

Laboratory test reports and fact sheets on nutrient recovery, biogas quality and options for biogas use, as well as effluent qualities and water reuse possibilities

# Deliverable 3.3

WP3 Treatment and recovery technologies

Task 3.3: Laboratory experiments for selected resource recovery technologies (energy, water and nutrients)

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# **SUMMARY**

The summary provides an overview of the most significant outcomes of the laboratory test reports and fact sheets on nutrient recovery, resources recovery, biogas production and water reuse for six different technologies. Each chapter of Deliverable 3.3 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 optimise resources recovery at the laboratory scale and provides indications on what happens next in adjusting and / or evaluating the respective technologies going forward.

ANDICOS<sup>™</sup> technology is used to anaerobically digest organic waste and/or wastewater under an increased organic loading rate. The increase in organic load can be obtained by filtering the wastewater to concentrate it up to 20 times with a Blue Foot Membranes (IPC membranes) filtration process. A 40 L anaerobic digester at IIT Kanpur was employed for anaerobic digestion of primary sludge (PS) sourced from the primary settling tank of STP Jajmau. PS, rich in lipids, fatty acids, and fibre, displayed substantial potential for methane production compared to secondary sludge due to its enhanced biodegradability. The digester initially operated with a 40-day hydraulic retention time (HRT) for 300 days and was later reduced to 30 days. Biogas production commenced after 300 days of inoculation, with an average daily production of 6 L from day 320 to 360, which subsequently decreased to 2 L per day by day 400. During this period, there was an approximate 20% reduction in total chemical oxygen demand (COD), and volatile fatty acids (VFA) concentration in the digester ranged from 100 to 110 mg/L as CaCO3. The volatile solids (VS) to total solids (TS) ratio fluctuated between 0.25 and 0.28; however, it decreased to 0.15 after day 400, leading to a decline in biogas production. The system's pH remained within the range of 7.2 to 7.4 throughout operation, and the total methanogenic activity at the end of 400 days was determined to be 18.1 ml methane per gram of volatile solids per day. With the drastic improvements of STP influent occurring the during the project, further laboratory scale work is investigating the treatment of different mixes of CETP and STP influent, as these may show opportunities to increase biogas production. During the project it was agreed to put less emphasis on mixing food wastes with sewage concentrates, so as to avoid mixing fecal and non-fecal waste streams, but further work could be done on combing septic tank waste streams with sewage waste streams to increase biogas production, because septic tanks waste collection and disposal remains an important activity in urban areas of India.

**Cleanblocks** are a mineral wool filtering material, that can be used for the treatment of polluted waters, like drain and wastewater. For resource recovery, mineral wool dissolution and subsequent phosphate precipitation was investigated pH mediated. While PHREEQC modelling demonstrated the potential for struvite precipitation in drain water by adding calcium or iron, experiments revealed that mineral wool dissolution did not occur at varying pH levels (4, 7, and 8.5) over four weeks. Instead, mineral wool acted as a buffer in both acidic and basic conditions. As a result, phosphate removal via mineral wool dissolution was deemed unfeasible. However, the modelling indicated that, under New Delhi conditions, phosphate recovery could still be achieved by introducing calcium or iron, leading to spontaneous phosphate precipitation in drain water. Therefore, the addition of calcium and/or iron remains a promising approach for phosphate removal in such settings. This can be a further line of research moving forwards.







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. The project's goal was to create a wastewater reuse system with resource recovery in urban and peri-urban areas of India. In this context, batch tests were conducted to examine the remobilization of heavy metals (Cr, Cu, Fe, Pb, and Zn) previously adsorbed. These tests involved varying pH levels (ranging from 3 to 8) and different NaCl concentrations (10 mg/L, 50 mg/L, and 100 mg/L). Samples were collected after 1 day and 1 week to assess the impact of contact time. The findings suggest that desorption primarily occurs for Cu and Zn at a pH of 3, to a lesser extent at pH 5, while Fe and Pb do not desorb at any pH level. Desorption takes place relatively quickly, typically within one day of pH adjustment, with minimal additional desorption after one week. To observe desorption effects, the NaCl concentration needs to be at least 50 mg/L. Combining different methods, such as a hybrid treatment approach using various alkali ions and acidic conditions, appears to be more effective for achieving desorption and resource recovery. While the pilot at Jajmau Sewage Treatment Plant (STP) was not fully operational until January 2023, and the adsorbents were not depleted sufficiently for exchange, regenerating them for recovering desorbed heavy metals could be considered as a research line moving forwards. Another research approach is the recovery of ammonium sorbed on the used zeolites.

