

DocDays VT 2023

Book of Abstracts



JULY 3rd, 2023
Graz University of Technology



DocDays VT 2023 Program.....	1
Oral Presentations	5
Session 1 – Energy, Sustainability and Paper Technology	5
Unlocking the Value of Sugar- and Coffee Side-Streams for Energy Applications.....	5
Recycling of Lithium-Ion Batteries: Sustainable Selective Leaching of Lithium of NMC Material by Using Water	7
Lifetime Investigation of PEM Water Electrolysis Cells	8
Water Dispersibility of Papers – Balancing Material Strength and Dispersibility.....	9
Session 2 – Powder Technology, Continuous Processes and Simulation	10
Filter Cake Dislodgement in Gas Cleaning	10
Design and Construction of Laboratory Scale Continuous Crystallizers Using Additive Manufacturing	12
Reformat Surrogate Mixtures in View of Liquid-Liquid Extraction Performance	13
Poster Presentations	14
Enhanced Hydrogen Production with Chemical Looping Through Doping Zirconium Oxide by Using Iron-Based Oxygen Carrier	14
Impact of Droplet Generation on Protein Stability During Drying: A Study Comparing Monodispersed Droplet Generator (MDG) with a Bi-Fluid Nozzle.....	16
Measuring Fluoride Emissions in Fuel Cell Effluent Water Using Photometry	18
Solid-State Stability of Advanced Lipid-Based Excipients upon Processing via Extrusion and 3D-Printing.....	19
Direct reduction of mineral iron carbonate ore with hydrogen combined with methane and methanol synthesis	20

DocDays VT 2023 Program

Monday 3rd July 2023

Time	Activity	Location
8:30 - 8:50	Registration	Lobby
8:50 - 9:00	<i>Welcome/Opening Session</i> Moderation: Prof. Ulrich Hirn	HS i9
	<i>Research Proposals – Session A: Pharmaceutical Engineering and Biotechnology</i> Chair: Prof. Stefan Radl	HS i9
9:00 – 9:20	Continuous Manufacturing of Highly Potent APIs <i>Daniel Moser</i>	
9:20 – 9:40	Euler-Lagrange and Hybrid Simulations in Stirred Tanks <i>Martin Fruhwirth</i>	
9:40 – 10:00	Liquid API Feeding in HME: Novel Options in Pharmaceutical Dosage Manufacturing <i>Lisa Kuchler</i>	
10:00 – 10:20	Carbon Dioxide as Feedstock in Microbial Cultivations: Gas Fermentation to Match Microbial Requirements and Technical Feasibilities <i>Halima Aliyu Alhafiz</i>	
	<i>Research Proposals – Session B: Bioenergy and Environmental Technology</i> Chair: Prof. Ulrich Hirn	HS i8
9:00 – 9:20	Identifying the Optimal Carbonization Degree of a Renewable Reducing Injectant Considering the Overall Blast Furnace Carbon Costs <i>Richard Deutsch</i>	
9:20 – 9:40	Impacts of Operating Conditions on the Lifetime of Polymer Electrolyte Fuel Cells <i>Joel Edjokola</i>	
9:40 – 10:00	Intelligent Glycerin Waste Recycling by Solid-Liquid Extraction <i>Max Vogl</i>	
10:00 – 10:20	Biobased Redoxflow Batteries <i>Dominik Wickenhauser</i>	

Time	Activity	Location
10:20 – 10:45	Coffee Break	Lobby
	Session 1: Energy, Sustainability and Paper Technology Chair: Prof. Marlene Kienberger	HS i9
10:45 – 11:05	Unlocking the Value of Sugar- and Coffee Side-Streams for Energy Applications <i>Julian Selinger</i>	
11:05 – 11:25	Recycling of Lithium-Ion Batteries: Sustainable Selective Leaching of Lithium of NMC Material by Using Water <i>Rafaela Greil</i>	
11:25 – 11:45	Lifetime Investigation of PEM Water Electrolysis Cells <i>Eveline Kuhnert</i>	
11:45 – 12:05	Water Dispersibility of Papers – Balancing Material Strength and Dispersibility <i>Andrea Pfennich</i>	
12:05 – 12:30	Poster session	HS i9 / Lobby
12:30 – 13:30	Lunch Break	
13:00 - 13:30	Q&A Administrative Aspects of the Doctoral Studies (Optional) Moderation: Prof. Heidi Gruber-Woelfler	HS i9
	Session 2: Powder Technology, Continuous Processes and Simulation Chair: Prof. Heidi Gruber-Woelfler	HS i8
13:30 – 13:50	Filter Cake Dislodgement in Gas Cleaning <i>Friedrich Holzinger</i>	
13:50 – 14:10	Design and Construction of Laboratory Scale Continuous Crystallizers Using Additive Manufacturing <i>Nico Nys</i>	
14:10 – 14:30	Reformate Surrogate Mixtures in View of Liquid-Liquid Extraction Performance <i>Maximilian Neubauer</i>	

