

Topics in Water Chemistry and Water Technology

2020

Annual Report of the Chair of Water Chemistry and Water Technology
and the DVGW Research Center at the Engler-Bunte-Institut

ENGLER-BUNTE-INSTITUT



Dear colleagues and friends,

last year it was the drought, which repeatedly did show on how endangered and vulnerable our water resources are. This year comes along with the COVID-19 pandemic. It would be nice if I could address less threatening topics in this introduction. But obviously we have to analyze the risks for water management coming along with the SARS-CoV-2 virus. Therefore, within this years report of Water Chemistry and Water Technology at the Engler-Bunte-Institut there is a short report on potential consequences, which might arise from the COVID-19 pandemic.

On the institute level, the technicians and PhD students have been able to continuously work in the labs throughout the last 10 months. Most of the running projects are in time. We did formulate strict rules on how to minimize the risk of a SARS-CoV-2 infection and luckily no positive case has been identified in the Chair of Water Chemistry and Water Technology. A positive message is that we have been able to host guest researchers in 2020. Keke Xiao joined our group as Humboldt stipend early this year.

My water chemistry and water technology research highlights of the year are the publication of the manuscript on MRI visualization of particles in settled aerobic granular sludge (Ranzinger et al.) and the use of LC-OCD to identify main processes during aerobic hydrolysis of waste water particulate material (Alvarado et al.). Both manuscripts are nicely showing the importance of more sophisticated methods in water research to improve process understanding.

Unbelievable but true is that the KIT managed (after a decade) to set up a brand new design for the internet presence (<http://wasserchemie.ebi.kit.edu/index.php>). It is worth to have a look.

Last December, I announced two conferences 2020 in my introduction for the annual report 2019. Despite COVID-19 we organized Biofilms 9 (finally as online version), which was really an experience. Find a short report on that within the issue. The Water Research Horizon Conference has been shifted to June 2021.

In 2020 five PhD students have been able to finally defend their theses. New projects have been won and new PhD students did join the group. The 'Wordle' does nicely highlight the topics, which Ulli Scherer and me extracted from the last annual report. It is a perfect overview of our research.



I would really like to introduce an annual report 2021 without any pandemic or other threat to humans or the environment. Let us see. I personally send my best wishes for 2021 to all of you.

Harald Horn



Biofilms 9 – Online Conference



Michael Wagner

Andrea Hille-Reichel

Gudrun Abbt-Braun

Funding:

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and Sponsors

Partners:

Prof. Johannes Gescher,
Institute of Applied Biology
(IAB), KIT

www.biofilms9.kit.edu

From September 29 to October 1, the Biofilms 9 was hosted at the EBI as an online conference due to the COVID-19 pandemic. The international conference series 'Biofilms' addresses non-medical biofilm research and is organized every two years by various research institutions in Europe. Besides the classical research topics of the biofilm community, this year's conference focused on 'Productive Biofilms', a topic that is currently rapidly gaining in importance.

The session on 'Biofilm Control' covered the presentation of established as well as novel approaches for the monitoring and controlling of biofilm development in a variety of technical applications (e.g. membrane-based processes). The presentations in the sessions 'Biofilm Heterogeneity' and 'Biofilm Matrix' focused on advancing the methods for elucidating heterogeneity and matrix. Furthermore, results on the influence of biofilm heterogeneity on the function of biofilms were reported. 'Biofilm Lifecycle' addressed scientists, who are interested in the relationships between genetics, signal molecules and regulatory mechanisms in biofilm formation and also biofilm dispersion. Electroactive and phototrophic microorganisms were in the focus of the session 'Productive Biofilms'. Several presentations showcased new breakthroughs in the field of microbial electrosynthesis processes using new aerobic biocatalysts.

Especially regarding the situation during the pandemic, we were happy to chair a lively conference with 7 keynotes, 30 talks, 115 poster presentations, 2 live demonstrations of exhibitors and 280 participants from more than 30 countries. The Biofilms 10 will be organized by Katja Bühler (UFZ) in 2022 in Leipzig.

Water Research Network Baden-Württemberg



Ulrike Scherer



Tanja Stahlberger

Funding:

Ministerium für
Wissenschaft, Forschung
und Kunst BW (MWK)

Partners:

Universities in BW

www.wassernetzwerk-bw.de

This year, many activities within the Water Research Network have been affected by the COVID-19 pandemic. Some experimental work in the three collaborative projects could not be carried out as planned, e.g. due to the closure of laboratories, strict conditions for field work and the intensification of childcare. The funding period has therefore been extended until the middle of next year. Accordingly, the Final Conference will also be held in 2021.

In 2020, a further call for proposals offering start-up funds for the preparation of joint projects was launched. Two initiatives have been funded: One of the University of Freiburg dealing with 'Illegal use of water resources' and another one, proposed by the University of Hohenheim, aiming at a better understanding of 'Rivers as integrated connecting factor between landscape, agriculture, urban settlement and delta'. On 18 and 19 February 2020, the project CHARM organised an international symposium on 'Powerful women in science – Future challenges in water resources research' at the University of Stuttgart. In line with the topics of CHARM, five female keynote speakers at different career stages were invited to present their research. At the suggestion of Jale Tosun (Heidelberg University), a Special Issue in WATER was published on 'Attention and water governance: An agenda-setting perspective' including many contributions from the Water Research Network Baden-Württemberg.

Standard Methods for the Examination of Water, Waste Water and Sludge

Due to the COVID-19 crisis, meetings of standardization panels of DIN, CEN and ISO have taken place only non-physically since mid-March 2020. This also held for DIN NA 119 01 03 Arbeitsausschuss 'Wasseruntersuchung', which is in charge of standardizing water analytical methods.

As requested in the contract between DIN and the German Federal Government from 1975, standardization projects to support legislation have been given top priority. At present, the determination of estrogenic substances is being standardized for ground water and surface water monitoring (DIN 38407 52, DEV F 52). Especially the very low environmental quality standard (EQS) for 17 α -ethinylestradiol, 0.035 ng L⁻¹, is an analytical challenge. For drinking water monitoring, a method is in preparation to quantify the C₄ to C₁₃ perfluorocarboxylic acids and perfluoroalkyl sulfonic acids that add up to the parameter 'Sum of PFASs' in the revised Drinking Water Directive (98/83/EC) to come. Further fluorinated organic compounds subject to a standardization project are difluoroacetic acid (DFA) and trifluoroacetic acid (TFA) to be determined in surface water, ground water and drinking water using LC-MS/MS.

Organically bound fluorine can also be quantified in terms of the sum parameter AOF by adsorption to activated carbon followed by combustion and ion chromatography, as specified in future DIN 38409-59 (DEV H 59). This method, which at present is in the stage of a draft standard, additionally allows for determination of adsorbable organic chlorine (AOCl), adsorbable organic bromine (AOBr) and adsorbable organic iodine (AOI).

Standardized methods for water examination of the technical committee DIN NA 119 01 03 AA become part of the loose-leaf collection 'Deutsche Einheitsverfahren zur Wasser-, Abwasser- und Schlammuntersuchung', jointly edited by the German Water Chemistry Society and the DIN Standards Committee 'Water Practice'.



