISTRATIVE INFORMATION

Technology name:	Photo-Activated Sludge	
Location of lab and pilot:	Lab - IHE Delft, The Netherlands	Pilot - New Delhi, India
Organization(s):	IHE Delft	IITD
Contact information:	Lead: Eldon Raj, Peter van der Steen and Mahmoud M. Habashy	Lead: Shaikh Ziauddin Ahammad and Ashish Lohar
	Contact: Eldon Raj	Contact: Shaikh Zia

### 6.2. BRIEF TECHNOLOGY DESCRIPTION

Microalgae-bacteria consortiums have proven to be an environmentally friendly and sustainable alternate to treat wastewater. Besides being a renewable biomass source, microalgae in wastewater treatment are a cost-effective and feasible method for carbon dioxide bio-fixation. The reason for using mixotrophic microalgae to treat wastewater is their capability to use organic and inorganic carbon as well as nitrogen and phosphorus available in wastewater for their growth, which results in the reduction of these substances' concentration. Also, the main benefit of incorporating microalgae for wastewater treatment is their generation of oxygen through photosynthesis, which is essential for the biodegradation of carbonaceous materials by heterotrophic bacteria. The photo-activated sludge (PAS) or the photo-sequencing batch reactor (PSBR) uses the symbiotic relationship between microalgae and aerobic bacteria in an open bioreactor to maximize the oxidation of ammonium and organic carbon using the oxygen produced by microalgae through photosynthesis.

### 6.3. TECHNOLOGY-SPECIFIC SUMMARY AND CONCLUSIONS

In the lab-scale bioreactors, the "survival of the fittest" strategy was used to increase lipid productivity by using microalgae-bacteria consortia in photo-sequencing batch reactors (PSBRs) settings. PSBR 1 was fed with synthetic brewery wastewater during the light period and synthetic liquid digestate during the dark period. PSBR 2 was operated under normal conditions, and it was fed with mixed synthetic brewery wastewater and liquid digestate during the light period. The PSBR 1 had higher COD, TOC, TIC,  $\text{NH}_4^+$  - N, and TN removal rates of 14.0, 1.0, 1.2, 1.2, and 0.9 mg/L.h, compared to PSBR 2 had removal rates of 2.0, 0.3, 0.7, 1.0, and 0.5 mg/L.h, respectively.



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## 6.4. PROCESS OVERVIEW AND FACTSHEETS

### 6.4.1. MICROALGAE-BACTERIA CONSORTIUM

Mixed strains of pure culture microalgae and bacteria were used in this lab research. The mixed microalgae strains were: *Chlorococcum* sp., *Chlorella* sp., *Spirulina* sp., *Anabaena variabilis*, and wild algae species from one of the Delft canals (Figures 6.1 and 6.2). The bacteria culture was collected from an aerobic granular sludge of an existing project that was treating synthetic wastewater at the IHE Delft Institute for Water Education. The microalgae cultures and activated sludge were mixed in a 4:1 v/v ratio to be used as the inoculum, where the activated sludge was added to enhance the settling characteristics of the microalgae during the settling phase.

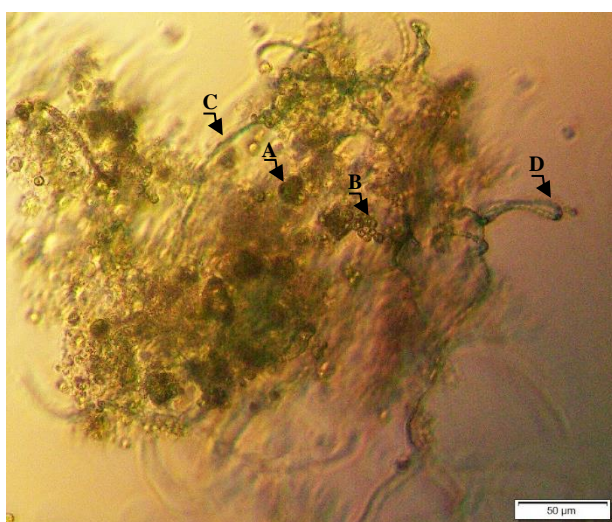


Figure 6-1: Four microalgal species grown in the laboratory. A: *Chlorococcum* sp., B: *Chlorella* sp., C: *Spirulina* sp., D: *Anabaena variabilis* (Scale 50 μm)

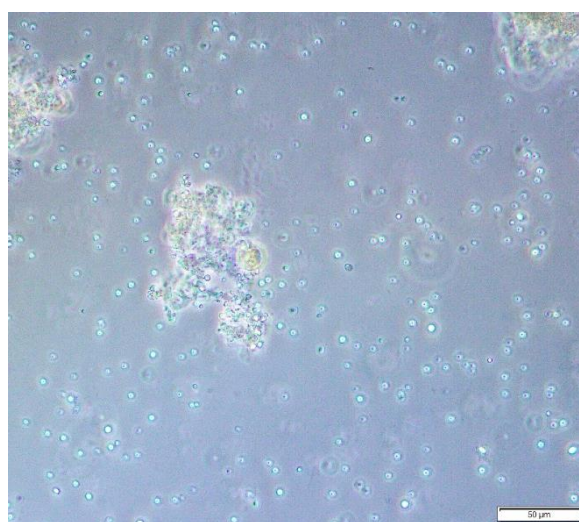


Figure 6-2: Wild algal species from one of the Delft canals (Scale 50 μm)

### 6.4.2. SYNTHETIC WASTEWATER

#### PSBR 1 (Primary bioreactor)

For the startup of PSBR 1, the microalgae-bacteria consortium was cultivated in two modified BG11 liquid media. Medium 1 contained only 100 mg/L of COD, 30 mg/L of TIC, and no nitrogen sources to mimic the effluent wastewater from a brewery factory in the ideal case, and medium 2 contained only 100 mg/L of  $\text{NH}_4^+$  - N and no carbon sources to mimic a liquid digestate effluent from an anaerobic digester in the ideal case.

In the first medium, 100 mg/L of sodium acetate trihydrate ( $\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$ ) and 30 mg/L of sodium bicarbonate ( $\text{NaHCO}_3$ ) were used as carbon sources for the biomass during the light period, and the concentrations were assumed to avoid overgrowth and contamination of heterotrophic bacteria. The rest of the ingredients can be seen in Table 6.1.

In the second medium, 100 mg/L of ammonium chloride ( $\text{NH}_4\text{Cl}$ ) was used as a nitrogen source ( $\text{NH}_4^+$  - N) for the biomass during the dark period, and the concentration was assumed based on



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the assumption that 10% of 2 g/L microalgae volatile suspended solids (VSS) would be the maximum total nitrogen, which was 400 mg/L - TN. The rest of the ingredients can be seen in Table 6-2.

Table 6-1. Modified BG11 medium 1 (synthetic brewery WW).

Parameters	Concentration (mg/L)
NH <sub>4</sub> Cl	0
CH <sub>3</sub> COONa.3H <sub>2</sub> O	100
NaHCO <sub>3</sub>	30
CuSO <sub>4</sub> .5H <sub>2</sub> O	0.03
FeSO <sub>4</sub> .7H <sub>2</sub> O	0.08
H <sub>3</sub> BO <sub>3</sub>	0.37
ZnSO <sub>4</sub> .7H <sub>2</sub> O	0.17
MnCl <sub>2</sub> .4H <sub>2</sub> O	0.24
Na <sub>2</sub> MoO <sub>4</sub> .2H <sub>2</sub> O	0.09
CaCl <sub>2</sub> .2H <sub>2</sub> O	16.12
MgSO <sub>4</sub> .7H <sub>2</sub> O	30.55
KH <sub>2</sub> PO <sub>4</sub>	44.28
Al <sub>2</sub> O <sub>3</sub>	0.20

Table 6-2: Modified BG11 medium 2 (synthetic liquid digestate).

Parameters	Concentration (mg/L)
NH <sub>4</sub> Cl	100
CH <sub>3</sub> COONa.3H <sub>2</sub> O	0
NaHCO <sub>3</sub>	0
NaH <sub>2</sub> PO <sub>4</sub> .H <sub>2</sub> O	50
KH <sub>2</sub> PO <sub>4</sub>	75
MgSO <sub>4</sub> .7H <sub>2</sub> O	25
CaCl <sub>2</sub> .2H <sub>2</sub> O	25



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PSBR 2 (Control bioreactor)

For the startup of PSBR 2, the microalgae-bacteria consortium was cultivated in a modified BG11 liquid medium that mimics a mixed feed supply of synthetic brewery wastewater and synthetic liquid digestate. The mixed wastewater contained 50 mg/L-COD and 50 mg/L  $\text{NH}_4^+$  - N. Table 6.3 shows the rest of the ingredients of the mixed wastewater.