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. The primary objective of the study was to enhance lipid productivity using microalgae-bacteria consortia in photo-sequencing batch reactors (PSBRs) with the "survival of the fattest" mechanism. This involved using COD- and TIC-rich brewery wastewater during the light period and ammonium-rich liquid digestate during the dark period. Lipid content analysis revealed that biomass from PSBR 1 exhibited significantly higher lipid content (54.3%) compared to PSBR 2 (18.6%). Despite the higher biomass productivity in PSBR 2 (19.2 mg-TSS/L.d), the lipid productivity was greater in PSBR 1 (4.1 mg/L.d), suggesting that the presence of light and carbon sources during the light period allowed microalgae in PSBR 1 to produce more lipids, which were partly consumed in the dark period for nitrogen assimilation. Thus, it is feasible to use the "survival of the fattest" approach for increasing lipid productivity in a lab-scale PAS system, with improved removal rates of various parameters in PSBR 1 compared to PSBR 2, albeit with lower biomass growth and chlorophyll-a concentration in PSBR 1. 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. The SFD MBR lab-scale experiments were conducted to showcase the efficacy and resilience of this technology in producing high-quality effluents suitable for irrigation while minimizing energy consumption, particularly in comparison to conventional MBR systems. Complete nitrification of ammonium to nitrate was consistently achieved under fully aerated conditions, with soluble phosphorus largely retained in the effluent, demonstrating efficient nutrient conservation. The key resources recovered from this process were nutrients (nitrate and phosphate), energy savings stemming from reduced







pressure filtration requirements, and water suitable for irrigation. While the effluents met irrigation standards in most aspects, higher E. Coli concentrations were observed due to the limited bacterial removal capacity of the dynamic membranes, which typically grow on 50 µm pore size supports. However, the system's efficient removal of suspended solids and the low effluent turbidity facilitated UV-based disinfection, well-suited for water reuse with on-demand, chemical-free operation. Once transferred at a larger scale, this technology will have to be tested for actual energy savings with respect to normal MBR, and also for overall resource recovery with respect to conventional activated sludge. Preliminary results from P recovery through acidification of SFD MBR pilot sewage sludge show that up to 50% of the P could be additionally recovered. However, the chromium concentrations in the acidified sewage sludge are exceeding the limit values as per the Stuttgart process, which chelates heavy metals through citric acid, could result in a promising recovered product for safe agricultural application. Cost-competitiveness of such struvite products needs to be further evaluated given its high energy and material inputs.

Structured adsorbents are based on the adsorption principle and is especially suited for effluents containing low amounts of contaminants. The study involved evaluating the performance of sorbent materials under equilibrium conditions, followed by the selection of the sorbent composition and optimization of Cr recovery and regeneration. Subsequent batch tests at VITO demonstrated exceptional adsorption and regeneration efficiencies, consistently reaching 95-100% over five cycles. Thus, the optimal sorbent composition (Sorbent 1 with 80% LDO and 20% Bentonite) was defined for the recovery of Cr, and a 2M NaCl solution with neutral pH was identify for optimal regeneration. Additionally, Cr sorption tests at IIT Kanpur, which included investigations of isotherms, kinetics, and the influence of the solid-to-liquid (S/L) ratio, provided valuable kinetic parameters for column modelling and design. Simulation results suggested that the columns should be operated at hydraulic loading rates of 0.5, 1, and 1.5  $m^3/(m^2.d)$ , with an influent Cr(VI) concentration of 1 mg/L and a minimum flow rate of 2 mL/min. Lab columns operated at Kanpur for over 20 days did not reach the breakthrough of Cr sorption. This suggests the long-term effectiveness of the columns, with Cr concentrations consistently below the maximum accepted limits (50 ppb). Finally, a column with a 5 cm diameter and a 75 cm bed depth was able to treat around 165 L of wastewater before reaching Cr concentrations exceeding 50 ppb, with an operational duration of about 42 days. Similarly, a column with a 6 cm diameter and the same bed depth could treat approximately 270 L of wastewater before surpassing the 50 ppb Cr concentration limit, with an operational duration of roughly 55 days. 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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#### **TECHNOLOGY: AEROBIC MEMBRANE BIOREACTOR** CHAPTER 1