Birgit C. Gordalla

Perfluorobutanoic acid (PFBA)	Perfluorononanoic acid (PFNA)
Perfluorobutane sulfonic acid (PFBS)	Perfluorononane sulfonic acid (PFNS)
Perfluoropentanoic acid (PFPA)	Perfluorodecanoic acid (PFDA)
Perfluoropentane sulfonic acid (PFPS)	Perfluorodecane sulfonic acid (PFDS)
Perfluorohexanoic acid (PFHxA)	Perfluoroundecanoic acid (PFUnDA)
Perfluorohexane sulfonic acid (PFHxS)	Perfluoroundecane sulfonic acid
Perfluoroheptanoic acid (PFHpA)	Perfluorododecanoic acid (PFDoDA)
Perfluoroheptane sulfonic acid (PFHpS)	Perfluorododecane sulfonic acid
Perfluorooctanoic acid (PFOA)	Perfluorotridecanoic acid (PFTTrDA)
Perfluorooctanesulfonic acid (PFOS)	Perfluorotridecane sulfonic acid

Target compounds listed in Annex III of the proposal for a revised European Drinking Water Directive (98/83/EC) and to be determined as 'Sum of PFASs'. The intended parametric value for the sum will be 0.10 $\mu\text{g L}^{-1}$. Perfluorinated compounds are of concern because most of them are persistent, bioaccumulative and toxic.

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GDCh)

The Umweltbundesamt
(UBA)

Review Article in Nature Sustainability: Rethinking Waste Water Risks and Monitoring in Light of the COVID-19 Pandemic

Partners:

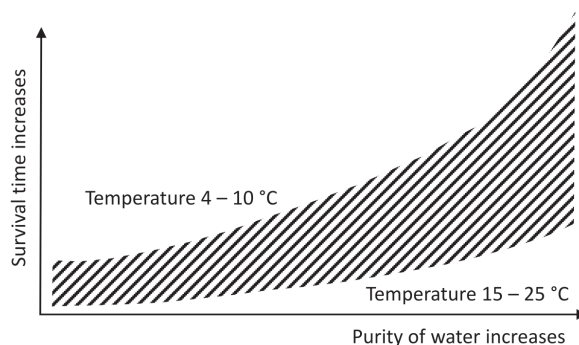
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 Christophe Dagot (France)
 Colin Hill (Ireland)
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Although SARS-CoVs are primarily respiratory viruses, SARS-CoV-2 may replicate in the gastrointestinal tract. Hence, the viruses also enter domestic waste water via human excretions and reach waste water treatment plants (WWTP). To date, only a few studies have succeeded in isolating infective SARS-CoV-2 from stool and urine samples. Nevertheless, controlled experiments indicate that SARS-CoVs are present in waste water for several days, which may lead to potential health risks via waterborne pathways, e.g. when treated waste water is discharged into the environment or reused for irrigation.

Factors that were found to affect SARS-CoVs infectivity in water and waste water include temperature, organic content and pH. Temperature is an important factor for the survival of virions. Longer retention of SARS-CoVs infectivity has been observed at lower temperatures, implying that in cold seasons and temperate climate zones the survival of SARS-CoV-2 may be increased (see Figure). In WWTPs, virions can potentially be removed through physical, biological and chemical processes. Although no specific data for SARS-CoV-2 are yet available, enveloped viruses are more likely to be removed together with particles. Thus, treatment processes that maximize retention and removal of solids (for example, membrane bioreactors) have been suggested as a particularly efficacious means to remove viral loads. However, sufficient inactivation of SARS-CoV-2 is not ensured and disinfection of treated waste water may currently be the most important step to ensure reliable inactivation. In addition, low-pressure membrane filtration, which includes microfiltration (MF) and ultrafiltration (UF), is an advanced technology used in waste water treatment with potential to provide a complete barrier to SARS-CoV-2 dissemination.

Monitoring of SARS-CoV-2 in waste water has recently been highlighted as a powerful tool to provide real-time information on the distribution complementary to individual testing. Moreover, this information can be used as an early warning signal for COVID-19 outbreaks and provides the ability to reinstate containment measures and allocate healthcare resources before COVID-19 infections become highly prevalent.

Overall, this review highlights the urgent need for enhanced monitoring, risk assessment and new risk management strategies for COVID-19 in waste water. For details, please see Bogler et al. (2020, open access).



Rough estimate of the survival time (t_{90}) of SARS-CoV or comparable enveloped viruses. It can be seen that temperatures below 10 °C increase the survival time, as does the purity of the water in which the viruses are found. The y-axis ranges from a few hours to several 100 days.

<https://www.nature.com/articles/s41893-020-00605-2>

Increasing Water Availability in Arid Areas: A Method to Assess the Impacts and Adaptability of Different Water Technologies for Sustainable Development

The decision to use a specific water source and the corresponding treatment technologies for the water supply of a city has multiple components; it is a long-term decision with high investment that will influence the development path of a territory. This is especially relevant for arid areas, where water resources are the main constraints to human growth and where their allocation is an important source of potential social, environmental and economic conflicts.

Taking the city of Arica (Chile), located in the North of the Atacama desert, as case study, this ongoing doctoral project has developed a decision-making tool based on a set of 23 indicators, with which three scenarios of urban water supply are being assessed. Using the Integrative Concept of Sustainability (ICoS) as the overarching theoretical approach and a participatory, interview based approach for the election of indicators, the tool incorporates areas of development relevant for citizens, farmers and state agencies. The scenarios are differentiated by the use of sea water desalination and/or the reuse of municipal waste water to fully or partially supply the drinking water for the city. From this assessment, four indicators are preliminarily considered to be core indicators: 'water availability for agriculture', 'just distribution of green areas', 'ground water over-exploitation rate' and 'household water expenditure'. They inform decision-makers about the most notorious impacts of implementing different water treatment technologies and water sources on the sustainable development of arid areas.



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Florencia Saravia

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Partners:

Dr. Helmut Lehn, Institute for Technology Assessment and System Analysis (ITAS), KIT

Review Article in WIREs Water: Making Europe go from Bottles to the Tap: Political and Societal Attempts to Induce Behavioral Change

Recently, initiatives that aim to promote the drinking of tap water are in place in Europe. Most prominently, the European Commission proposed revisions for the Drinking Water Directive, which includes strategies for promoting the consumption of tap water, including to improve access, upgrade quality standards, and enhance transparency concerning the benefits of tap water. National initiatives in European countries pursue similar objectives: Many initiatives are launched by water providers (26 %), actively promoting tap water as their product. Nonprofit organizations (19 %) mostly focus on environmental and consumer protection. The private sector (11 %) is also involved with some initiatives as they regard tap water a business opportunity. Interestingly, local as well as some national governments have launched tap water advertising campaigns, too. The great majority of initiatives mentions environmental protection (79 %). Among these, about 53 % explicitly refer to the goal of reducing plastic pollution and almost 15 % state that they strive to reduce CO₂ emissions.

Nevertheless, consumption of bottled water in some European countries, such as Italy or Germany is still increasing. Research has shown that sensory qualities, together with safety concerns, are responsible for low levels of tap water consumption. However, perceived risks often differ from science-based estimates of the risks. For details, please see Tosun et al. (2020, open access).