Table 6-3. Modified BG11 medium 3 (synthetic mixed WW).

Parameters	Concentration (mg/L)
$\text{NH}_4\text{Cl}$	50
$\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$	50
$\text{NaHCO}_3$	15
$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	0.015
$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	0.04
$\text{H}_3\text{BO}_3$	0.185
$\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$	0.085
$\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$	0.12
$\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$	0.045
$\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$	20.56
$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	27.78
$\text{KH}_2\text{PO}_4$	22.14
$\text{Al}_2\text{O}_3$	0.1
$\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$	25

**6.4.3. EXPERIMENTAL SETUP**PSBR 1 (Primary bioreactor)

PSBR 1 was operated for 70 days using ideal wastewater as a feed supply. The primary bioreactor (PSBR 1) was operated with 24 h cycles; each cycle was divided into 10 h light and 14 h dark periods. PSBR 1 had a volume of 2.5 L (operating volume of 2 L). During the light period, synthetic brewery WW was supplied to the primary bioreactor, while, during the dark period, synthetic liquid digestate was supplied to the primary bioreactor. The system was regulated using ADI 1030 Bio Controller (Applikon Biotechnology, The Netherlands) and BioXpert software. The pH was maintained at 7.5 by pumping 0.5 M HCl and 1.0 M NaOH, while the temperature was maintained at room temperature ( $22 \pm 5$  °C). During the light period, the light was provided using four 40 W white lamps (Philips, The Netherlands). The schematic of the PSBR 1 setup can be shown in **Error! Reference source not found. 6.3.**





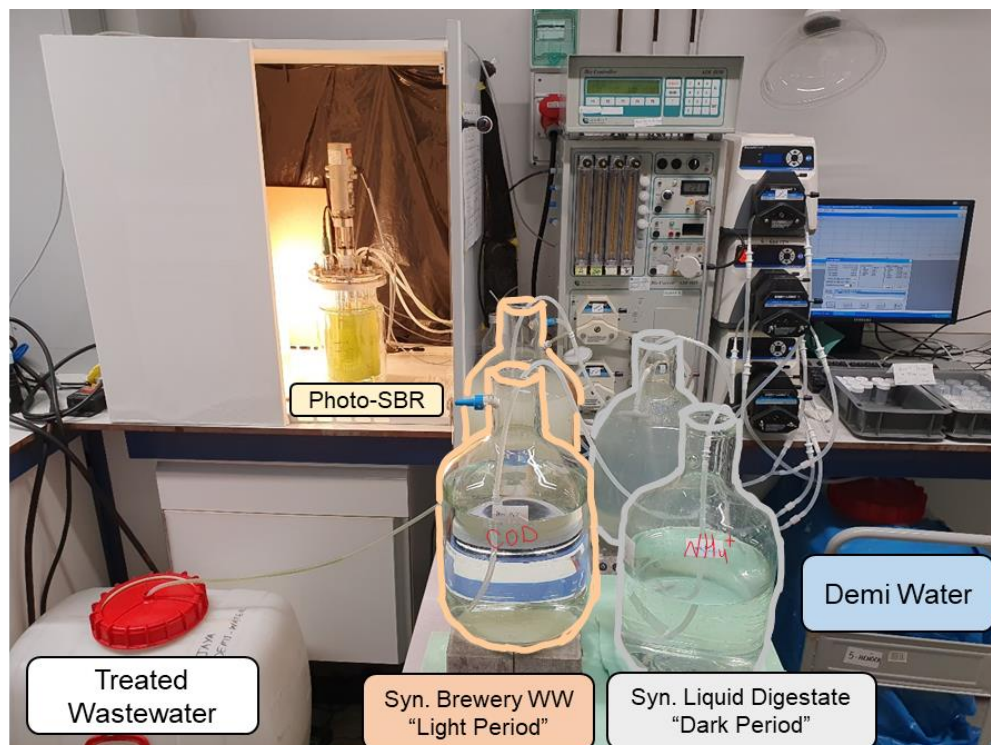


Figure 6-3: Schematic of the PSBR 1 setup.

PSBR 1 was maintained at a hydraulic retention time (HRT) of 2 d, without maintaining a certain solids retention time (SRT). At the beginning of the light period, 1 L of diluted synthetic brewery wastewater that contained 100 mg/L-COD was supplied to the primary bioreactor for 30 min. using a peristaltic pump (Masterflex, USA), and the system was mixed at 200 rpm. The primary bioreactor was left for 8 hours to be continuously homogenized at 200 rpm. Then, it was left for 1 hour for the primary bioreactor content to settle while the mixing was off. Finally, 1 L of the supernatant was withdrawn from the system during the decanting phase for 30 min. Furthermore, at the start of the dark period, 1 L of diluted synthetic liquid digestate that contained 100 mg/L of  $\text{NH}_4^+ - \text{N}$  was supplied to the system for 30 min. Then, the primary bioreactor was left for 12 h to be continuously homogenized at 200 rpm in dark mode. After that, the primary bioreactor was left for 1 h for the settling phase with no mixing. Finally, 1 L of the supernatant was withdrawn from the system for 30 min. and the whole cycle would be repeated. The operational cycle of PSBR 1 with the exact timings for the light and dark periods can be seen in Figure 6.4.



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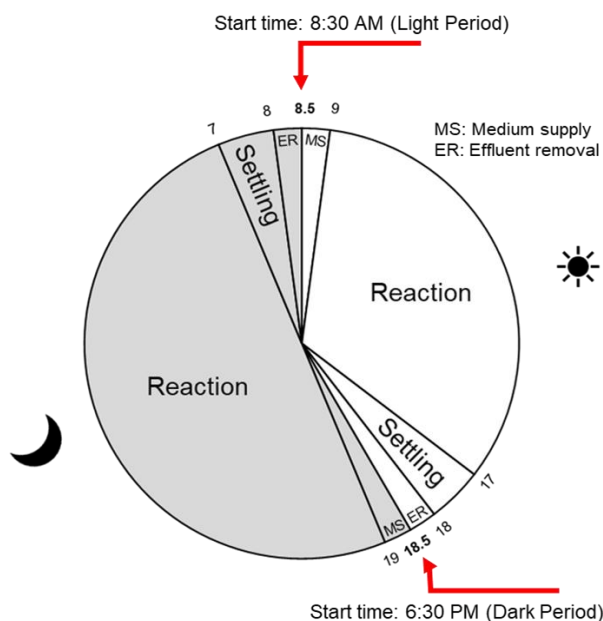


Figure 6-4: Operational cycle of PSBR 1.

The COD and nitrogen concentrations in the feed supply of PSBR 1 during the light and dark periods are summarized in Table 6.4Table 66-4. However, due to time restrictions, the real case was not assessed, and the actual experiment was conducted under the ideal case only.

Table 66-4: COD and nitrogen concentrations in the feed supply of PSBR 1 during the light and dark periods.

	<b>Feed Supply Concentration (mg/L)</b>			
	Light Period		Dark Period	
	COD (CH <sub>3</sub> COONa.3H <sub>2</sub> O)	Nitrogen (NH <sub>4</sub> Cl)	COD (CH <sub>3</sub> COONa.3H <sub>2</sub> O)	Nitrogen (NH <sub>4</sub> Cl)
Ideal Case*	100	0	0	100
Real Case**	100	1	15	100

\*COD: N = 100:0 (light period) and 0:100 (dark period)

\*\*COD:N = 100:1 (light period) and 0.15:1 (dark period)

### 3.2.3.2 PSBR 2 (Control bioreactor)

PSBR 2 was operated as a control bioreactor to be compared with the results from PSBR 1. PSBR 2 was operated for 50 days with 24 h cycles (10 h:14 h light: dark cycle) using diluted mixed synthetic brewery wastewater and synthetic liquid digestate as a feed supply. The diluted mixed wastewater was supplied only during the light period, which contained 50 mg/L-COD and 50 mg/L NH<sub>4</sub><sup>+</sup> - N (COD: N = 1:1). The environmental and operational conditions were the same as PSBR 1. However, the reaction duration was 22 h since there was no feed supply during the dark period. Also, there was only one settling and decanting phase after the dark period, which lasted for 1 hour and 30 min, respectively. Figures 6.5 and 6. 6 represent the schematic setup and the operational cycle of PSBR 2. Moreover, the control bioreactor (PSBR 2) was cleaned at the end of every week, with no manual removal of any biomass. Therefore, the SRT was not controlled during the experiment. Also, on day 23, different bio-augmented microalgae species (i.e., from Delft canals) were added to PSBR 2.