Time	Activity	Location
14:30 - 15:00	Poster Session (+ best poster/presentation voting) / Coffee Break	HS i9 / Lobby
15:00 – 15:45	Keynote speaker: Dr. Theresa Hörmann-Kincses Moderation: Prof. Thomas Wallek	HS i9
15:45 - 16:15	<i>Farewell/ Closing Session</i> Moderation: Prof. Wolfgang Bauer Prof. Viktor Hacker Prof. Johannes Khinast Best Presentation Award Best Poster Award	HS i9
16:15 - 20:00	Social Event - Barbeque	

Poster Session

1. **Fabio Blaschke:** *Enhanced Hydrogen Production with Chemical Looping Through Doping Zirconium Oxide by Using Iron-Based Oxygen Carrier*
2. **Johanna Dieplinger:** *Impact of Droplet Generation on Protein Stability During Drying: A Study Comparing Monodispersed Droplet Generator (MDG) with a Bi-Fluid Nozzle*
3. **Matthias Heidinger:** *Measuring Fluoride Emissions in Fuel Cell Effluent Water Using Photometry*
4. **Moazz Abdelhamid:** *Solid-State Stability of Advanced Lipid-Based Excipients upon Processing via Extrusion and 3D-Printing*
5. **Sascha Kleiber :** *Direct reduction of mineral iron carbonate ore with hydrogen combined with methane and methanol synthesis*

Oral Presentations

Session 1 – Energy, Sustainability and Paper Technology

July 3, 2023 10:45 – 12:05

Lecture Hall: i9

Chairperson: Prof. Marlene Kienberger

10:45

Unlocking the Value of Sugar- and Coffee Side-Streams for Energy Applications

Presenting Author: Julian Selinger

Selinger J.^{1,2,*}, Stock S.³, Hobisch M.¹, Schlemmer W.¹, Egger M.¹, Meinander K.², Wilson B.⁴, Abbas Q.⁵, Hummel M.², Spirk S.¹

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The food processing industry is generating enormous amounts of (poly)saccharide- and extract rich waste throughout the year. However, these valuable biomasses lack comprehensive upcycling opportunities. Here, we investigate the potential of waste/by-products from the sugar- and coffee production with the aim of replacing fossil sources in electrochemical applications.

We have investigated the potential of spent coffee grounds, coffee silver skins, beet pulp and sugar molasses for the use in supercapacitors. All by-products, except molasses, were carbonized and activated with potassium hydroxide at elevated temperatures to produce activated carbon. Molasses was used as a substrate for bacteria derived nanocellulose separators. Redox-active caffeic acid was extracted from spent coffee grounds. The carbons were extensively characterized, showing surface areas up to 3000 m²/g with pore sizes down to the diameter of the later employed electrolytes. The carbons were assembled into free-standing electrodes. To increase the total capacitance of the supercapacitor, caffeic acid was mixed with the electrolyte prior testing. The supercapacitors were assembled, and their performance was tested by cyclic voltammetry and galvanostatic charge-discharge experiments using environmentally friendly electrolytes.

We could achieve capacitances up to 193 F/g with sulfuric acid as electrolyte. Thus, we could outperform the commercial reference with its 112 F/g by more than 50%. Further we showed that caffeic acid has a positive

impact on the performance. The tests of the bacterial cellulose separators showed that we almost reached the capacitance values of conventional glass separators. Overall, our study provides an important insight into the potential of industrial side streams as valuable resources for energy applications from renewable raw materials. This underlines the need for further innovation in this area to support the transition to a more sustainable and circular economy.