Ulrike Scherer

Partners:

Prof. Dr. Jale Tosun and Simon Schaub, Institute of Political Science, Heidelberg University, Germany

<https://onlinelibrary.wiley.com>

DEMO-BioBZ – Development and Demonstration of an Energy-Efficient Bio-Electrochemical Waste Water Treatment



Andreas Netsch



Michael Wagner

In the quest for sustainable waste water treatment technologies, microbial fuel cells have emerged as a promising opportunity with a unique combination of chemical oxygen demand (COD) and nitrogen removal and the direct generation of electricity. Following the *BioBZ* project, awarded with the 'Deutscher Nachhaltigkeitspreis' in 2018, the *DEMO-BioBZ* project aims to use the gained knowledge in bio-electrochemical systems (BES) for the upscaling into an industrial scale BES (e.g., microbial fuel cell, MFC) applied in municipal waste water treatment.

The project aims - including the design and installation of submers-modules, the advance of material development for membrane electrode assemblies for cost optimization, as well as the optimization of energy harvest - will be addressed in an interdisciplinary network of partners. We are involved in the integration of bio-electrochemical nitrogen elimination in the cathodic chamber of a MFC system as well as in the online determination and control of biofilm accumulation on the electrodes of the MFC system. Electrodes are cost-efficiently reduced by manufacturing electrodes from a graphite/polypropylene (C/PP) compound material.

With the use of biofilm sensors (*DEPOSENS*), biofilm accumulation on the C/PP anode is monitored non-invasively and online. In a first step, the compatibility of biofilm sensors with the C/PP compound material is evaluated. Results revealed that data about biofilm accumulation on anodes correlate with data collected on standard stainless steel based biofilm sensors. Next steps include the optimization of data interpretation as well as the correlation to the overall BES performance.

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CUTEC Forschungszentrum
and Institute of Chemical
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Process Engineering, both
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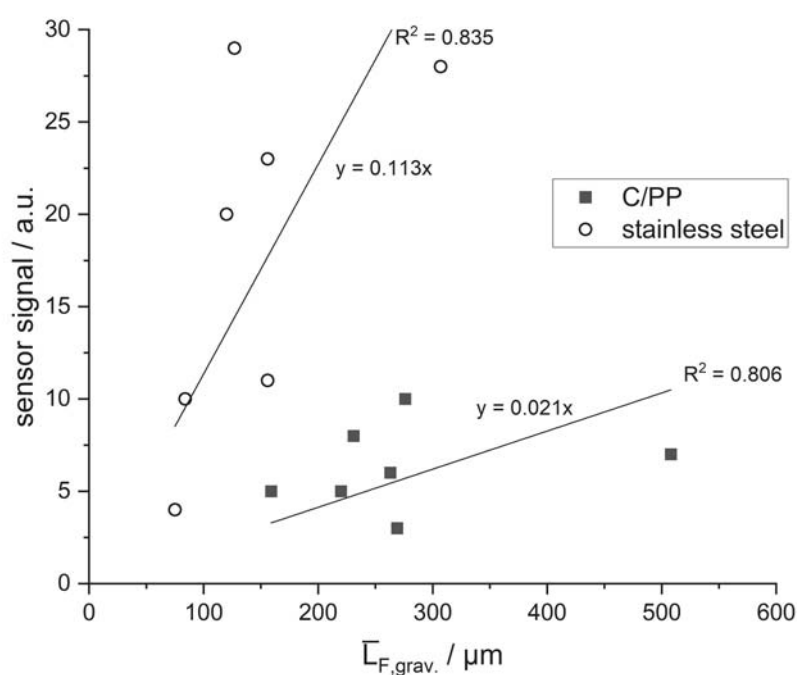
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Comparison of biofilm accumulation in C/PP and stainless steel pipe sensors.

BioElectroPlast – Microbial Electrosynthesis for Production of Bioplastics from Flue Gas

The project *BioElectroPlast* aims at the microbial production of the biodegradable biopolymer polyhydroxybutyrate (PHB) from flue gas. The thermophilic, acidophilic and electrolithoautotrophic bacterium *Kyrpidia spormannii* was used as biocatalyst. This electroactive *Knallgas* bacterium is able to directly use electrons as energy and electron source to reduce carbon dioxide and intracellularly accumulate PHB as storage compound. Hence, the development of strategies for harvesting the PHB saturated biomass from a continuously operated reactor is necessary to subsequently extract the valuable bioproduct.

As *Kyrpidia spormannii* grows as biofilm on graphite cathodes, harvesting by mechanically shearing off the biofilm could easily damage the cathode surface. A more moderate strategy would be the intensification of shear stress by increasing flow velocity. However, since the biofilms proved to be firmly attached to the substratum, detachment experiments were not satisfactory. Finally, given that the biofilm grows on a cathode, it seemed obvious to try to harvest the PHB-rich biomass by controlled water splitting. The hydrogen bubbles formed on the cathode surface detach the biofilm by tearing round holes into the biofilm matrix. This process was visualized *in situ* using optical coherence tomography (OCT). Holes formed by hydrogen bubbles are nicely identified in height maps generated from OCT datasets (see purple/blue circles in the Figure). The applied potential at the cathode was increased stepwise, each time held for 2 minutes and then stopped (0 V). With increasing potentials and thus longer treatment, more biomass was detached as shown by the decrease of biovolume and substratum coverage.

For biotechnological downstream processing, the detached biomass can easily be separated from the liquid phase and the produced hydrogen can be used to cultivate autotrophically growing precultures.

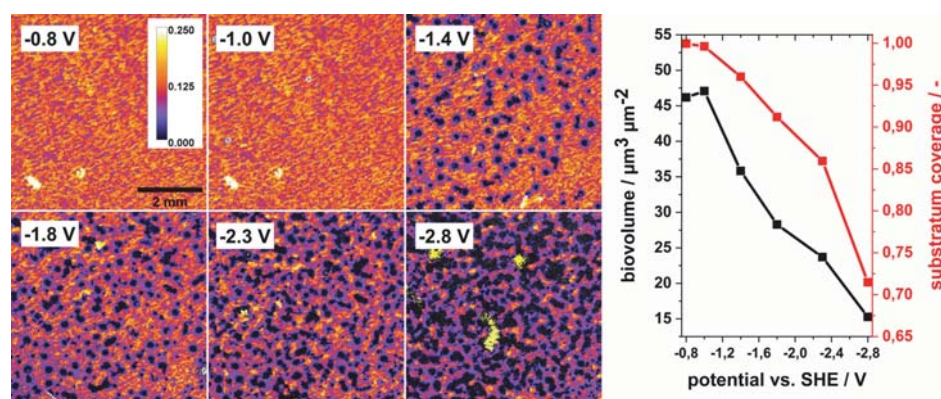


Max Hackbarth



Andrea Hille-Reichel

Michael Wagner

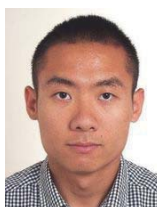


Time series of height maps generated from OCT datasets (left) and quantification of biovolume ($\mu\text{m}^3 \mu\text{m}^{-2}$) and substratum coverage (-) during the course of the harvesting experiment (right).