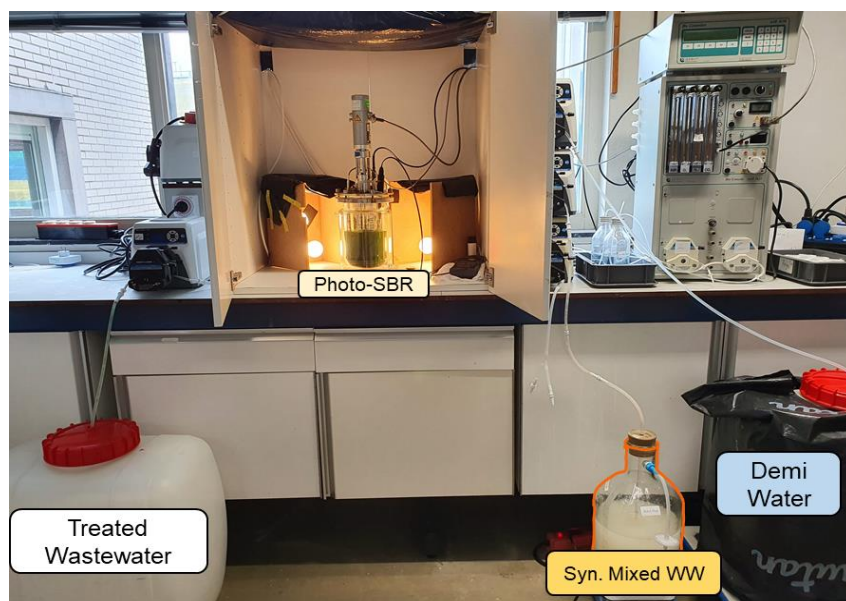


Figure 6-5: Schematic of PSBR 2 setup.

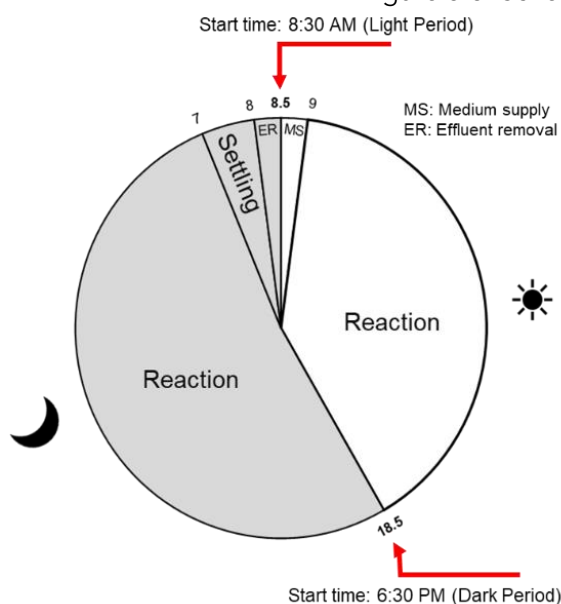


Figure 6-6: Operational cycle of PSBR 2.



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## 6.5. LABORATORY EXPERIMENTS PER WASTEWATER TREATMENT

### 6.5.1. SUMMARY AND CONCLUSIONS

#### → Lab-scale operation for water treatment and process and design optimization

At the start of the light period, there were some variations in the inlet COD and TIC concentrations due to the uneven mixing of the medium supply and distilled water, as shown in Figure 6.7. Moreover, the COD concentration decreased from ~100 to 0 mg/L over the light period during the day, with the highest removal rate in the first 2 hours of the day. Also, the effluent COD concentration decreased after every cycle, until it reached 0 mg/L on day 21. The COD removal rate increased over time, from 8.1 mg/L.h (71.3% COD removal) on day 8 to 14.0 mg/L.h (100.0% COD removal) on day 21.

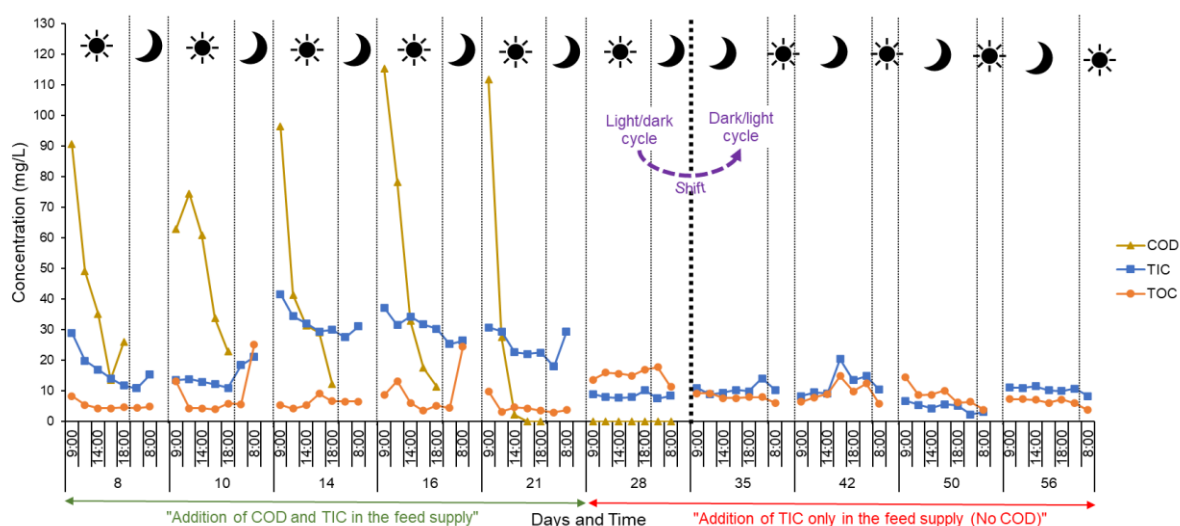


Figure 6-7: Removal of COD, TOC, and TIC for PSBR 1 during 70 operating days.

Nevertheless, after day 21, the net oxygen production in PSBR 1 was 0%, which means that the oxygen consumption by the heterotrophic bacteria outweighed the oxygen production by the microalgae during the light period. Therefore, it was assumed that the heterotrophic bacteria were dominating the primary bioreactor, and to control their growth, it was decided to stop supplying COD to PSBR 1 and depend only on the inorganic carbon as a carbon source for the biomass. Thus, there was no COD concentration recorded from day 28 onwards (Figure 7). Furthermore, the TIC concentration showed the same trend as the COD, where it decreased over the light period during the day. The average TIC removal rate was 1.2 mg/L.h (27.8% average TIC removal) during the first 28 days. Besides, the TIC concentration was increasing during the dark period. However, due to the limited working hours in the lab, only two samples were measured during the dark period, one at the start of the period and the other at the end. Hence, it was decided to switch the light and dark cycle starting from day 35, so it would be possible to analyse more samples during the dark cycle. The TIC concentration was increasing during the dark cycle during days 35 (from 11.0 to 14.1 mg/L) and 42 (from 8.3 to 14.8 mg/L), then it started to decrease at day 50 (from 6.7 to 2.4 mg/L). However, on day 56, the TIC concentration showed a slight increase in the middle of the dark cycle (11.5 mg/L),



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and then it decreased again to 10.8 mg/L. Overall, the average increase in the TIC production rate during the dark cycle was 0.4 mg/L.h with an average TIC increase of 36.5%.

In addition, the TOC concentration mostly showed a reducing trend during the day, except for day 14, when the TOC concentration was increasing throughout the day (from 5.4 to 9.1 mg/L) (**Error! Reference source not found.** 7). The average TOC removal rate was 1.0 mg/L.h (52.5% average TOC removal) during the first 28 days. Nonetheless, on day 14, the TOC concentration increased from 5.4 to 9.1 mg/L in the first 6 hours, then it decreased to 6.7 mg/L after 2 hours. On day 28, it showed a gradual increase as well, where the TOC concentration increased from 13.7 to 17.0 mg/L at the end of the light cycle. On the other hand, the TOC concentration was increasing during the dark cycle. However, after stopping the addition of the COD to the feed supply on day 28, the TOC concentration started to decrease during each dark cycle with an average TOC removal rate of 0.4 mg/L.h (29.1% average TOC removal). Nevertheless, the presence of TOC during the dark cycle after stopping the supply of COD was due to some expected reasons: some interferences occurred while measuring the TOC due to the availability of other chemicals in the feed supply, and the presence of dead cells left in PSBR 1 due to feeding of protozoa on the biomass.