# **1.1. ADMINISTRATIVE INFORMATION**

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

# **1.2. BRIEF TECHNOLOGY DESCRIPTION**

The aerobic membrane bioreactor (MBR) system is a lab-scale reactor with a capacity of 8 litters 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 temperaturecontrolled 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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CHAPTER 1 - Technology: AEROBIC MEMBRANE BIOREACTOR





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. LABORATORY EXPERIMENTS FOR RESOURCE RECOVERY**

The MBR technology was not researched on the base of resource recovery. Thus, the lab-scale experiments performed under the Pavitra Ganga projected aimed for optimization of the system. Further information can be found in Deliverable 3.2.







# **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	IITKanpur, VITO, IEX
	Lead: IIT Kanpur	Lead: IIT Kanpur
Contact informations	Contact: Aditya Sharma,	Contact: Aditya Sharma, Prof
Contact mormation.	Prof Bose, Sofie Van Ermen	Bose, Sachin Shah, Sofie Van
	,	Ermen

# 2.2. BRIEF TECHNOLOGY DESCRIPTION

ANDICOS<sup>™</sup> 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<sup>™</sup> 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 as a Waste water Line and a Solid waste Line (Figure 2-1)

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



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#### **2.3.** TECHNOLOGY-SPECIFIC SUMMARY AND CONCLUSIONS

This chapter focuses on the anaerobic treatment step of the Andicos concept. During the lab experiments, anaerobic digestion tests of primary sludge are done. The lab tests focus on the biogas production related to the feed and on the biogas quality. The change in the sewage characteristics (D3.2) had a major impact on the composition of the concentrate stream due to the unwished biodegradation that took place in the membrane reactor. As no concentrated sewage was continuously produced during the lab test no lab anaerobic digestion tests of the concentrated sewage could be performed.

#### 2.4. LABORATORY EXPERIMENTS FOR RESOURCE RECOVERY: BIOGAS PRODUCTION

Primary sludge (PS) typically exhibits higher biodegradability and energy capacity compared to secondary sludge. This can be attributed to the presence of lipids, fatty acids, and fibre in PS, which enhance its potential for methane production in contrast to the microbes and extracellular polymeric substances found in secondary sludge (SS). A 40 L anaerobic digester was constructed at IIT Kanpur to treat primary sludge (PS) obtained from the primary settling tank (PST) of the Jajmau sewage treatment plant. The digester was initially operated with a hydraulic retention time (HRT) of 40 days for 300 days, which was later reduced to 30 days. Biogas production was measured using a gas flow meter connected to the digester's gas outlet. Regular analysis of pH, total and soluble chemical oxygen demand (COD), total solids (TS), volatile solids (VS), and volatile fatty acids (VFA) was conducted biweekly to evaluate the digester's performance.



Figure 2-2: Laboratory-scale sludge digestion unit

#### **Conclusions:**

- Biogas production commenced after 300 days of inoculation. The average daily biogas production from day 320 to 360 was 6 L, which subsequently decreased to 2 L per day until day 400.
- The average reduction in total COD during this period was approximately 20%. The VFA concentration in the digester ranged from 100 to 110 mg/L as CaCO3.



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- The VS/TS ratio in the effluent varied between 0.25 and 0.28. However, after day 400, this ratio decreased to 0.15, resulting in a decline in biogas production.
- The pH of the system remained within the range of 7.2 to 7.4 throughout the operation.
- The total methanogenic activity at the end of 400 days was determined to be 18.1 ml methane per gram of volatile solids per day.







# 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 mormation:	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.