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Partners:
Prof. Johannes Gescher,
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Environmental Process
Engineering, University of
Bremen, Germany
EnBW Energie Baden-
Württemberg AG, Germany

RECICL – Reactor Cascades for the Biotechnological Conversion of Waste into Platform Chemicals



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Rowayda Ali

Florenca Saravia

Andrea Hille-Reichel

Michael Wagner

The *RECICL* project aims at the optimization of propionic acid (PA) production from food waste by combining acidic hydrolysis, bioelectrochemical systems (BES) and membrane filtration. An anaerobic hydrolysis reactor was operated with food waste at a pH value of 6.0 ± 0.1 and mesophilic ($30\text{ }^{\circ}\text{C}$) temperature to produce volatile fatty acids (VFAs). Soft goat cheese was used as inoculum to optimize PA productivity. High PA production of up to 105 mmol L^{-1} and a maximum yield of 54 mg g^{-1} PA per volatile solids added were obtained.

As one of the primary steps in PA recovery, a pretreatment of the fermentation broth was developed including two steps: removal of large particles from the fermentation broth by using a separation unit (with $60\text{ }\mu\text{m}$ mesh size), followed by removal of the other suspended particles by a submerged microfiltration membrane system with different pore sizes of $0.1\text{ }\mu\text{m}$, $0.45\text{ }\mu\text{m}$, and $0.8\text{ }\mu\text{m}$. The microfiltration membranes showed a good performance resulting in VFA rich and particle-free solution.

BES such as microbial fuel/electrolysis cells were applied with different microorganisms to further achieve purification of propionic acid from the VFA-rich solutions. The goal is to have butyric and acetic acid microbially degraded whereas propionic acid remains in the liquid. To achieve this goal, microorganisms were cultivated as biofilms on electrodes made from carbon veil (CV) and porous stainless steel (SS), respectively. Methods were developed to characterize and *in situ* quantify the biofilm structure on electrodes by means of optical coherence tomography (see Figure). It turned out that the error of quantitative analysis of biofilm developing on SS electrode is much smaller in comparison to the one growing on CV. Main reason is the irregular structure of the CV, which is composed of heterogeneously distributed carbon fibers. To allow for the correlation of biofilm structure and VFA purification, electrodes made from SS are to be preferred.

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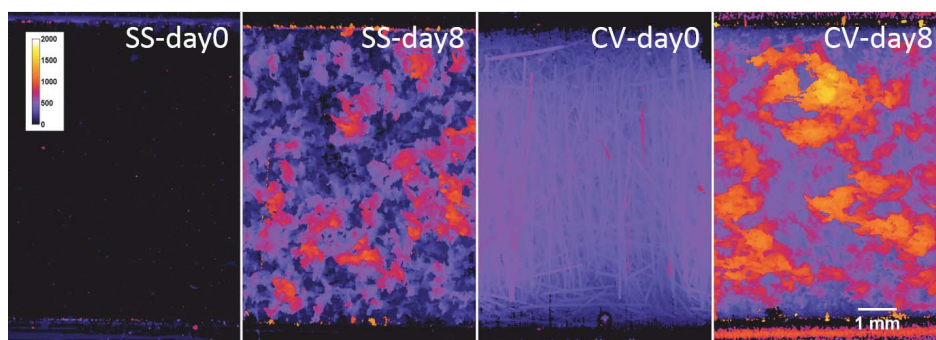
Islamic Development Bank
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Programme, Saudi Arabia

Partners:

Prof. Johannes Gescher,
Institute of Applied Biology
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Environmental Process
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Biofilm topographies (height map, substratum-biofilm interface) of two electrodes (SS vs CV) in BES. Image size is $6 \times 8\text{ mm}^2$. The unit of the calibration bar (in the graph on the left) is μm . Flow direction is from left to right.

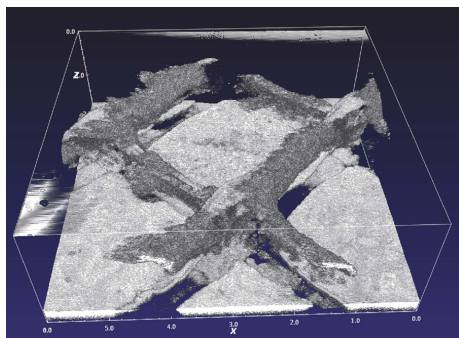
ProBioLNG – Nanofiltration/Reverse Osmosis for Organic Acids Concentration and Biofouling Detection

The aim of the project *ProBioLNG* is the development of a sustainable process chain for the production, distribution and utilization of liquefied biogas (BioLNG). At the beginning of the chain, biogas is produced by a two-stage fermentation. The main goal of the DVGW Research Center at the Engler-Bunte-Institut, Water Chemistry and Water Technology is to apply nanofiltration (NF)/low pressure reverse osmosis (LPRO) to the effluent of the first reactor. In this way, the ultra-fast degradable organic compounds can be concentrated, optimizing the quality of the biogas and minimizing the volume of the methane reactor.

The effluent coming from the first reactor (hydrolysate) will be pre-treated using a microfiltration (0.22 μm). The permeate from the microfiltration has both a dissolved organic carbon (DOC) and a total nitrogen of about 10 g L⁻¹ and relatively high salt concentration (electrical conductivity 21 mS cm⁻¹). Most of the DOC (approx. 70 %) consists of volatile fatty organic acids, principally acetic acid (12 g L⁻¹). The concentration of the organic acids is investigated in a membrane set-up designed to operate under anaerobic condition with pressures up to 40 bar. In the course of the first experiments, several membranes have been tested to find the most adequate one for the purposes of this work. Results show that up to 85 % of all organic acids can be retained by the membrane.

Moreover, optical coherence tomography (OCT) is used as monitoring tool to optimize membrane filtration. According to the water composition, biofouling will be a key issue for the application of NF/LPRO in the two-stage fermentation.

Further investigation will focus on the development of the OCT-monitoring system combined with on-line sensors to optimize membrane performance. Results should provide a deeper understanding on biofouling development and biofilm structure in spiral wound membrane modules.



OCT image of the biofilm growing on the feed spacer (left). Photograph of the membrane set-up used to concentrate the solution (right).



Giorgio Pratofiorito



Florencia Saravia

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DVGW Research Center at the Engler-Bunte-Institut, Gas Technology, KIT

Institut für Fahrzeugsystemtechnik, Institutsteil Mobile Arbeitsmaschinen, KIT

Air Liquide Forschung und Entwicklung GmbH, Frankfurt am Main, Germany

CLAAS Selbstfahrende Erntemaschinen GmbH, Harsewinkel, Germany

LIQUIND 24/7 GmbH, Berlin, Germany

Processing of Hydrothermal Liquefaction Products with Membrane Technologies



Ali Sayegh

Florence Saravia

As an attempt to increase renewables in transport energy fuels, the EU launched the *NextGenRoadFuels* project. In this project, biogenic urban resources defined as sewage sludge from treated waste water, food waste and construction wood waste is co-processed by our partners in single hydrothermal liquefaction (HTL) facilities. This leads to the production of bio crude oil as well as solid, liquid and gaseous byproducts.

The liquid byproduct has high concentrations of dissolved organic carbon (30 g L^{-1} DOC) and total nitrogen (12 g L^{-1} TN), of which 50 % is in the form of ammonium. Organics should be concentrated and introduced back into HTL, while ammonium should be recovered separately. In addition, oil-water-solid emulsions should be processed to recover the oil phase from the water phase.