11:05**Recycling of Lithium-Ion Batteries: Sustainable Selective Leaching of Lithium of NMC Material by Using Water****Presenting Author: Rafaela Greil**Rafaela Greil^{1,*}, Joevy Chai^{1,2}, Georg Rudelstorfer¹ and Susanne Lux¹.¹ Institute of Chemical Engineering and Environmental Technology, Graz University of Technology, Graz/Austria² Chemical Engineering Department, Universiti Teknologi PETRONAS, 32610, Bandar Seri Iskandar, Perak, Malaysia*rafaela.greil@tugraz.at

Lithium-ion batteries (LIBs) have been a key factor for the breakthrough in electric mobility (e.g., cars) and portable electrical devices (e.g., cell phones and laptops). LIBs are one of the most important forms of energy storage and contain a lot of valuable substances. Lithium is one of the main elements for these batteries owing to its favorable properties (e.g., lightest metal, highest electrochemical potential and highest energy density of all metals). The metal can be extracted from salt lakes and ores, whereby the extraction of lithium from salt lakes requires large amounts of water and is time-consuming (18–24 months). The extraction of ores is an expensive and energy-intensive process. At the end of their period of use, only a fraction of batteries is recycled and a majority of LIBs end up in landfills. The continuously increasing demand of LIBs will lead to a forecasted lithium shortage of about 47 million tons by 2050, if lithium is not recycled¹. All of these factors lead to the urgent need of developing a recycling concept to recover lithium in high purity. More than 90 % of spent battery raw materials can be recycled by hydrometallurgical processes which have been intensively studied in recent years. However, a major challenge is the different composition of the cathode and anode material. The recycling of valuable metals from LIBs has the advantage that the materials can be directly reused. Within this work, we demonstrate a selective sustainable hydrometallurgical leaching process to recycle lithium using water as sustainable leaching agent. The selective leaching of lithium from black mass (shredded electrode material, NMC material) is done in batch-mode using pure water without any oxidizing agents. The influence of different process parameters (temperature, solid/liquid-ratio and mixing rate) is investigated. Up to 30 % of lithium can be leached from the black mass in a one-step process. Furthermore, a kinetic modelling is executed. The direct recycling of lithium carbonate is a promising concept and it can be used as raw material for new LIBs. No strong leaching (e.g., H₂SO₄) and oxidizing agents (e.g., H₂O₂) are used and the energy consumption is reduced because no pretreatment takes place.

Sources

[1] Mater. Adv., 2021, 2, 3234-3250

11:25

Lifetime Investigation of PEM Water Electrolysis Cells

Presenting Author: Eveline Kuhnert

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Polymer electrolyte membrane water electrolysis (PEMWE) is a promising technology for the production of green hydrogen with zero carbon emissions. The technology has several advantages, including high efficiency, fast response time, and low operating temperatures. Moreover, PEMWE can be coupled with renewable energy sources (RES), such as wind-, water- and solar power. Despite significant scientific progress in the past decades, there are still some challenges to overcome in scaling up the technology and reducing cost of materials. Therefore, the investigation of lifetime phenomena and monitoring of degradation is crucial to advance the technology, making it more efficient and cost-effective, and positioning it to compete in the market.

In this work, different accelerated stress test (AST) protocols have been designed to assess the performance and durability of individual electrolyzer components in the membrane electrode assembly (MEA) as well as the complete electrolyzer cell¹. Membrane failure due to H₂ crossover was addressed in a membrane-focused AST and operation dynamics from RES-PEMWE coupling were tested in an AST protocol that reflects real-life conditions from a 70kW photovoltaic (PV) plant based in Austria.

The ASTs have been tested in a 5 cm² PEMWE single cell by various characterization techniques, including in-situ and ex-situ cell tests. The PEMWE cell has been electrochemically analyzed by means of polarization behavior and impedance spectroscopy throughout a range of currents (0.01 A cm⁻² to 3.0 A cm⁻²) at different temperature setpoints (40 to 80°C). Based on this complementary investigation a novel set of ASTs for the benchmarking of baseline components is described, which can help the research community to quickly assess component durability and efficiently process novel materials².

1. Kuhnert, E., Heidinger, M., Sandu, D., Hacker, V. & Bodner, M. Analysis of PEM Water Electrolyzer Failure Due to Induced Hydrogen Crossover in Catalyst-Coated PFSA Membranes. *Membranes* **13**, 348 (2023).
2. Kuhnert, E., Hacker, V. & Bodner, M. A Review of Accelerated Stress Tests for Enhancing MEA Durability in PEM Water Electrolysis Cells. *International Journal of Energy Research* **2023**, 1–23 (2023).