# 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	
	Lead: Merle de Kreuk	Lead:
Contact information:	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% SiO2, 15-20% CaO, 15-19% Al2O3 and 6-9% Fe2O3 (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

The pH-mediated mineral wool dissolution and subsequent phosphate precipitation were investigated. Although PHREEQC modelling showed the potential of struvite precipitation in drain water by the addition of calcium or iron, mineral wool dissolution did not occur at various pHs. As such phosphate removal using mineral wool is unlikely.

# 4.4. LABORATORY EXPERIMENTS FOR SELECTED RESOURCE RECOVERY TECHNOLOGIES

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

#### $\rightarrow$ Lab-scale operation for resource recovery

#### Summary:

- Phosphate recovery
  - PHREEQC Modelling using drain water quality parameters showed that the concentrations of NH4 and PO4 allow for struvite precipitation in the New Delhi conditions.
  - Modelling further showed that mineral wool minerals such as Calcium and iron could, when dissociated precipitate with the phosphate.
  - Mineral wool dissolution did not occur when incubated with different pH of 4, 7 and 8.5 over 4 weeks.
  - Mineral wool functions as a buffer in acidic or basic conditions.

#### Conclusions:

- Phosphate removal trough dissolution of mineral wool is not possible.
- The addition of Ca and/or Iron will result in spontaneous phosphate precipitation in drain water.

**References:** Nicole van Jaarsveld, MSc thesis: "Phosphate removal from wastewater by mineral wool filters." 2019



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#### $\rightarrow$ Process and design optimizations

Summary: Not applicable. See task 3.3.1.

#### Conclusions:

• Phosphate cannot be recovered using mineral wool filters.

#### ightarrow Research interactions between pilot and lab-scale for resources recovery

**Summary**: Not applicable. See task 3.3.1.

#### Conclusions:

• Phosphate cannot be recovered using mineral wool filters.







# 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):	НВО	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 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 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 investigate the recovery potential of the heavy metals adsorbed on zeolites, various desorption tests were conducted. The desorption potential was evaluated in two different ways using batch tests in which the loaded zeolites were brought into contact with different solutions. 1: Treatment with different NaCl concentrations, 2: Treatment with different pH values.

# 5.4. LABORATORY EXPERIMENTS FOR SELECTED RESOURCE RECOVERY TECHNOLOGIES

# 5.4.1. SUMMARY AND CONCLUSIONS

#### ightarrow Lab-scale operation for resource recovery

The project aims to develop a wastewater reuse system with resource recovery in urban and periurban regions of India. After a previously conducted adsorption of the heavy metals Cr, Cu, Fe, Pb and Zn, the extent to which these can be remobilised under the influence of different pH values



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and NaCl concentrations is investigated in batch tests. The selected pH ranged between 3-8 and the NaCl concentrations were 10 mg/L, 50 mg/L and 100 mg/L. Samples were taken after 1 day and after 1 week to investigate the influence of the contact time.

The following conclusions are derived from the operation of the wastewater reuse system, for resource recovery:

- Desorption occurs in the following order Cu > Zn > Cr at a pH of 3 and to a minor degree at pH 5.
- No desorption of Fe and Pb at all pH values.
- Desorption occurs within one day after pH adjustment, with no significant increase in desorption after one week.
- NaCl concentration needs to be  $\geq$  50 mg/L to see desorption effects.
- The highest desorption of Zn is followed by Cu.
- No desorption of Cr, Fe and Pb at all NaCl concentrations.
- The methods used alone are not sufficient to achieve effective desorption of all metals.

References: measuring report references: Practical lab reports of "Engineering Studies" master course 2020 at HBO

#### $\rightarrow$ Process and design optimizations

Adjusting the pH value alone or adding NaCl concentrated water is not sufficient to desorb all heavy metals. Lead in particular does not desorb under the methods used. To achieve a better result, a combination of different methods seems to be effective. A hybrid treatment approach with different (earth) alkali ions combined with acidic conditions could increase the desorption effects and thus, resource recovery.

References: measuring report references: Practical lab reports of "Engineering Studies" master course 2020 at HBO

#### $\rightarrow$ Research interactions between pilot and lab-scale for resources recovery

Since the pilot at STP Jajmau was not commissioned until January 2023, the adsorbents used are not exhausted enough to exchanged them. Therefore, it does not yet make sense to regenerate them to recover the desorbed heavy metals. however, this can be conducted at a later stage of the pilot test.