In this context, the Chair of Water Chemistry and Water Technology at the Engler-Bunte-Institut (EBI) combines the tasks of ammonium separation and oil recovery in one semi-dead-end filtration process. In this system, the driving force of filtration is a vacuum pressure on the permeate side of the membrane. In addition, air stripping of 40 L h^{-1} is done on the feed side of the membrane to create shear force, thus reducing the cake layer formation. Furthermore, for better control of fouling, either relaxation or backwash was applied. Two poly-ether sulfone membranes were used: One is a microfiltration (MF) membrane defined by $0.45 \mu\text{m}$ pore size, and the other is an ultrafiltration (UF) membrane defined by 100 kDa molecular weight cutoff.

Results show that permeability of the UF membrane is more stable than that of the MF membrane, with backwash being a better option than relaxation. Oil droplets are small enough to pass through pores of $0.45 \mu\text{m}$, decreasing hydrophilicity of the membrane, and hence permeability. This is not the case for the UF membrane, which retains oil. Furthermore, it was shown that this process allows to strip and recover approx. 90 % of ammonia, while only 10 % of DOC is lost in volatile form. The membrane filtration/stripping method opens new possibilities to treat HTL-water streams and recover ammonium.

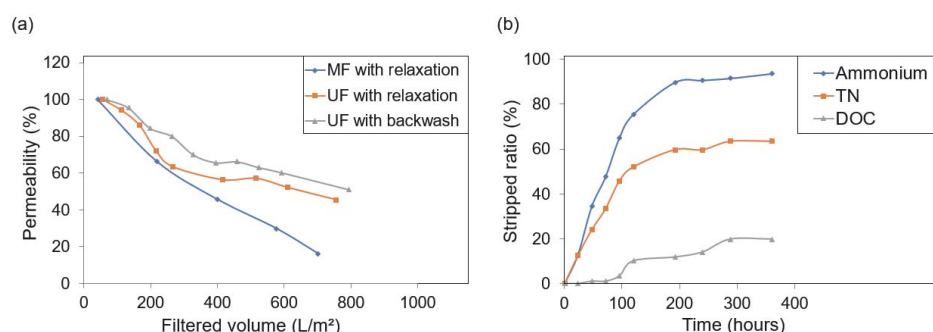
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and other partners from
Norway, Denmark, the
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Spain, Italy and Greece



(a) Permeability of the liquid by-product. (b) Stripped ratio of ammonium, TN and DOC.

www.nextgenroadfuels.eu

Complete Treatment of Pig Manure by Pilot Scale Membrane Filtration Process

The handling of liquid manure from animal husbandry is undisputedly a very current topic not only in Germany, but also worldwide. Liquid manure is usually used as a nitrogen-rich agricultural fertilizer, especially in regions with a high livestock population. This has a considerable impact on ground water quality and leads to an exceeding of the nitrate limit value according to the Drinking Water Ordinance ($50 \text{ mg L}^{-1} \text{ NO}_3^-$).

Recent studies on a fully integrated membrane filtration system for digestate water treatment have underlined the need to develop economical and robust filtration processes. Here, we report on the effectiveness of a cross-flow pilot scale ceramic microfiltration (MF) membrane ($0.2 \mu\text{m}$ pore size) filtration system for the complete treatment of pig manure.

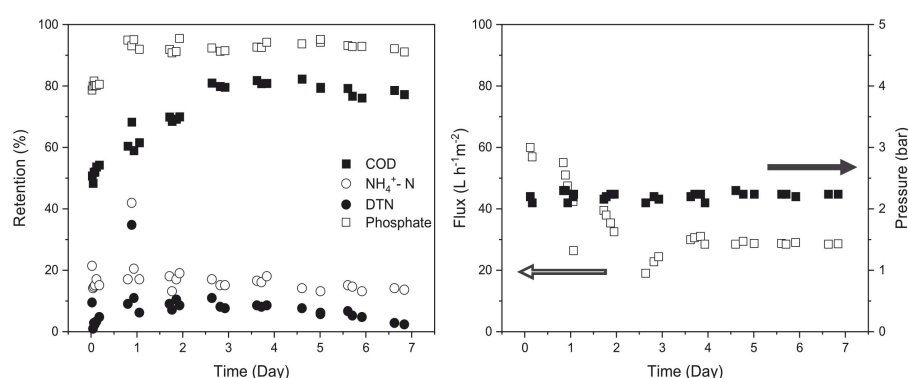
Results show that a ceramic MF membrane can retain 80 % chemical oxygen demand (COD) and more than 90 % phosphate in the concentrate. The latter is of high interest for phosphate recovery. Compared to municipal waste water the phosphate is mainly in the particulate and not in the liquid phase.

90 % dissolved total nitrogen (DTN) and 80 % $\text{NH}_4^+\text{-N}$ remain in the filtrate. During the operation of the membrane unit, the permeate flux decreases initially until the end of the second day due to the initially considerable fouling of the membrane. From the third day onwards, however, it stabilizes at a flux rate of $30 \text{ L h}^{-1}\text{m}^{-2}$. The continuous filtration of pig manure through ceramic microfiltration membranes can remove total suspended solids (TSS) by more than 98 %. The product is a particle-free solution enriched with ammonium nitrogen, which can be recycled or further processed for regenerative ammonia production.



Prantik Samanta

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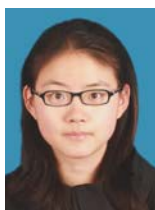


COD, DTN, $\text{NH}_4^+\text{-N}$ and phosphate retention (left). Permeate flow and pressure during continuous filtration of pig manure through a $0.2 \mu\text{m}$ ceramic MF membrane filtration system on a pilot scale (right).

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(BMBF)

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BIORESTEC UG, Germany
Institute for Sanitary Engineering and Waste Management (ISAH), Leibniz University Hannover, Germany
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Understanding the Role of Organic Matter on the Occurrence and Fate of Antibiotics and Antibiotic Resistance Genes in Sewage Sludge



Keke Xiao
(Guest Researcher)