11:45

Water Dispersibility of Papers – Balancing Material Strength and Dispersibility

Presenting Author: Andrea Christine Pfennich

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Water- dispersability of papers is useful in various applications as it disintegrates into its fibers after usage, and also is a fully biodegradable material. In this work, we have first introduced a test for the disintegration performance of different paper grades. Based on that disintegration and comprehensive statistical analysis we have performed a quantitative analysis on the technological and physical mechanisms responsible for a good paper dispersibility in water.

Regarding technological parameters for paper production, we identified lignin content, degree of refining and addition of starch as relevant factors reducing paper dispersibility. Addition of a debonding agent, a surfactant, was not found to be effective here.

In order to clarify the physical mechanisms governing paper disintegration behavior we analyzed paper properties related to mechanical strength and water uptake. We found a strong correlation between wet- and dry tensile strength of paper, both of which were highly affecting the dispersibility. Water uptake in the network, or water uptake into the fibers (WRV), or fiber wetting (contact angle) were not, or only very moderately, related to paper dispersibility. The only water absorption related paper property correlated to the disintegration results was liquid penetration speed measured with ultrasonic testing.

We are concluding that the same mechanisms that are creating dry strength – high density and strong fiber-fiber bonding – are also responsible for a bad disintegration behavior. Principal component analysis revealed that paper strength and water penetration speed are not governed by different latent variables but instead are all strongly associated with the first principal component. This suggests that the same mechanisms are responsible for reduction of water penetration speed and wet/dry strength. A future solution of the problem might thus be to decouple network strength and water penetration, e.g. by identifying suitable additives that impart bonding strength to the network without reducing the access of water to break the fiber-fiber bonds.

Session 2 – Powder Technology, Continuous Processes and Simulation

July 3, 2023 13:30 – 14:30

Lecture Hall i9

Chairperson: Prof. Heidi Gruber-Woelfler

13:30

Filter Cake Dislodgement in Gas Cleaning

Presenting Author: Friedrich Holzinger

Holzinger, F. ^{1,*}, D'Ercole, V. ¹, Schneeberger, M. ², R  ther, M. ², Krammer, K. ¹

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Regeneration of selected textile filter bags of a set in a bag house (for the reduction of particulate emissions in industrial exhaust gasses) liberates these from the formed cake of pollutants often by a high-pressure-short-period jet pulse of pressurized air [1]. The liberated filter cake disintegrates, but diffusive small cake fragments should not redeposit on nearby filter bags as soon as filtration reinitiates as this lowers their filtration efficiency [2].

Influencing the properties of the cake fragments such as size, shape, and velocity is useful for more fragments to settle out of the filtration zone e.g., by imprinting a pattern on the textile filter medium. However, parameter studies on the effect of operating parameters on the regeneration efficiency are ambiguous due to filter arrangement and different nozzle characteristics, and do not inform about the cake fragment's sizes and their course as they are based on static sample measurements [3-4].

Here we demonstrate the influence of pressurized-air pressure increase rate on size and velocity of the fragments as part of a parameter study. Among 5 experiments the number of fragments that liberate of the overall filter area ranged early after their detachment between 156 and 437. At a preset pressure increase rate at the nozzle outlet of 12.5 bars/s and a filtration velocity of 20 m/min prior to the jet pulse the mean projected area diameter of these fragments was 0.98 mm, they translated at 0.0309 m/s on average right before their detachment and at 0.0103 m/s immediately afterwards. At 12.5 bars/s and 2 m/min the mean fragment was 2.73 mm, translating at 0.0833 m/s before and at 0.0319 m/s after their detachment. At 5 bars/s and same filtration velocity the mean fragment was 2.64 mm, translating at 0.053 m/s before and at 0.0104 m/s after their detachment. Large fragments are obtained at the low filtration velocity of the normative standard and settle fast. At these conditions, only a low pressure increase rate leads to fragments that slowly propagate.

Based on that study one may prefer to lower the pressure increase rate to 5 bars/s instead of increasing the filtration velocity as more fragments settle out for two reasons: 1) due to their increased size and 2) their slower

initial speed. The investment in a pressure controller will pay off as the filtration cycle of a filter bag will not be lowered by redeposition of small fragments (nearby) and therefore the filters lifetime will increase.