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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	Pilot - New Delhi, India
	Netherlands	
Organization(s):	IHE Delft	IITD
	Lead: Eldon Raj, Peter van	Lead: Shaikh Ziauddin
Contact information	der Steen and Mahmoud M.	Ahammad and Ashish Lohar
Contact mormation.	Habashy	
	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 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

Concerning resource recovery in the form of lipids, PSBR 1 had higher lipid content and productivity of 54.3% and 4.1 mg/L.d, while PSBR 2 had lower lipid content and productivity of 18.6% and 3.6 mg/L.d, respectively. The "survival of the fattest" approach has proved that more storage compounds (i.e., lipids) can be extracted from microalgae, which possess the potential of using these microorganisms as a promising source of biofuel. In practical applications, such as the treatment of effluent from the Barapullah drain, the lipid-rich algal biomass can be harvested and processed to recover lipids for biofuel production and other uses. The remaining algal biomass can be used for animal feed or other applications. In addition to lipid extraction, algae biomass can also be used for biogas production via anaerobic digestion, providing an additional renewable energy source. In contrast, treated wastewater from the photoactivated sludge (PAS) system can be reclaimed and used for non-potable purposes such as irrigation, industrial processes, and cooling water, thereby conserving water resources.



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#### 6.4. LABORATORY EXPERIMENTS FOR SELECTED RESOURCE RECOVERY

#### 6.4.1. SUMMARY AND CONCLUSIONS

The main objective was to enhance lipid productivity using microalgae-bacteria consortia in photosequencing batch reactors by assessing the technical feasibility of the "survival of the fattest" mechanism using COD- and TIC-rich brewery wastewater as a feed supply during the light period and ammonium-rich liquid digestate as a feed supply during the dark period.

The lipid content was measured for PSBR 1 and PSBR 2 after reaching the steady state (Figure 6-1). It was found that the biomass cultivated in PSBR 1 had higher lipid content (54.3%) compared to the one cultivated in PSBR 2 (18.6%). Also, the measurement of PSBR 1 had a standard deviation of ~8.5% (n=3) compared to PSBR 2 which had a standard deviation of ~2.0% (n=3); however, the lowest lipid content measured for PSBR 1 (47.3%) was still higher than the highest lipid content measured for PSBR 2 (20.3%).



Figure 6-1: Comparison of the lipid content and productivity between PSBR 1 and PSBR 2 (n=3).

In addition, despite the higher biomass productivity of PSBR 2 (19.2 mg-TSS/L.d) over PSBR 1 (7.6 mg-TSS/L.d), the lipid productivity was higher in the case of PSBR 1 (Figure 6-1**Error! Reference source not found.**). The lipid productivity of PSBR 1 biomass (4.1 mg/L.d) was ~12% higher than PSBR 2 biomass (3.6 mg/L.d). Microalgae in PSBR 1 were able to produce more storage compounds, i.e., lipids, due to the presence of light and carbon sources during the light period. In the presence of ammonium in the dark period, microalgae consumed part of these storage compounds to assimilate the nitrogen, increasing biomass productivity.

Based on the analysis performed and stated above, the following conclusions were derived regarding the lipid production on the lab-scale PAS system:







- The "survival of the fattest" hypothesis proved its feasibility to increase the lipid productivity of the microalgae-bacteria consortium cultivated in a photo-sequencing batch reactor using synthetic wastewater as a feed supply.
- There was always a remnant of the carbon and ammonium sources after each cycle as the biomass was not able to remove 100% of the TIC during the light period and NH<sub>4</sub><sup>+</sup> - N during the dark period.
- The results showed that higher lipid content and productivity could be achieved in PSBR 1 (54.3% and 4.1 mg/L.d, respectively) compared to the biomass that was cultivated under normal conditions in PSBR 2 (18.6% and 3.6 mg/L.d, respectively).
- The cultivated biomass in PSBR 1 was able to have higher COD, TOC, TIC, NH4<sup>+</sup> 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.
- The nutrient limiting conditions did not favour the biomass growth and hence, the biomass productivity and chlorophyll-a concentration were low in PSBR 1 with values of 7.6 mg-TSS/L.d and 1.5 mg/L, respectively. While the biomass productivity and chlorophyll-a concentration of PSBR 2 were 19.2 mg-TSS/L.d and 4.9 mg/L, respectively.