As shown in Figure 6.8, the ammonium concentration was being consumed by the biomass during the dark period. However, on day 10, the results revealed the presence of nitrite in PSBR 1, where the nitrite concentration reduced overnight from 1.5 to 0.8 mg/L. The nitrite concentration kept increasing over each cycle until it reached 12.4 mg/L at the end of the dark cycle of day 21. Also, the results showed the presence of nitrate by day 16, where the nitrate concentration measured at the end of that day was 1.5 mg/L. Based on the nitrite and nitrate concentration results, it was decided to add a nitrification inhibitor (10 mg/L N-Allylthiourea) on day 28 to prevent the nitrifiers from nitrifying the ammonium and leave it for the microalgae to consume it instead. After adding the nitrification inhibitor, it was realized that less ammonium was nitrified to nitrite and nitrate, while the nitrite and nitrate concentrations were decreased. The ammonium effluent concentration increased from 22.6 mg/L (day 21) to 45.1 mg/L (day 28), whereas the nitrite effluent concentration decreased from 12.4 mg/L (day 21) to 9.2 mg/L (day 28). Furthermore, the net oxygen presence in PSBR 1 started to increase again after day 21, when the nitrification inhibitor was added (Figure 6.8).



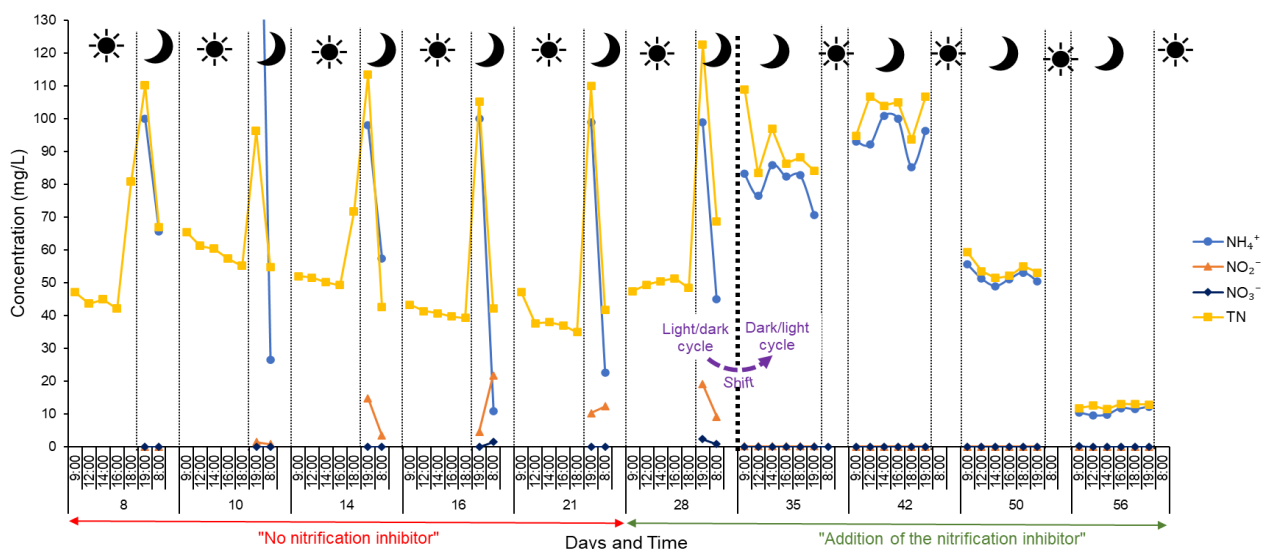


Figure 6-8: Removal of ammonium, nitrite, nitrate, and total nitrogen for PSBR 1 during 70 operating days.

After day 35, the light and dark cycle was switched to have more data to analyse during the dark period. The results showed that the ammonium removal rate decreased from 7.4 mg/L.h (89.1% removal) on day 16 to 1.0 mg/L.h (8.4% removal) on day 42 after adding the nitrification inhibitor, which showed that the ammonium was mostly removed by nitrification and not fully consumed by the microalgae. Also, both the nitrite and nitrate concentrations remained below the detection limit throughout the whole operating period, after adding the nitrification inhibitor.

Moreover, after realizing the low ammonium removal efficiency (8.4%) by the microalgae when the nitrification inhibitor was added, it was decided to reduce the initial ammonium concentration in the feed supply till it was ensured that the whole ammonium would be consumed during the dark period. Accordingly, the initial ammonium concentration decreased from 100 to 20 mg/L on day 50. However, the system did not adapt quickly to the change in the ammonium concentration and the initial concentration on that day was 55.6 mg/L instead of being ~20 mg/L, due to some accumulation of ammonium in the primary bioreactor. Thus, the results showed that the ammonium concentration decreased from 55.6 to 49.0 mg/L (11.9% removal). Then, further reduction in the ammonium concentration was applied, where the concentration decreased from 20 to 10 mg/L on day 56. The system was left for 7 days to adapt to the new concentration and ammonium accumulation from the previous cycle was avoided. However, the ammonium concentration on day 56 did not decrease significantly, where it changed from 10.5 to 9.6 mg/L (8.6% removal).

Finally, the total nitrogen concentration was measured during each cycle to help in the nitrogen balance preparation and calculate the total organic nitrogen (TON) at the end of the research. The total nitrogen concentration was following the same trend as the ammonium concentration (**Error! Reference source not found.**). However, on day 10, the ammonium inlet concentration was too high (243.9 mg/L), where the total nitrogen inlet concentration was 96.1 mg/L. This indicates that there was a measurement error that happened during measuring the ammonium, which gave that high value. Also, the highest total nitrogen removal rate was 5.9 mg/L.h (62.4% removal) on day 14. Table 6.5 shows the nitrogen balance of PSBR 1 on days 16 and 35. Due to nitrification occurred on day 16, the ammonium removal by the microalgae was low (78.8%). However, when the nitrification was inhibited, the ammonium removal by the microalgal increased to 100.0% on day 35.



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Table 6-5. Nitrogen balance of PSBR 1 for days 16 and 35.

Day	Initial nitrogenous concentration (mg/L)					Final nitrogenous concentration (mg/L)					NH <sub>4</sub> <sup>+</sup> - N uptake by microalgae (mg/L)	NH <sub>4</sub> <sup>+</sup> - N removal by microalgae (%)
	NH <sub>4</sub> <sup>+</sup>	NO <sub>2</sub> <sup>-</sup>	NO <sub>3</sub> <sup>-</sup>	TN	TON	NH <sub>4</sub> <sup>+</sup>	NO <sub>2</sub> <sup>-</sup>	NO <sub>3</sub> <sup>-</sup>	TN	TON		
16	100	4.6	0	105	0.4	11	22	1.5	42	7.5	70.1	78.8
35	83	0.2	0	109	25.8	71	0.2	0	84	12.8	12.0	100.0

### → Research interactions between pilot and lab-scale for water treatment

In the lab-scale, at IHE Delft, two bioreactors (PSBR 1 and PSBR 2) of working volume 2 L were operated with 24 h cycles, divided into a 10 h light and 14 h dark period. PSBR 1 was operated for 70 days, and it was fed with synthetic brewery wastewater during the light period and synthetic liquid digestate during the dark period. Whereas PSBR 2 was operated for 50 days, and it was fed with mixed synthetic brewery wastewater and liquid digestate during the light period.

In the pilot-scale PAS of ~1500 L capacity, the reactor is presently being operated with one cycle/day to determine the nutrient removal efficiencies under the prevailing climatic conditions (temperature, light intensity, and specific loading rate fluctuations). The following parameters will be monitored in the reactor: BOD, COD, TOC, TIC, TSS, VSS, PO<sub>4</sub>-P, NH<sub>4</sub>-N, TN, NO<sub>2</sub>-N, NO<sub>3</sub>-N, DO, CO<sub>2</sub>, pH, temperature, chlorophyll - *a*, lipid content, biomass productivity, heavy metals, emerging contaminants, and microbiological parameters (of interest) for bacterial and algal activity tests.

The following conclusions are derived from the interactions between the lab-scale and the pilot PAS system:

- The PAS in Delhi has been operated under the following conditions: Fill - 1 hour; React - 21 hours; Settle 1 hour; Decant 1 hour; total volume of drain water treated = 600 L/day. Regular monitoring of all the parameters of interest, thrice per week, at the beginning of July 2023.

## 6.6. TROUBLESHOOTING AND PROCESS SAFETY

During the laboratory studies at IHE Delft, troubleshooting problems that require more measurements or safety-related issues were not encountered.