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- [2] C. Kanaoka and M. Amornkitbamrung, "Effect of filter permeability on the release of captured dust from a rigid ceramic filter surface," Powder Technology, Bd. 118, 113-120, 2001.
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- [4] J. Binnig, A. Bredin, J. Meyer and G. Kasper, "Dimensional analysis of the cleaning pulse intensity in a surface filter test rig," Powder Technology, no. 211, pp. 275-279, 2011.

13:50

Design and Construction of Laboratory Scale Continuous Crystallizers Using Additive Manufacturing

Presenting Author: Nico Nys

Nys, N. ^{1,*}, König, M. ¹, Buchgraber, L. ¹, Neugebauer, P. ^{1,2} Jones, M. J. ³ and Gruber-Wölfler, H. ^{1,2}.

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In the production process of active pharmaceutical ingredients many unit operations are necessary to end up with a suitable product. Crystallization is one of those, and resembles the link between upstream and downstream processing. It is dependent on the solution composition after the chemical synthesis and defines the starting parameters for all downstream unit operations. Therefore proper control and specification of the crystalline product is of utmost importance.

Besides tendencies to implement continuous processes in pharmaceutical industry, most of the unit operations are still based on batch operations. This is due to the lower investment and research costs. Furthermore, the comparably small amounts of product needed are often too low, to justify the implementation of a continuous process.

Nonetheless, the development of continuous crystallization processes is targeted by the industry because of better process control and the prevention of batch-to-batch product variation.

In this work we show different approaches to target the development of a laboratory scale crystallizer for continuous applications in the production of active pharmaceutical ingredients. These are, on the one hand, based on current implemented concepts in large scale crystallization of inorganic salts and, on the other hand, show a novel concept for a fully functional crystallizer. The main instrument used for the realization of the prototypes was additive manufacturing. Short production time of novel customized geometries, modular design strategies and iterative improvement of the prototypes, by using the latest experimental results for design variations, allowed rapid process improvements. The results of the experiments clearly demonstrate that the crystallizers are fully functional as designed and facilitate the formation of particles with the desired properties. Even the mode of operation can be changed easily from currently cooling crystallization to other process types. Furthermore, it will be presented that the use of additive manufacturing not only enables flexible and tailor-made design of the crystallizer parts, but also provides the possibility to adapt the geometry quickly, depending upon experimental results.

14:10**Reformate Surrogate Mixtures in View of Liquid-Liquid Extraction Performance****Presenting Author: Maximilian Neubauer**Maximilian Neubauer^{1,*}, Thomas Wallek¹¹ Graz University of Technology; Institute of Chemical Engineering and Environmental Technology, Inffeldgasse 25 C

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If biofuels are utilized in combination with fossil hydrocarbon fuels, partially substituting the latter as outlined in the Renewable Energy Directive of the European Union, a promising process configuration is the extraction of the biofuel constituents directly into hydrocarbon fuels (e.g. gasoline) or blending stocks (e.g. catalytic reformat). Costly solvent recovery is not necessary and only the removal of limited amounts of water in the organic extract phase is needed in order to meet fuel specifications.

However, the reliable and accurate simulation of such processes is difficult due to the complexity of hydrocarbon fuels, which often consist of hundreds of components, making the application of rigorous thermodynamics difficult. Fuel surrogates are mixtures that mimic the properties of real fuels with only a small number of components, simplifying calculation and simulation procedures.

Here, we demonstrate the generation of fuel surrogates with respect to liquid-liquid extraction performance for the first time. Experimental tie line data from batch extraction experiments was incorporated into the calculation procedure, yielding surrogates which accurately depict the properties of the real fuel in regard to liquid-liquid extraction performance.

This allows for simple and fast process simulations via rigorous thermodynamics for processes involving hydrocarbon fuels as extraction agents, while at the same time achieving accuracy and reliability. Consequently, this contributes to a reduction of process development time and thus cost savings.