#### **References:**

Mahmoud Mohamed Habashy Ahmed Abdelhamid (2021) Enhancement of lipid productivity using microalgae-bacteria consortia in photo-sequencing batch reactors. MSc Thesis, IHE Delft Institute for Water Education, Delft, the Netherlands.







# 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,	Pilot: Jajmau WWTP Kanpur,
	Italy	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 MBR, the advantages of SFD MBR are: higher productivity (fluxes up to hundreds L m<sup>-2</sup> h<sup>-1</sup>), 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 water reclamation in remote areas.

# 7.3. TECHNOLOGY-SPECIFIC SUMMARY AND CONCLUSIONS

SFD MBR bench scale process optimization aims at defining operating conditions that minimize the cost of operation, both in terms of energy requirements, workforce, and by-products. In terms of energy, the limited pressure required for filtration (below 200 mbar) allows gravity-driven operation, allowing for savings compared to conventional MBR technology. The development of automatic maintenance strategies aims at obtaining long operating periods without the need to manually clean the filtration modules, thus reducing the need for human interventions. The possibility of operating under high sludge retention times (SRT) above 30 days limits the biomass growth and the consequent sludge production.

# **7.4.** LABORATORY EXPERIMENTS FOR SELECTED RESOURCE RECOVERY TECHNOLOGIES (ENERGY, WATER AND NUTRIENTS)

**Struvite recovery:** Five non-digested and digested sewage sludges from 3 different sewage treatment plants in Kanpur and Delhi and the SFD-MBR pilot were sampled during August to September 2023. Samples were dried and shipped to FHNW for further processing and analysis (acidification, ICP-OES and ICP-MS analysis for total P and heavy metal contents and release during acidification). Total P and heavy metal concentrations of the five sewage sludges are shown in Figure 7-1. P concentrations are highest from Jajmau SFD-MBR pilot plant sewage sludge (12 ±



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0.13 g/kg DM) and from Okhla sewage sludge (16 ± 2.05 g/kg DM). However, P concentrations found are lower than common ranges of 2.6 - 3.4% phosphorous in dewatered sewage sludge (Cydzik-Kwiatkowska & Nosek, 2020). Some heavy metal concentrations detected in the sewage sludges exceed the EU standards for organic fertilizers (EU-Lex 2019/1009) and the Indian standards for Phosphate Rich Organic Manure (Fertiliser Control (Amendment) Order, 2013). The chromium (Cr) levels in sewage sludge from Jajmau SFD-MBR pilot exceeds the Indian fertilizer standards by 300 times, likely due to the tanneries close to the Jajmau STP area. Cadmium (Cd) are also exceeding the Indian standards up to 100 times, as found for the sewage sludges from Jajmau SFD-MBR pilot, Bingawan and Sajari STPs, indicating industrial activities close to the STPs. Sewage sludge from Kanpur STPs thus cannot be applied directly in agriculture as heavy metal contents are exceeded. These findings are in line with a recent publication (Sude et al., 2024) that analysed 22 sewage sludges in the Ganga River Basin which were found to have high levels of heavy metals and pathogens in the samples. Comparably, the sewage sludge from the new Okhla STP (commissioned in 2023) is compliant with the Indian standards for heavy metals in organic fertilizers.



Figure 7-1: Total concentrations (g/kg DM) of five sewage sludges sampled in Kanpur and Delhi (samples analysed in triplicates) and compared to EU-organic fertiliser standards (red lines; DM: EUR-Lex 2019/100) and Indian standards for phosphate rich organic manure (black lines, Fertiliser Control (Amendment) Order, 2013)

Since sewage sludge should not be applied directly to agricultural fields, phosphorous needs to be recovered from the sewage sludge. Experiments on struvite precipitation in continuously stirred batch reactors adjusted for pH and the addition of an Mg source are currently conducted as part of a MSc thesis at FHNW (July 2023 – June 2024). As a first step, the acidification of the sewage sludges is investigated to increase the recovery of phosphorous and to study the rate of heavy metal dissolution. Sewage sludge samples were acidified by gradually adding 95% sulphuric acid and stirred in a leaching reactor for 1h at 500rpm (Figure 7-2).