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## CHAPTER 7 TECHNOLOGY: SELF FORMING DYNAMIC MEMBRANE BIOREACTOR

### 7.1. ADMINISTRATIVE INFORMATION

Technology name:	SFD MBR (Commercial name: "TARON®")	
Location of lab and pilot:	Lab: IRSA CNR, Bari, Italy	Pilot: Jajmau WWTP Kanpur, India
Organization(s):	IRSA CNR	IITK, Xylem
Contact information:	Contact: Alfieri Pollice	Contact: Prof. P. Bose

### 7.2. BRIEF TECHNOLOGY DESCRIPTION

The SFD MBR is an evolution of the conventional ultrafiltration based MBR, where filtration nets are used as supports to favour the spontaneous accumulation of a sludge layer (cake, or dynamic membrane DM), which becomes the main filtration medium. With an effluent quality similar to conventional MBRs, the advantages of a SFD-MBR are: higher productivity (fluxes up to hundreds  $\text{L m}^{-2} \text{h}^{-1}$ ), lower energy requirements (transmembrane pressure - TMP - below a few hundred mbar, suitable to gravity-driven operation), cheaper support materials (nylon or polyethylene nets). These characteristics make the SFD-MBR a simpler and more robust technology concerning higher-pressure membrane systems, possibly suited for decentralized applications (even solar powered and off-grid) and water reclamation in remote areas.

### 7.3. TECHNOLOGY-SPECIFIC SUMMARY AND CONCLUSIONS

The SFD MBR was assessed at the bench scale under different operating conditions and using different filtration nets. Experiments were conducted using real primary settled municipal wastewater from local treatment plants. Effluent quality was evaluated for assessing the systems' performance and for its compliance with standards for reuse in irrigation. Operational aspects such as automatic maintenance approaches, cleaning frequencies, and optimal sludge age were also evaluated. Results will be useful for pilot plant operation.



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## 7.4. PROCESS OVERVIEW

A general process overview of the bench scale SFD MBR is presented in Figure 7.1.

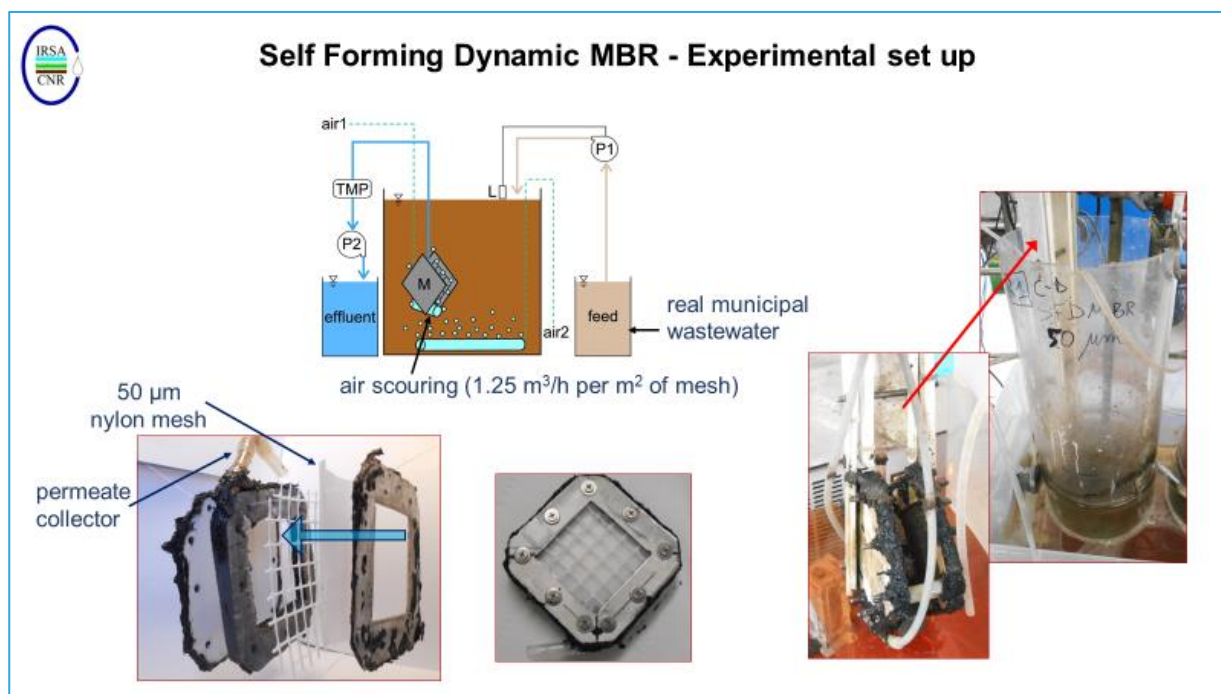


Figure 7-1: General process overview.

Different SFD MBR configurations were assessed at the bench scale for process optimization. Figure 7.2 shows the scheme adopted for comparing two different automatic maintenance approaches, the periodic air mass load, and the continuous air scouring.

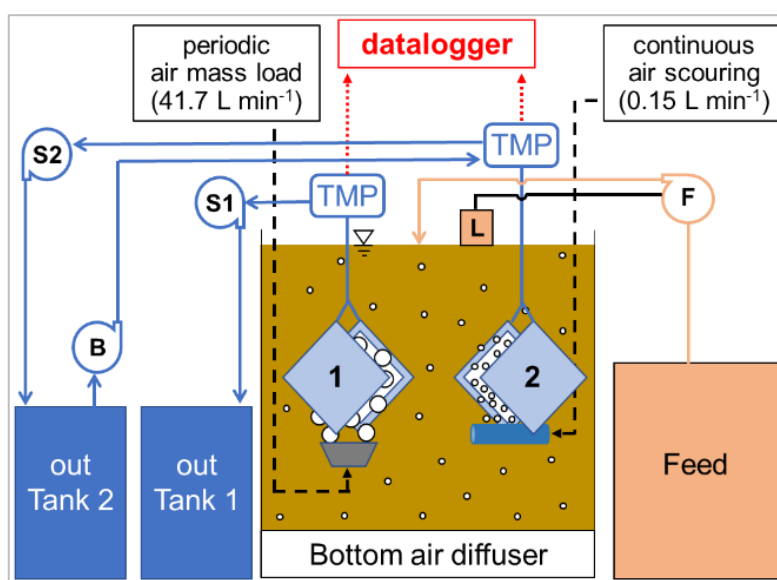


Figure 7-2: Alternative configuration of the bench scale SFD MBR.

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The filtering materials used to support the growth of the dynamic membranes are nylon or PET nets with pores ranging between 20 and 100  $\mu\text{m}$ . As shown in Figure 7.3, these nets sustain the development of the cake layers that are the main filtering material.

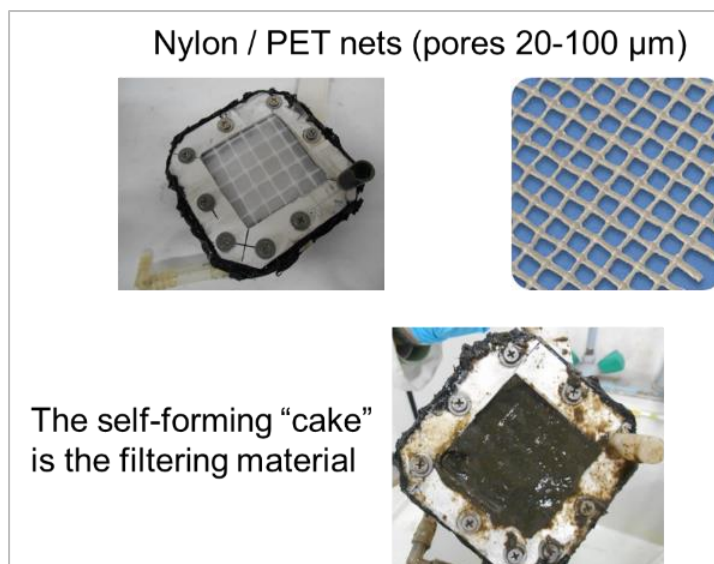


Figure 7-3: Clean supporting material and bench scale dynamic membrane.



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## 7.5. FACTSHEET: CORE PARAMETERS ON WATER TREATMENT AND RESOURCE RECOVERY

Table 7-1: General factsheet: Self Forming Dynamic Membrane Bioreactor

In:fluent type(s)	Pre-settled municipal sewage (mostly)	Influent to SFD MBR		
	Pre-settled agro-industrial wastewater (winery and canning)	Influent to SFD MBR		
Parameter		Units	Value	Comments
Influent quality	COD	mg/L	400-450	Avg COD of agro-industrial wastewater was 1.0 and 1.9 g/L
	BOD	mg/L		Only measured in agro-industrial wastewater (about 0.45 and 0.7 g/L)
	TSS	mg/L	200	
	N <sub>tot</sub>	mg/L	60-85	Agro-ind ww 40 and 70 mg/L
	P <sub>tot</sub>	mg/L	13	Agro-ind ww 11 and 12 mg/L
Effluent quality	COD	mg/L	20-40	Except for outliers and industrial effluent (40-80 mg/L)
	TSS	mg/L	n.d.	Consistently below detection limits, except industrial effluent (5-50 mg/L)
	Turbidity	NTU	1.2-4.1	Except for outliers and industrial effluent
	N <sub>tot</sub>	mg/L	60-80	Complete nitrification
	P <sub>tot</sub>	mg/L	9-12	
	Membrane net flux	L/(m <sup>2</sup> h)	60-75	Agro-industrial SFD MBR operated with 38 LMH



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Technology-defining Rates/ Ranges	Cleaning frequency	1/week	0.33-3.5	Except for outliers
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## 7.6. LABORATORY EXPERIMENTS PER WASTEWATER TREATMENT AND REUSE TECHNOLOGIES

### 7.6.1. SUMMARY AND CONCLUSIONS

#### → Lab-scale operation for water treatment

Lab-scale experiments were conducted to investigate the SFD MBR from the point of view of biological processes and dynamic membrane filtration. Tests aimed at evaluating the process performance in terms of effluent quality and operation sustainability. To do this, different conditions were adopted and maintained for sufficiently extended periods to allow reliable evaluations and margins for further optimization. The basic requirement of all experiments was to obtain an effluent quality compatible with standards for irrigation.