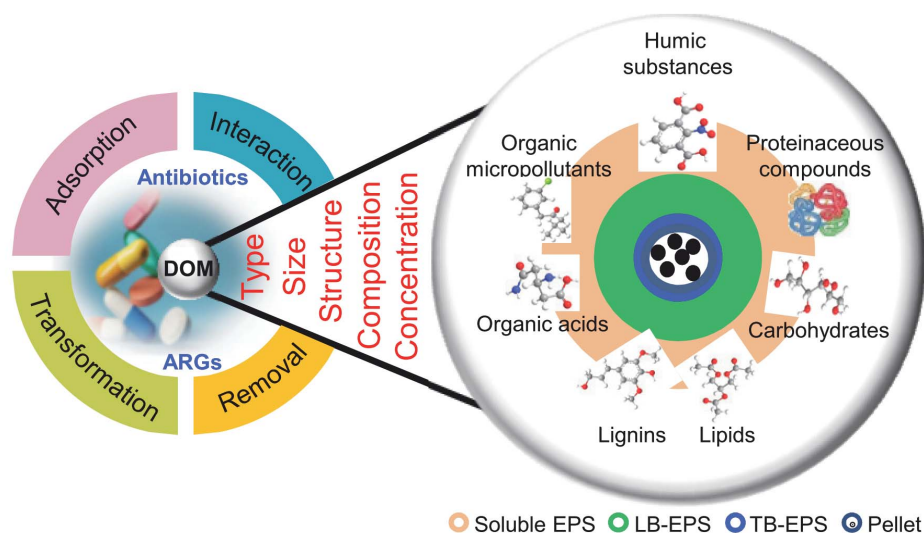


Gudrun Abbt-Braun

Various antibiotic compounds are present in sewage sludge due to their limited mobility and low biodegradation. Meanwhile, the selection pressure originating from residual antibiotics in sludge may encourage the formation and spread of antibiotic resistant bacteria and antibiotic resistance genes (ARGs) in a long term. Dissolved organic matter (DOM) of sludge plays an important role in organic micropollutants removal. The subunits of DOM, including proteinaceous compounds, carbohydrates, humic substances, lipids, lignins, organic acids, and other biological derived substances, showed different capability towards the adsorption of antibiotics. Meanwhile, advanced oxidation processes like sulfate-based processes are typically used to remove antibiotics and ARGs in sludge. However, the presence of DOM inhibited the oxidation of aromatic amines of anilines and sulfonamides initiated by excited triplet states of DOM (3DOM*).

Given the importance of DOM in both the presence of antibiotics and ARGs in sludge and their removal during the sulfate-based processes, it is crucial to understand the role of DOM for the occurrence and fate of antibiotics and ARGs in sewage sludge. It is expected that this project can provide new insights into the role of DOM in the removal of antibiotics and ARGs in sludge and facilitate the reuse of sludge in agricultural application.

Funding:
Alexander von Humboldt
Foundation



DOM properties determine the behavior of antibiotics and ARGs in sludge. EPS: Extracellular Polymeric Substances; LB-EPS: Loosely-Bound EPS; TB-EPS: Tightly-Bound EPS.

Removal of Heavy Metals from Aqueous Solutions Using a Customized Electro-Membrane Process

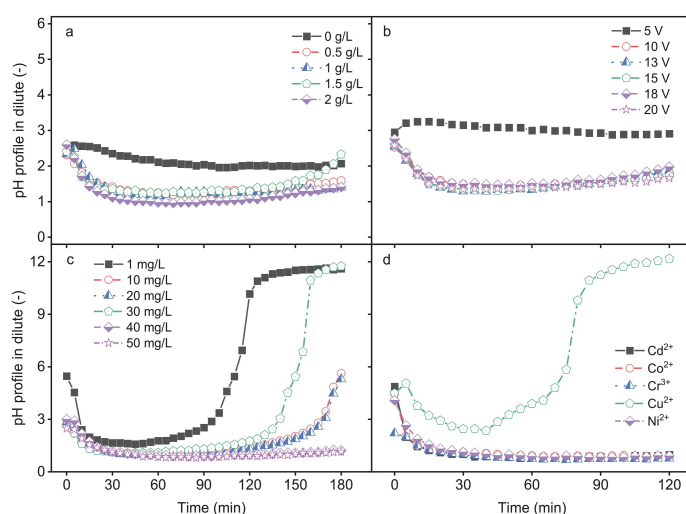
Many natural resources of water are contaminated with heavy metals (HM) and inorganic ions that make water unsafe for drinking or for other potable water applications. In addition, raw water may contain higher concentrations of natural organic matter (NOM) which requires additional treatment.

In this study, a customized bipolar electrodialysis (BMED) is used for the removal of HM from aqueous solutions. The process is configured by integrating a bipolar membrane in the feed side of a conventional electrodialysis. The stack of BMED is completely assembled with 5 two-chamber units (feed and concentrate chambers) by using standard cation exchange membranes (PC SK), bipolar membranes and anion exchange membrane (PC Acid 60) and electrodes (anode: Pt/Ir-MMO coated Ti-stretched metal, cathode: stainless steel). The thickness of the feed and concentrate channels is about 1.5 mm and 0.5 mm, respectively. The sizes of the cell used are 110 x 110 mm and the active area of each membrane is 64 cm². The process is expected to adjust pH of the dilute solution, to reduce the solution stack resistance and to influence the chemical compound species distribution without addition of acid or base. The results show that the factors such as feed ionic strength (0 – 2 g L⁻¹ of NaCl), applied electrical potential (5 – 20 V), HM concentration (1 – 50 mg L⁻¹) and the type of HM (Cd²⁺, Co²⁺, Cr³⁺, Cu²⁺ and Ni²⁺) differently influence the pH of the dilute (see Figure). The change of pH shows 4 steps: Decrease and stabilization in acidic pH range, followed by an increase and stabilization in alkaline pH range. However, all those steps were achieved only with the lower concentration of HM and with Cu²⁺. The process achieved a removal efficiency of HM higher than 95 % when the feed concentration of HM, ionic strength and applied potential were ≤ 30 mg L⁻¹, 1.5 g L⁻¹ of NaCl and 18 V, respectively. A lower electrical conductivity removal efficiency and higher current flow were observed.

The process is able to remove HM and has additional features over existing electro-membrane processes. Those features are self pH adjustment of dilute and reduction of stack resistant by maintaining higher current flow.



Samuel Bunani
(Guest Researcher)



(a) Impact of ionic strength, (b) impact of electrical potential, (c) impact of HM concentrations and (d) impact of the type of HM on the pH profile in dilute as a function of time.

Funding:
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Foundation
Karlsruhe Institute of
Technology (KIT)

Fixation of Microplastics from Water Using Organosilanes for Agglomeration Followed by Flotation



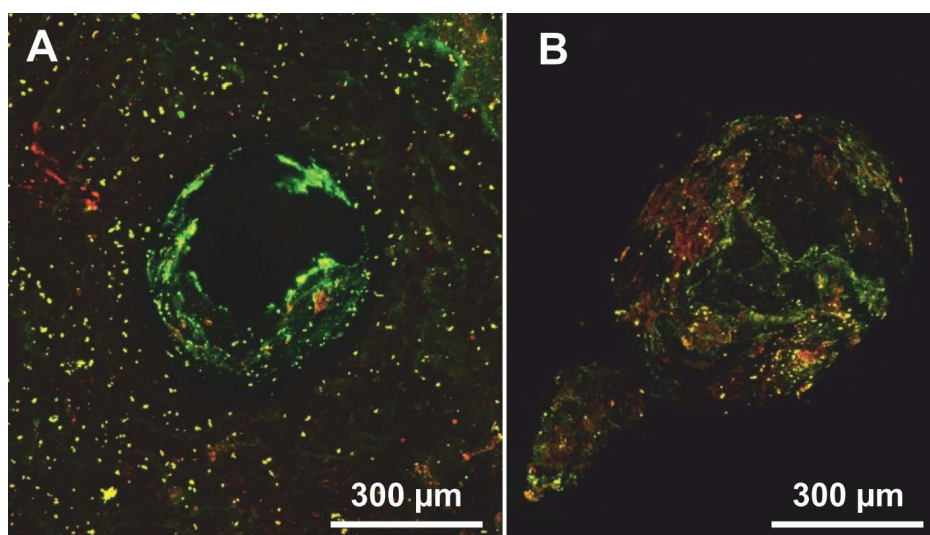
Michael Sturm

An increasingly serious and widespread problem is the emission of plastics and microplastics (MP) into the water cycle. Nowadays, MP are ubiquitous and can be found in all parts of the environment.