Poster Presentations

Enhanced Hydrogen Production with Chemical Looping Through Doping Zirconium Oxide by Using Iron-Based Oxygen Carrier

Presenter: Fabio Blaschke

Fabio Blaschke^{*1}, Marjan Bele² and Viktor Hacker¹

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Green hydrogen will contribute to the achievement of the "net zero emissions" target as an energy carrier and in the chemical industry. The chemical looping hydrogen (CLH) technology is a new innovative option for the production of green hydrogen from renewable resources in industrial scale [1]. However, pure iron-based oxygen carriers in CLH need the addition of support material, as without this addition the material is immediately deactivated due to the high degree of sintering of iron. Here we demonstrate novel support material which exceed the benchmark value of $\text{Fe}_2\text{O}_3/\text{Al}_2\text{O}_3$ 80/20 wt% in large-scale applications as reported in the literature [2]. The investigations show that not only the chemical inertness or the morphology is the bottleneck for a suitable upscaling, as assumed in the literature. In addition, microscopic phenomena play a key role in pellet stability and hydrogen production. Based on our HT-XRD and DSC characterization, a support material has also to stabilize the active compounds of $\text{Fe}_3\text{O}_4/\text{Fe}$ in the reactor during the cyclisation. The yttrium stabilized zirconia with 8 mol % Y_2O_3 (YSZ8) and YSZ10 oxygen carrier (OC) materials have converted, stored and produced hydrogen at over 10 mol H_2/kg OC on a 250 g scale for over 100 cycles in the fixed bed chemical looping system [3]. This experimental results show that matching of the crystal phases to the cubic structure of the active material is essential for the successful operation of a large-scale fix-bed reactor. The work reveals new fundamental principles for the design and provides insight into the influence of microscopic effects on the stability of oxygen carriers for chemical looping hydrogen systems.

Acknowledgements

Financial support by the Austrian Science Fund (FWF): P 34824 is gratefully acknowledged.

References

- [1] S. Nestl, G. Voitic, M. Lammer, B. Marius, J. Wagner, and V. Hacker, "The production of pure pressurised hydrogen by the reformer-steam iron process in a fixed bed reactor system," *J. Power Sources*, vol. 280, pp. 57–65, 2015, doi: 10.1016/j.jpowsour.2015.01.052.

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Impact of Droplet Generation on Protein Stability During Drying: A Study Comparing Monodispersed Droplet Generator (MDG) with a Bi-Fluid Nozzle

Presenter: Johanna Dieplinger

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Droplet generation is a very important step during the drying of any liquid formulations, as it has a great influence on the properties of the dried particles, like the size. However, classical gas-based atomization (bi-fluid nozzle) might not always fit all applications. For instance, it is challenging to produce large particles (> 50 µm) when using this droplet generation technique. Alternative technologies, like monodispersed droplet generators (MDGs), could be of interest when producing large flowable particles via solution drying ¹.

In this study we aimed to compare DROPPPO®, one of several different kinds of available MDGs that generate droplets using a piezo-electric transducer (PZT)², with a conventional bi-fluid nozzle to understand the impact of these nozzles on protein stability.

During lab-scale drying of 20%ww ProtX-formulations (aq.), it was observed that the drying capacity of the equipment used was not enough to dry the large DROPPPO® droplets. As we aimed to understand how different droplet generation technologies affect protein stability and particle characteristics, different powder analyses were done. Comparing the two nozzles showed, that ProtX is stable when dried, irrespective of the nozzle used. Both nozzle types resulted in a similar monomer content of $90.45 \pm 0.16\%$ to $90.64 \pm 0.06\%$. Furthermore, $5.24 \pm 0.07\%$ to $5.39 \pm 0.06\%$ of aggregates were detected with a moisture content of $13.23 \pm 0.72\%$ for DROPPPO® and $4.67 \pm 0.15\%$ for the bi-fluid nozzle. SEM analysis showed larger particles produced by the bi-fluid nozzle (\varnothing 10 – 60 µm) and smaller particles produced by DROPPPO® (\varnothing 500 nm – 30 µm), although larger particles were expected with DROPPPO®. We hypothesize that smaller particles result from satellite droplets generated during jet breakage, which are smaller in size after drying, and were probably the only particles able to be dried in our lab-scale equipment ³.

In this work, we showed that it is possible to produce stable protein powders using DROPPPO® with comparable performance to the bi-fluid nozzle in terms of stability. Therefore, flowable, monodispersed particles of larger size can be produced using solution drying. In the future, we aim to use equipment with higher drying capacities to dry the droplets generated by this nozzle adequately.

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Measuring Fluoride Emissions in Fuel Cell Effluent Water Using Photometry

Presenter: Matthias Heidinger

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Polymer electrolyte membrane fuel cells (PEFC) degrade according to several different mechanisms. Among them, chemical membrane degradation is a major concern. The chemical degradation of perfluorinated sulfonic acid (PFSA) based membranes can be detected through the emission of fluoride in effluent water. While it is possible to detect fluoride in water, existing methods have their drawbacks, be it sample volume, measurement time or cost.