The SFD MBR technology allows much higher fluxes (between several tens and several hundreds of LMH) than those normally adopted for conventional MBR. Furthermore, higher sludge retention time (SRT) values can be maintained, resulting in more efficient degradation processes with improved flexibility towards quality and quantity influent fluctuations.

Experimental activities were conducted at the labs of IRSA CNR in Bari (Italy), with bench scale plants having operating volumes in the range of a few litres and membrane surfaces of 6x6 cm normally coupled to form standard modules of 72 cm<sup>2</sup>.

Tests were conducted with two types of wastewaters: 1) municipal wastewater collected at a local WWTP; and 2) agro-industrial wastewater from two different companies nearby, a winery and a canning factory. The latter were only used for initial testing, while most of the activities are currently conducted with municipal wastewater.

In terms of effluent quality, the results of all the tests performed have shown that:

- Excellent COD removal (always above 90%) was consistently achieved under all the different tested conditions, suggesting efficient biomass retention as a key to high biodegradation performances.
- Effluent TSS below detection limits was obtained in all experiments with municipal wastewater, while earlier tests with agro-industrial wastewater suffered some losses of solids that affected the average effluent quality.
- Turbidity values well below 5 NTU were always obtained, and this had at least two relevant consequences. Firstly, the possibility of adopting UV-based disinfection methods, and secondly the possibility of adopting these effluents for drip irrigation without consequences on clogging of drippers.
- In terms of nitrogen degradation, all experiments were conducted under completely aerated conditions, and complete nitrification was always observed. Phosphorus was also mostly maintained, and the effluents have shown overall nutrient concentrations potentially interesting for reuse in irrigation. Indeed, the content of nitrate and phosphate in effluents would allow for savings in chemical fertilizers and increased crop production.



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Figure 7-4: Laboratory-scale SFD MBR filtration set-up (left) and filtration module (right)

#### → Process and design optimization

Lab-scale SFD MBR process optimization involved the evaluation of various aspects and parameters characterizing the biological process and the dynamic membrane filtration.

1. Preliminary experiments were oriented toward defining the most efficient coupling of mesh size and air flow rate in terms of effluent quality and module cleaning requirements. In particular, the influence of mesh pore size and air scouring on the formation and stabilization of the cake layer in a bench scale SFD MBR for municipal wastewater treatment were evaluated. In different sets of tests, two nylon meshes having pore sizes of 20 and 50  $\mu\text{m}$  were used, and three different air flow rates were assessed for scouring the filter surface. For each experimental run, the quality of the effluents produced and the cleaning requirements were evaluated under steady-state conditions.
2. Another comparative test was conducted to evaluate filtering materials having different mesh sizes (20, 50, and 100  $\mu\text{m}$ ) under SRT values of 15 and 50 days. This study aimed to evaluate the influence of the mesh pore size on the system's performance under different solid retention time (SRT) values. For this purpose, the results of four long-term tests were compared. Meshes having pore size values of 20 and 50  $\mu\text{m}$  were assessed under SRT of 15 d and meshes with 50 and 100  $\mu\text{m}$  pore sizes were compared under SRT of 50 d, all under the same operating conditions.
3. A third set of experiments was focused on the evaluation of different SRT (15, 30, and 50 days). A long-term experiment was conducted, and three runs were performed under identical conditions and different SRT values (15, 30, and 50 days), with the aim of: (i) verifying the reliability of this technology under different bioprocess operating conditions; (ii) evaluating the influence of the SRT on process performance.
4. The last set of tests compared three different dynamic membrane maintenance approaches to evaluate their sustainability and effectiveness. Continuous air scouring was compared



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with two intermittent (periodic) automatic cleaning strategies based on backwash and air mass load, respectively.

In terms of process optimization, the bench-scale testing has produced the following results:

- In the first experiments, larger mesh size (50  $\mu\text{m}$ ) resulted in slower cake formation and mesh clogging and allowed us to identify three phases in the life of the dynamic membrane: formation, maturity, and clogging (TMP > 100 mbar). The effluent turbidity is higher during the first and last phases than during the maturity phase. Smaller mesh size (20  $\mu\text{m}$ ) showed higher cleaning requirements (daily) but produced effluents with lower and more stable turbidity values. Continuous air scouring was suggested as a key factor for ensuring effective operation. On the other hand, a too intense air scouring (500  $\text{mL}\cdot\text{min}^{-1}$ ) may result in a lower effluent quality, especially when the larger mesh size (50  $\mu\text{m}$ ) is adopted. Operation with the 50  $\mu\text{m}$  mesh and a low air scouring intensity (150  $\text{mL}\cdot\text{min}^{-1}$ ) can be considered as an optimal compromise between high effluent quality and limited cleaning requirements. Indeed, hydrodynamic conditions that minimize the cleaning frequency tend to extend the duration of the maturity stage of the self-forming membrane, enhancing the effluent quality.
- The second set of tests suggested that the mesh pore size has limited effects on the cleaning requirements and a small influence on the effluent quality (only when DM is under formation or under high suction pressures). On the contrary, when the DM is in its maturity stage and the TMP is close to zero, the effluent quality was observed to be independent of the mesh pore size. Moreover, steady biological and physical operating conditions result in rapid formation and good stability of the DM. In this regard, high SRT values play a primary role. On the other hand, when this stability cannot be ensured (e.g., under short SRT and/or variable feed conditions), a relatively small pore size of the supporting medium (20  $\mu\text{m}$  or close to it) can favour the DM development.
- The third experiment showed that the SRT can considerably influence the system's performance, and better effluent quality was observed for operation under the higher SRT values. Indeed, the latter was observed to provide much better results in terms of effluent quality, cleaning requirements, and overall process resilience towards operational variations. Moreover, this study highlights the importance of biological process stability for the development of an effective DM.
- Recent results have shown that the SFD MBR always produced high quality effluents, largely independent of the DM maintenance strategy adopted. In terms of operation sustainability, the mass air load supply was the most effective cyclic and temporized DM maintenance strategy, as it resulted in much lower frequencies of on-site manual cleaning. Both the tests conducted with this strategy displayed extended periods exceeding two weeks without the need for manual cleaning, and the test with shorter mass air supply duration allowed for 3 weeks of continuous operation between manual interventions.



### → Research interactions between pilot and lab-scale for water treatment

Some differences existed between the bench scale SFD MBR configuration and the pilot plant (Xylem Taron®). While under both configurations the biological process reactors can be considered similar (except that the pilot has an anoxic section for pre-denitrification), the filtering modules and their operation were based on the same principles but have different layouts. The bench scale adopted square filtering modules completely sank in the bioreactor, and the permeate was extracted through suction pumps. The Taron® pilot plant was equipped with rotating disks only partially submerged in the mixed liquor, and the permeate flows through the filters by gravity. Despite these differences and the relevant scale gap, the two systems showed comparable results in terms of effluent quality, and some indications can be obtained from the bench-scale plant at least for the main bioprocess operating parameters and filtration maintenance, which can be adopted at the pilot-scale.

The main conclusions of the bench scale experiments that can be considered for testing at the pilot plant are the following:

- Working with higher suspended biomass concentration (thus higher SRT, e.g., above 20-25 d) has more advantages than disadvantages. The advantages are: (i) better sludge filterability, possibly due to the lower content of colloids (SMP/EPS) (ii) improved bioprocess stability and degradation performance also in the presence of fluctuations of the influent quality, (ii) lower net biomass production with related savings in terms of excess sludge management and disposal. Possible shortcomings of high MLSS values are the increased aeration requirements and the longer time needed to reach a steady state upon start-up.
- Continuous rotation and backwash of the disk filters (current procedure) could possibly be replaced by intermittent (periodic) maintenance of filtering surfaces. This would result in energy savings and should not affect the effluent quality. Moreover, continuous fine bubble scouring of the external disk surface could be replaced by periodic mass air load supply.