CHAPTER 7 - Technology: Self Forming Dynamic Membrane Bioreactor



Figure 7-2: Acidification experimental set up at FHNW

ICP-OES was used to assess the leachate qualities (heavy metals, P). The acidification experiments (Figure 7-3) for Okhla STP sewage sludge and the sewage sludge of the SFD MBR Pilot show that the released amount of dissolved phosphorous increased with decreasing pH. Digested sludge, as from Okhla STP released up to 70% of P, as observed in previous studies (Quist-Jensen et al., 2018). Non-digested sludge, as from the SFD MBR pilot, released around 50% of the phosphorous. Heavy metal dissolution was higher for the Okhla sewage sludge than for the sludge from the SFD MBR pilot. However, with a 6% release of Cr at pH 0.86, the concentrations of Cr in the leachate from the SFD-MBR pilots still exceeds the limit values as per the Fertilizer Control (Amendment) Order, 2013.



Figure 7-3: Release of P and heavy metals into liquid phase at various pH values

The struvite precipitation experiments are on-going beyond the duration of the project. The precipitation experiments are aligned to the Stuttgart process, which uses citric acid to chelate heavy metals in the acidified leachate, before precipitation. This should guarantee a good quality of struvite that is applicable in agriculture. Final results and conclusions will be written-up in the



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MSc thesis, to be submitted in June 2024, with firm plans to submit a peer-reviewed publication. The MSc thesis and peer reviewed publication will further include an economic evaluation of sludge acidification and struvite precipitation and discuss its cost-competitiveness as a recovered product regarding the use of chemicals, equipment, and operational costs.

#### **References:**

- EU-Lex 2019/1009 (2019). Regulation (EU) 2019/1009 of the European Parliament and the Council of 5 June 2019 laying down rules on the making available on the market of EU fertilising products and amending Regulations (EC) No 1069/2009 and (EC) No 1107/2009 and repealing Regulation (EC) No 2003/2003.
- Fertiliser Control (Amendment) Order, 2013 (2013). Ministry of Agriculture. Government of India.
- Sude, G., Rajpal, A., Tyagi, V.K. *et al.* Evaluation of sludge quality in Indian sewage treatment plants to develop quality control indices. Environ Sci Pollut Res 31, 17578-17590 (2024). <u>https://doi.org/10.1007/s11356-023-25320-1</u>
- Quist-Jensen CA, Wybrandt L, Løkkegaard H, Antonsen SB, Jensen HC, Nielsen AH, Christensen ML. Acidification and recovery of phosphorus from digested and non-digested sludge. Water Res. 2018 Dec 1;146:307-317. doi: 10.1016/j.watres.2018.09.035. Epub 2018 Sep 26. PMID: 30292955.

# 7.4.1. SUMMARY AND CONCLUSIONS

#### ightarrow Lab-scale operation for resource recovery

All SFD MBR lab-scale experiments aimed to demonstrate the effectiveness and robustness of this technology and its suitability for producing effluents with quality compatible with irrigation reuse and with lower energy requirements concerning conventional MBR (low pressure filtration). Furthermore, all tests were conducted under completely aerated conditions, to maximize nutrient conservation in the effluent. Indeed, complete nitrification of ammonium to nitrate was always obtained, and most soluble phosphorus was maintained in the effluent.

Therefore, the main resources recovered by this process were nutrients (nitrate and phosphate), energy (in terms of savings concerning conventional MBR), and water suitable for irrigation. Preliminary results from P recovery through acidification of SFD MBR sewage sludge show that up to 50% of the P could be additionally recovered.

All tests described in previous section 7.6 consistently provided effluents compatible for reuse in irrigation, except for the microbial pollution indicator E. Coli, whose concentrations were above the standards for water reuse (10 CFU/100 mL). In fact, dynamic membranes develop on supports having a pore size of 50  $\mu$ m, thus bacterial removal is limited. On the other hand, the high removal of suspended solids and the extremely low effluent turbidity (always below 5 NTU) allows the after use of UV-based disinfection systems. These are particularly suited to water reuse applications because they can be operated on-demand (e.g., in line and upon irrigation) and do not imply the addition of chemicals, and therefore do not generate disinfection by-products.