To reduce the emission of MP and avoid negative impacts of MP on processes such as sea water desalination or sea salt extraction, there is a need for a cheap and easy applicable method for MP removal from water. Previous studies determined the best suited organosilanes to remove MP from water. When being added to water, the organosilanes collect the MP in large agglomerates floating on the water surface. Due to a water-induced sol-gel process, the liquid organosilanes subsequently form solid hybridsilicas and fix the MP chemically.

In a comparative lab scale study, we applied the process for biologically treated municipal waste water, sea water and demineralized water at temperatures ranging from 7.5 – 40 °C. The different temperatures and types of water showed no negative effect on MP removal. Additionally we compared the efficiency of the process for common polymer types with different properties and chemical compositions. Using organosilanes with nonpolar organic groups, the removal efficiency decreases with increasing polarity of the MP. Highly polar polymers can be removed efficiently by increasing the polarity of the organic group or using higher concentrations of nonpolar organosilanes.

Currently, the effect of biofilm coverage of MP on the removal process is tested as MP present in the environment are usually covered by biofilms, which change the properties and surface chemistry of MP. As final step, the process should be tested and verified for continuous application in waste water and sea water.



Maximum intensity projections of confocal laser scanning microscope (CLSM) image stacks acquired of MP covered with biofilms. Colors: red = nucleic acids; green = extracellular polymeric substance (EPS) glycoconjugates. Therefore, green areas show EPS and red/bright green areas show parts strongly colonized with bacteria.

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Can Carbon Uptake During an Anaerobic Feeding Phase of Aerobic Granules Be Observed via ^{13}C -NMR?

New technologies in waste water treatment aim at cost reduction and compliance of current discharge limits. The aerobic granular sludge (AGS) technology has gained interest due to the possibility to remove carbon, nitrogen and phosphorus sources within one reactor, which saves pumping cost and area needed to build a waste water treatment plant.

In AGS systems, a high density of slow-growing microorganisms like phosphorus accumulating organisms (PAOs) and glycogen accumulating organisms (GAOs) is desired. Especially PAOs are of interest as they are responsible for phosphorus removal within AGS systems.

Overall, there are few methods to monitor locally resolved metabolic performance of microorganisms. We hypothesize that by application of nuclear magnetic resonance (NMR) methods, it must be possible to track ^{13}C -enriched substances *in situ* and non-invasively. Previous studies have revealed the metabolic pathways of ^{13}C -enriched acetate taken up by PAOs within activated sludge via NMR spectroscopy. The limitation is the significant lower sensitivity of ^{13}C compared to ^1H . However, the microbial density of AGS is high compared to activated sludge and the aim of the project must be to generate high ^{13}C concentrations within the AGS during the anaerobic feeding phase.

Therefore, the first part of the project was the design of a sequencing batch reactor. Requirements were the possibility to create anaerobic as well as aerobic conditions in a cyclic order for a small reactor of about 100 mL. The upcoming step is the treatment of AGS within the system until an uptake of 200 mg acetate/g TSS is reached within one anaerobic cycle.

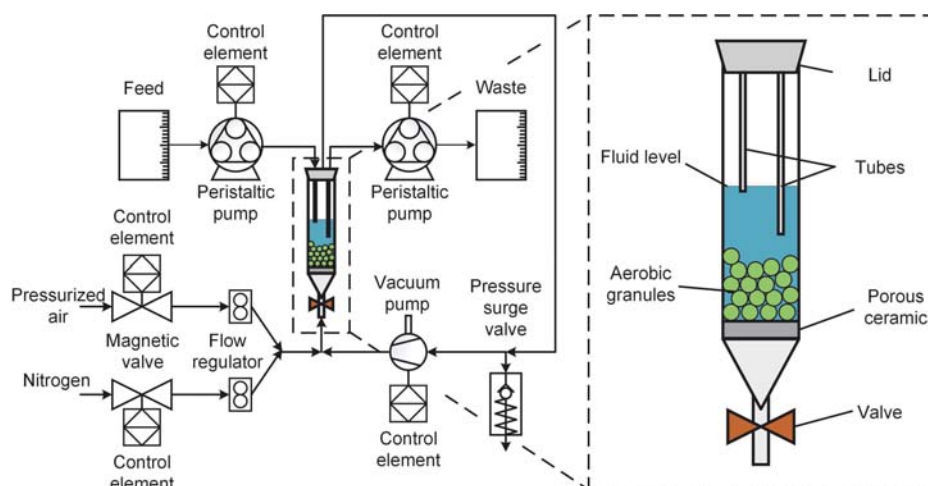


Florian Ranzinger



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Schematic drawing of the sequencing batch reactor setup. Devices and control elements are depicted on the left. A close up of the reactor is shown on the right side.

Funding:
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Germany

Method Development for the Determination of 18 Antibiotics in River Water and Sediments Impacted by Treated Waste Water



Amélie Chabilan

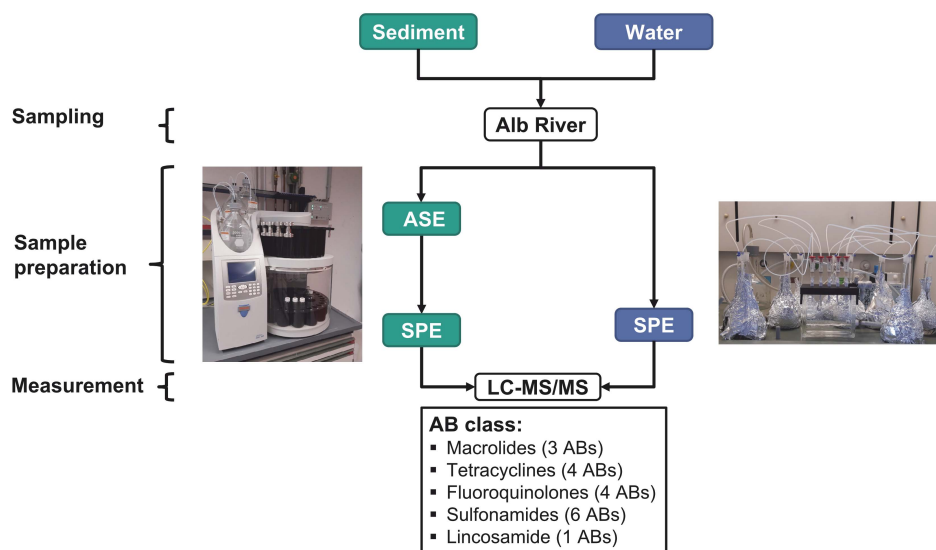


Ewa Borowska

Antibiotics (ABs) are applied against bacterial infection in human and veterinary medicine and represent one of the most important class of pharmaceuticals. Due to their excessive usage, ABs are often discussed in the context of formation and spread of antibiotic resistance (AR). ABs can reach the aquatic environment through different dissemination routes such as discharge of treated waste water or runoff of liquid manure applied to fields. Despite years of studies, the role of ABs in the dissemination of AR, especially in rivers receiving waste water treatment plants effluents, is not fully understood. One of the reasons are analytical challenges to perform a quantification, especially in river sediments that have a very complex matrix and where ABs are only present in a ng/g concentration range.