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## 7.7. TROUBLESHOOTING AND PROCESS SAFETY

### 7.7.1. SAFETY MEASURES

- Aspects related to the safe installation and operation of the bench scale SFD MBR plants refer to the adoption of materials and procedures that are suited for these applications. Hydraulic and electric components are always purchased and assembled according to safety procedures. Bench scale plant operation (sampling, regulations, monitoring) are conducted by experienced personnel equipped with all relevant IPD and maintaining the required safety protocols.
- Aspects related to process monitoring, and specifically related to laboratory analyses. Chemical and microbiological analyses are conducted at the labs according to standard safety procedures.

### 7.7.2. TROUBLESHOOTING

- All bench scale experiments aim to investigate optimized operating conditions in terms of process effectiveness (maximize effluent quality) and sustainability (minimize interventions of operators). To achieve these objectives, plant operation is conducted under different process conditions and the results are compared and critically evaluated.
- Common operational issues are related to periodical filter clogging with consequent increase of the transmembrane pressure (TMP), leading to loss of filtration flow rates and finally requiring manual cleaning of the modules. The TMP is continuously monitored through pressure gauges, recorded with a datalogger, and displayed on a PC monitor. This allows us to detect the filtration instabilities from the beginning and organize the cleaning activities.
- Other unforeseen issues refer to all common aspects normally related to the operation of experimental bench-scale wastewater treatment plants and specifically refer to the functioning of equipment (pumps, blowers, mixers, etc.) and measuring devices (e.g., probes, sensors, controls). Skilled operators conduct continuous checking and observation to timely detect and solve all potential problems.



## CHAPTER 8 TECHNOLOGY: STRUCTURED ADSORBENTS

### 8.1. ADMINISTRATIVE INFORMATION

Technology name:	Structured Adsorbents (SA)	
Location of lab and pilot:	Mol, Belgium	Kanpur, India
Organization(s):	VITO	IITK
Contact information:	Lead: E. Seftel	Lead: P. Bose
	Contact: E. Seftel, B. Michielsen	Contact: P. Bose

### 8.2. BRIEF TECHNOLOGY DESCRIPTION

This technology using structured adsorbents is based on the adsorption principle and is especially suited for effluents containing low amounts of contaminants, but above the accepted limits for maximum contaminant level, where other techniques (such as precipitation) may not work. The adsorption process offers flexibility of design and operation and in many cases produces treated effluents suitable for re-use. The structured sorbents have high binding capacities and fast kinetics and are used for the removal of contaminants such as heavy metals (Cr).

### 8.3. TECHNOLOGY-SPECIFIC SUMMARY AND CONCLUSIONS

#### Task 3.2 Laboratory experiments per wastewater treatment and reuse technologies

The focus of this task was on the preparation of structured sorbents based on environmentally friendly clay-type composites. Given the fact that the speciation of chromium in the streams present at the Indian Jajmau site is complex, e. g. both  $\text{Cr}^{3+}$  and  $\text{Cr}^{6+}$  forms present, the efforts were in adapting the structured sorbent composition for enabling the sorption of all Cr contaminant species. The lab tests performed at VITO demonstrated that the optimum composition for both  $\text{Cr}^{3+}/\text{Cr}^{6+}$  removal is obtained with structured Sorbent 1 with a composition of 80wt% LDO/20wt% Bentonite. This shows a Cr adsorption capacity of ~35mgCr/g.

Also, the regeneration procedure was optimized at VITO to enable the re-use of the structured sorbent after saturation. This allowed the selection of the best regeneration solution, namely after saturation, the columns packed with structured sorbents can be regenerated using 2M NaCl solution at pH neutral.

The selected composition was upscaled and ~5kg of structured sorbent were sent to IIT Kanpur for validation tests at batch mode, and further integrated into columns for demonstration. Concrete experimental testing plans were formulated and discussed with IIT Kanpur. Cr sorption tests were done at IIT Kanpur to investigate the isotherms and kinetics of the Cr recovery process, as well as the influence of the S/L ratio. The kinetics parameters were used as input for the modelling of the columns and determination of the hydraulic operation parameters.



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#### 8.4. PROCESS OVERVIEW

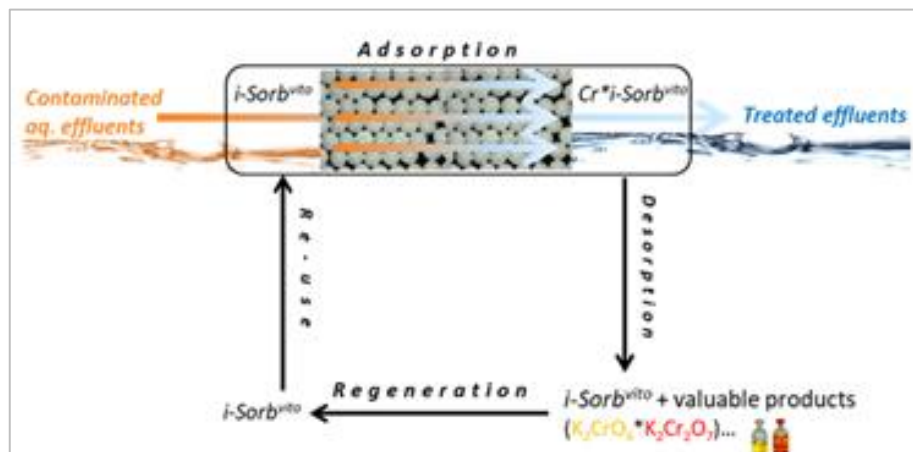


Figure 8-1: General process overview.

The process involves the use of sorbent materials that can uptake contaminants from polluted wastewater. The adsorption process offers flexibility of design and operation and in many cases produces treated effluents suitable for re-use. The technology has the advantage that the sorbent composition can be tailored for high capacity, fast kinetics, good hydraulic conductivity, easily replaceable and safe. After use, the sorbents can be regenerated, and the metals can be desorbed to recover the resource and the regenerated sorbents can be re-used.

##### **Advantages technology**

- High sorption capacity (in the range of 35mgCr/g)
- Fast kinetics (of both adsorption and stripping steps)
- Good mechanical and chemical stability
- The developed sorbent materials are shaped into granulates enabling their application in column set-ups with high hydraulic conductivity, easily replaceable and safe.
- Low-cost production
- Regeneration abilities with 2M NaCl solution at neutral pH for recovery of Cr and allowing the regeneration and multiple use of the sorption material.



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## 8.5. FACTSHEET: CORE PARAMETERS ON WATER TREATMENT AND RESOURCE RECOVERY

Table 8-1: General factsheet: Structured Adsorbents

Parameter		Units	Value	Comments
Influent type(s)	IPC membrane filtration permeate			
Influent quality	COD	mgCOD/L		
	TSS	gTSS/L		
	pH		8.3-8.6	
	Cr	mgCr/mL	1 - 2	
	others...			
Effluent quality	COD	mgCOD/L		
	TSS	gTSS/L		
	pH		12	
	Cr	mgCr/L	< 0.05	
	others...			
Technology-defining Rates/ Ranges	Temperature range	°C	RT	
	pH range	-	8 - 12	



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	Dimensions columns	m	0.75/0.25	Height/diameter
	Hydraulic loading rate	m <sup>3</sup> /m <sup>2</sup> /d	0.5 - 1.5	For 1mgCr/L in influent
	Flow rate	mL/min	2 to 4	Estimation based on lab testing at IIT Kanpur
	Others...			
Maintenance	Cleaning frequency	days	25	Estimation based on lab testing at IIT Kanpur
	Cleaning products		NaCl 2M	Striping solution for Cr after saturation of column



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## 8.6. LABORATORY EXPERIMENTS PER WASTEWATER TREATMENT AND REUSE TECHNOLOGIES

### 8.6.1. SUMMARY AND CONCLUSIONS

#### → Lab-scale operation for water treatment

Several compositions of structured sorbents were prepared. The compositions were differentiating to enable the sorption of mixtures of  $\text{Cr}^{3+}/\text{Cr}^{6+}$  in different ratios. To capture the  $\text{Cr}^{3+}$  a cationic clay, bentonite was used. For the removal of the  $\text{Cr}^{6+}$ , an LDO (Layered double oxide) was taken. The characterization of the granulated adsorbent was performed to assess their structural and porosity characteristics (e. g. XRD,  $\text{N}_2$  sorption, Hg porosity). After laboratory tests at VITO, Sorbent 1 with 80wt% LDO/20wt% bentonite was selected as an optimum composition for further testing. Sorbent 1 has an adsorption capacity for Cr of  $\sim 35\text{mgCr/g}$ .