As already mentioned, complete nutrient recovery was achieved, allowing potential economic and environmental savings in terms of limiting the need for chemical fertilizers during crop cultivation.







Energy savings are related to the possibility of operating membrane filtration by exploiting gravity. In fact, the normal operating pressure for SFD MBR is below 20 mbar, and in any case, it never exceeds 200 mbar. Therefore, a head loss of 20 cm between the influent wastewater and the effluent is enough to ensure filtration. This is about one order of magnitude less than the pressure required for conventional MBR, resulting in much lower energy requirements.

# $\rightarrow$ Process and design optimizations

See Deliverable 3.2, section 7.6.

#### ightarrow Subtask 3.3.3: Research interactions between pilot and lab-scale for resource recovery

See Deliverable 3.2, section 7.6.







# **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	ΙΙΤΚ
	Lead: E. Seftel	Lead: P. Bose
Contact information:	Contact: E. Seftel, B.	Contact: P. Bose
	Michielsen	

# **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

# Laboratory experiments for selected resource recovery technologies (energy, water, and nutrients)

Sorbent 1 was upscaled to produce ~5kg in granulated form and sent to IIT Kanpur for application testing using real permeate water samples.

Batch tests at IIT Kanpur showed a 93-96% Cr removal efficiency at an S/L ratio or 2.5g/100mL influent. The rate constant i.e., K<sub>f</sub> obtained in the range of 0.1 to 2 g/mg/hr.

# 8.4. LABORATORY EXPERIMENTS FOR SELECTED RESOURCE RECOVERY TECHNOLOGIES

# 8.4.1. SUMMARY AND CONCLUSIONS

#### ightarrow Lab-scale operation for resource recovery

The performance of the sorbent materials under equilibrium conditions was assessed. The composition of the sorbent was selected in Task 3.2, and the Cr recovery and regeneration of the



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selected materials were optimized. After optimization, batch tests at VITO were performed to demonstrate the adsorption/regeneration over 5 cycles. Results indicated 95-100% adsorption and regeneration efficiencies over the 5 cycles performed.

#### Conclusions:

- The composition of the structured sorbents was selected: Sorbent 1 with 80wt%LDO/20wt%Bentonite.
- The regeneration of the Sorbent after Cr adsorption, namely Cr elution, was optimized: 2M NaCl solution with neutral pH.
- The potential for resource recovery (Cr) was demonstrated at the lab-scale at VITO over 5 cycles.

**References:** protocol containing a brief description of testing parameters and master thesis of Mitra De Geest prepared at VITO, both shared with IIT Kanpur. Results are published in an open access scientific article: De Geest, M.; Michielsen, B.; Ciocarlan, R.-G.; Cool, P.; Seftel, E.M., Structured LDH/Bentonite Composites for Chromium Removal and Recovery from Aqueous Solutions. Molecules 2023, 28, 4879. <u>https://doi.org/10.3390/molecules28124879</u>

#### ightarrow Process and design optimizations

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, determination of the hydraulic operation parameters, and design of the columns.

#### Conclusions:

- 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 m<sup>3</sup>/m<sup>2</sup>/d and influent Cr(VI) concentration of 1 mg/L.
- the minimum flow rate, 2mL/min

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

#### ightarrow Research interactions between pilot and lab-scale for resource recovery

Three columns are packed with approximately 5kg of structured adsorbent prepared at VITO and sent to IIT Kanpur. The columns are now in operation, after ~20 days the breakthrough is not yet reached.

#### Conclusions:

- The results show the long-term operation of the columns packed with structured Sorbent 1, the Cr concentrations are below the maximum accepted limits (50ppb)
- Column with 5cm diameter and bed depth of 75cm: reduces Cr concentration below 50µg/L of ~165 L of wastewater (operation time of ~42days)



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• Column with 6 cm diameter and bed depth of 75cm: reduces Cr concentration below  $50\mu g/L$  of ~270 L of wastewater (operation time of ~55days)

**References:** short testing report (as ppt slides) prepared by IIT Kanpur and master thesis of Ms. Henna Shaji.