For the quantification of ABs in river water and sediments, an elaborated sample preparation as well as a sensitive and robust analytical method is necessary. ABs present in sediment samples are firstly extracted using accelerated solvent extraction (ASE). Later on, the extracts are purified and concentrated by solid phase extraction (SPE). The latter technique is also applied for water samples (see Figure). The ABs in prepared samples are quantified by liquid chromatography coupled to mass spectrometry (LC-MS/MS).

Our analytical method includes 18 ABs (3 macrolides, 4 tetracyclines, 4 fluoroquinolones, 6 sulfonamides and 1 lincosamide). The limits of detection for the ABs are in the range of 5 to 10 ng/L. Water and sediment samples used in the method development were collected from a local mid-sized mountain stream river before and after the discharge of treated waste water, and spiked with a multi-mix of ABs. Among all tested SPE protocols, the highest recovery values (50 – 180 %) were achieved when hydrophilic and lipophilic sorbents (HLB) and combination of HLB with cation exchange sorbents were used for water and sediment samples, respectively.



Flow chart of the proposed analytical method for the determination of ABs in sediments and river water.

Funding:
Baden-Württemberg Stiftung

Degradation of Cytostatic Drugs in Water by Oxidation Processes: Kinetics, Degradation Products and Toxicity

Cytostatic drugs are a class of pharmaceuticals used during chemotherapy to treat a variety of cancers. Due to a combination of different factors including increasing life expectancy in developed countries and environmental factors, the usage of cytostatic drugs is likely to increase. As in the case of other pharmaceuticals, cytostatic drugs taken by patients are partly transformed in human body, and formed metabolites together with intact drugs are excreted with urine and/or feces and directed to waste water. As cytostatic drugs often exhibit mutagenic, carcinogenic and teratogenic properties, they may be toxic to water organisms, or even humans, since they are often not readily degraded during waste water treatment.

The PhD project focuses on the fate of selected cytostatic drugs that are known to be recalcitrant to waste water processing and have been detected in surface waters. The ability of different oxidative processes like ozonation and advanced oxidative processes (AOPs) like UV/H₂O₂ to degrade these persistent substances will be tested in laboratory scale experiments. With the help of a sensitive and selective LC-MS/MS-method, the kinetics of these reactions will be determined to gain a fundamental understanding of these processes. Additionally, the structure of transformation products that are formed during these treatments will be elucidated by high resolution mass spectrometry (HR-MS) and different biological tests will be used to assess the toxicity of these transformation products.



Stephan Zimmermann

Ewa Borowska

Funding:

Karlsruhe Institute of Technology (KIT)

ContiBio-Elect – Continuous Bioproduction Using a Tailored Biocatalyst for Electrode Assisted Fermentation

The subject of this project is the design of a reactor for continuous, anaerobic oxidation processes. The conversion we focus on is the oxidation of glucose to the platform chemical acetoin by a bacterial strain of *Shewanella oneidensis*. The removal of the emerging electrons is ensured by cultivating the bacteria as a current conducting biofilm on suitable anode materials. These electrons are further used to synthesize hydrogen as by-product on the cathode.

In preliminary experiments, optimum flow conditions, substrate concentrations and applied voltage for the cultivation of several *Shewanella oneidensis* strains are investigated in microfluidic cells.

It is our motivation to achieve the high space-time-yields known of existing (and often oxic) batch systems in anaerobic continuous production characterized by a high carbon yield and low generation of biomass and by-products.



Maximilian Miehle

Andrea Hille-Reichel

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Federal Ministry of Education and Research (BMBF)

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Head of the laboratory
Standardization
Deputy head, membrane techn.

PhD Students

M.Sc. Rowayda Ali	Production of platform chemicals (Dr. rer. nat. since December 2020)
M.Sc. Alondra Alvarado	Characterization of organic carbon in anaerobic and aerobic waste water treatment
M.Sc. Dámara Araya Valenzuela	Decision support for the implementation of water technologies in arid areas
M.Sc. Annika Bauer	Membrane distillation, waste water recycling (Dr.-Ing. since August 2020)
Dipl.-Ing. Fabian Brunner	Aerobic granules for waste water treatment
M.Sc. Amélie Chablan	Antibiotics in aquatic environment
Dipl.-Ing. Laure Cuny	Productive biofilms
M.Sc. Luisa Gierl	Mechanical and material properties of biofilms (Dr. rer. nat. since November 2020)
M.Sc. Lizheng Guo	Chlorine disinfection, biofilms in drinking water distribution systems (Guest PhD Student)
M.Sc. Max Hackbarth	Microbial electrosynthesis
Dipl.-Ing. Oliver Jung	Organic fouling in reverse osmosis and nanofiltration (Dr.-Ing. since November 2020)
M.Sc. Stephanie Kaschewski	Using smart water data to minimize risk in residential water quality
M.Sc. Oliver Kehl	Influence of aeration in modelling the treatment performance of activated sludge plants
M.Sc. Jinpeng Liu	Membrane filtration
M.Sc. Maximilian Miehle	Continuous bioproduction using a tailored biocatalyst for electrode assisted fermentation
M.Sc. Andreas Netsch	Energy-efficient bio-electrochemical waste water treatment
M.Sc. Giorgio Pratofiorito	Nanofiltration/reverse osmosis for organic acids concentration and biofouling detection
M.Sc. Florian Ranzinger	Visualization of water and biofilms in porous media
M.Sc. Prantik Samanta	Treatment of manure by membrane processes
M.Sc. Ali Sayegh	Membrane treatment of industrial waste water
M.Sc. Tim Schwarzenberger	Mono- and poly-chromatic UV disinfection
M.Sc. Lara Stelmaszyk	PCR- and cultivation-based methods for antibiotic resistance genes
M.Sc. Michael Sturm	Removal of microplastics from sea water
M.Sc. Stephan Zimmermann	Degradation of cytostatic drugs in water by oxidation processes

Guest Researchers

Dr. Samuel Bunani
Dr. Keke Xiao

Hybrid self-regenerating electromembrane process for removal of heavy metals from water
Interaction of organic matter with antibiotics and antibiotic resistance genes in sewage sludge

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Sylvia Heck

Staff meetings at the EBI during the COVID-19 pandemic.



RECENT PUBLICATIONS

Peer-Reviewed Journal Publications

Ali, R., Saravia, F., Hille-Reichel, A., Gescher, J., Horn, H., 2021. Propionic acid production from food waste in batch reactors: Effect of pH, types of inoculum, and thermal pre-treatment. *Bioresource Technology* 319, 124166.

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Publication Series of the Institute

Schriftenreihe des Bereichs Wasserchemie und Wassertechnologie und der DVGW-Forschungsstelle am EBI:

Volume 79: Bauer, A., 2020. Einsatz der optischen Kohärenztomographie zur kontinuierlichen Überwachung von Scaling bei der Membrandestillation.

Volume 80: Gierl, L., 2020. Untersuchungen zum Einfluss von Eisen auf die Struktur und mechanischen Eigenschaften von *Bacillus subtilis* in Biofilmen.

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