After this, elaborated batch tests were performed both at VITO and at IIT Kanpur to study the adsorption isotherms, the effect of time (kinetics), the effect of S/L ratio, and optimization of the stripping solution for Cr recovery and sorbent regeneration.

The following conclusions were derived from the Lab-scale operation of the structured adsorbents for wastewater treatment:

- Batch tests showed that the increased S/L ratio increases the Cr recovery efficiency.
- Using a S/L ratio of 2.5 g/100mL influent, a Cr removal efficiency of 93-96% was achieved.
- Desorption of Cr and regeneration of the structured adsorbent was optimized: a solution containing 2M NaCl with neutral pH.

**References:** measuring report references: Protocol for lab tests structured sorbents

#### → Process and design optimization

The first stage of testing included the lab tests in batch mode to the study of kinetics and isotherms for Cr sorption, with selecting the optimum conditions for column setup. Batch tests showed that the increased S/L ratio increases the Cr recovery efficiency. In parallel, the desorption of Cr and regeneration of the structured adsorbent was optimized, so it was ready to serve to complete the protocol for multi-cycling column operation. Investigation of the sorption kinetics allowed the calculation of the  $K_f$  and was used as input for the modelling of the columns and determination of the hydraulic operation parameters.

The following conclusions were derived from the Lab-scale operation of the self-structured adsorbents regarding process and design optimization:

- The kinetic results were used as input for the modelling and design of the column setup.
- Simulation results indicated that the column should be operated at hydraulic loading rates of 0.5, 1 and  $1.5 \text{ m}^3/\text{m}^2/\text{d}$  and influent  $\text{Cr(VI)}$  concentration of 1 mg/L.
- The minimum flow rate was 2mL/min.

**References:** short testing report (as ppt slides) prepared by IIT Kanpur and shared with VITO



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## → Research interactions between pilot and lab-scale for water treatment

Column design was done according to the feasibility tests and modelling information: height of 0.75 m and diameter of 0.25 m. 3 columns are packed with approximately 5kg of structured adsorbent (see Figure 8.2). Support material such as glass wool is used for the support of adsorbents (columns with inbuilt support).

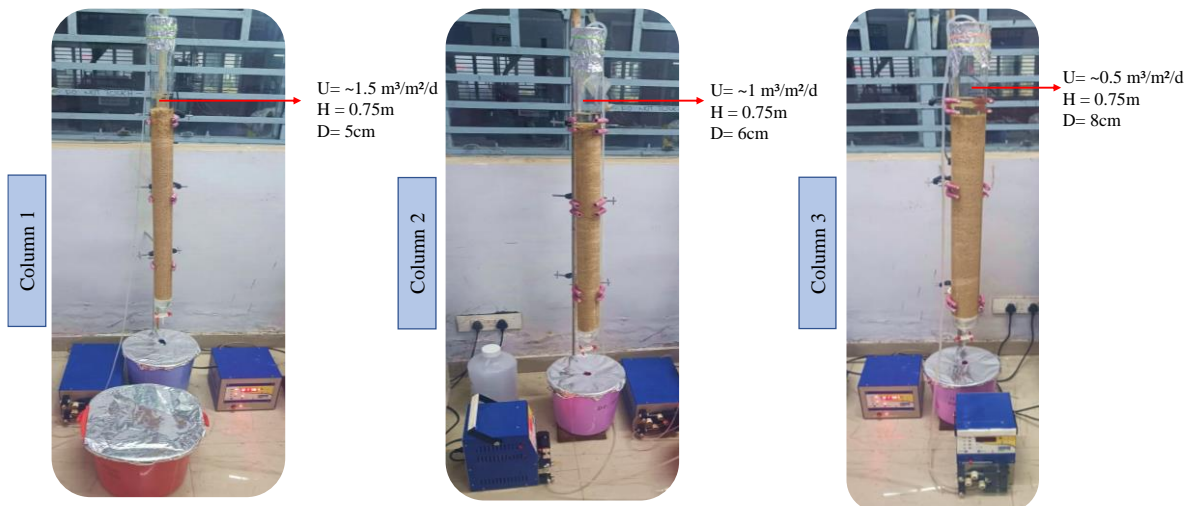


Figure 8-2: Columns filled with Structured Sorbent 1 operating with flow rate 2mL/min and hydraulic loading rate (U) of 1.5 m<sup>3</sup>/m<sup>2</sup>/d (left), 1 m<sup>3</sup>/m<sup>2</sup>/d (middle) and 0.5 m<sup>3</sup>/m<sup>2</sup>/d (right)

The columns ran for approximately 50 days. The breakthrough was reached as a function of column bed volume.

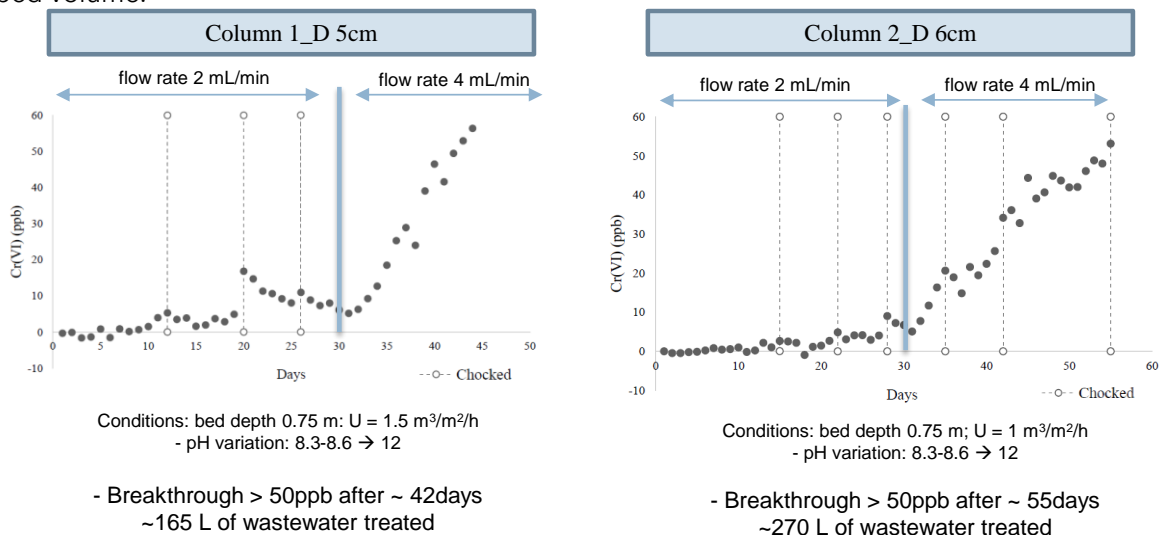


Figure 8-3: Breakthrough curves of Cr(VI) adsorption on structured adsorbents under flow conditions.

The results showed that the volume of treated wastewater is a function of the amount of structured sorbent packed in the column. Approximately 165 L of wastewater having 1 mg/L of Cr(VI) concentration can be treated by using a column with a 5 cm diameter and 75 cm bed depth using the structured adsorbent before releasing into any water stream. In this case, the breakthrough at Cr concentrations of > 50µg/L was achieved after ~42 days (see Figure 8-3, left). If the column



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diameter is 6 cm, the amount of wastewater that can be treated in the same conditions is approximately 270 L, e.g., before reaching the breakthrough of Cr concentration  $> 50\mu\text{g/L}$  (Figure 8-3, right). Furthermore, the pH of the permeate fed into the column ranged from 8.3 – 8.6 while the effluent permeate was highly basic, e.g., of approximately 12. Accordingly, before further usage or disposal, the pH of effluent should be altered.

The colour change observed can be seen in Figure 8.4.



Figure 8-4: Colour changes observed when using Structure Adsorbent 1.

After saturation, the columns packed with structured sorbents can be regenerated according to the procedure optimized at a lab-scale: using 2M NaCl solution at pH neutral.

## 8.7. TROUBLESHOOTING AND PROCESS SAFETY

### 8.7.1. SAFETY MEASURES AND TROUBLESHOOTING

As the solutions contain chromium, the appropriate precautions must be taken:

- Support material such as glass wool is used when packing the columns: it is advised to use basic protective equipment, such as protective gloves and a face mask wear gloves when handling the glass wool.

As the columns choked during long time operation:

- The mechanical stability of the structured sorbents should be further improved by increasing the silica content during the granulation procedure.

pH observed to increase during operation ( $\text{pH}_i 8.3 - 8.6 \rightarrow \text{pH}_f 12$ )

- Before further usage or disposal, the pH of effluent should be altered.