In (Heidinger et al., 2023), we present a photometric method for detecting fluoride in water in a fast and reliable way that requires small sample amounts and produces results comparable to established methods like fluoride sensitive electrode (FSE).

After initial calibration, measurements are done in just 60 s per sample, which is a significant improvement, compared to the up to 24 min per sample for ion chromatography (Marocco et al., 2021). Sample amounts were also reduced to 900 µl per sample in comparison to around 5 to 15 ml for FSE measurements. Comparing the results to measurements with a FSE yields a good statistical correlation, determined via Bland-Altman, while the standard deviation of the method was consistently low.

Our photometric method presents an alternative way of measuring fluoride emissions that has the potential to be up scaled. Through this new method, we were able to achieve high precision for small sample quantities in low concentrations with a good statistical significance.

Heidinger, M., Kuhnert, E., Mayer, K., Sandu, D., Hacker, V., & Bodner, M. (2023). Photometric Method to Determine Membrane Degradation in Polymer Electrolyte Fuel Cells. *Energies* 2023, Vol. 16, Page 1957, 16(4), 1957. <https://doi.org/10.3390/EN16041957>

Marocco, P., Sundseth, K., Aarhaug, T., Lanzini, A., Santarelli, M., Barnett, A. O., & Thomassen, M. (2021). Online measurements of fluoride ions in proton exchange membrane water electrolysis through ion chromatography. *Journal of Power Sources*, 483, 229179. <https://doi.org/10.1016/J.JPOWSOUR.2020.229179>

Solid-State Stability of Advanced Lipid-Based Excipients upon Processing via Extrusion and 3D-Printing

Presenter: Moaaz Abdelhamid

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The use of lipid-based excipients (LBEs) in pharmaceutical formulations is well-established and remains advantageous due to characteristics such as being natural, biodegradable, non-toxic, and acting as solubility and permeability enhancer for improving the performance of poor soluble active pharmaceutical ingredients (APIs). Recent research efforts have been dedicated to examine the functionality of the available conventional LBEs with advanced pharmaceutical technologies such as extrusion and 3D-printing. However, many challenges like product instability and API polymorphism were recorded. This work demonstrates the use of polyglycerol esters of fatty acids (PGFA), a recently described as next-generation LBE, to manufacture stable products and to maintain the loaded API substance amorphous for longer time at its original polymorph. Upon composition tuning, the PGFA blends exhibited similar rheological behavior showing non-linear drop in complex viscosity upon heating at their melting temperature of around 50 °C. The X-ray analysis confirmed stable solid-state with stable α -form under all conditions. The filament produced via liquid feeding based-extrusion was less porous, hence more flexible than the filament produced via solid-lipid extrusion, yet, they both exhibited the same solid-state, showing that the lipid is stable under different heat stresses. There was no difference between the 3D-printed tablets produced from either filament. After aging for 3 months, the filaments and the tablets kept their stable lipid solid-state. The API was found in amorphous state after processing, and showed slow rate of recrystallization upon storage. With these results of PGFA performing extraordinarily under extrusion and 3D-printing, the list of pharmaceutical functioning excipients, especially lipid-based, is growing. Coupling of advanced technologies with tailored excipients can open the door for realizing applications that could not be achieved otherwise.

Direct reduction of mineral iron carbonate ore with hydrogen combined with methane and methanol synthesis

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CO₂ emission mitigation strategies necessitate holistic technology concepts, especially in high-emission sectors like the iron industry, which is responsible for a major part of industrial CO₂ emissions. A novel technology for direct reduction of mineral iron carbonate with hydrogen reduces CO₂ emissions by more than 60 %, compared to the conventional two-step pig iron production process of mineral iron carbonate; simply due to reaction stoichiometry. However, for a sustainable and CO₂ emission-lean iron production the consecutive valorization of the process gas, which mainly consists of unconverted hydrogen and released CO₂, but also of partially upgraded reduced carbon species, such as carbon monoxide, methane, and methanol, is necessary. Here we demonstrate the promising holistic technology concept of direct reduction of mineral iron carbonate with hydrogen and the valorization of the process gas by catalytic CO/CO₂ hydrogenation over Ni/MgO and Cu/MgO catalysts to methane and methanol, respectively.

During direct reduction of mineral iron carbonate, complete conversion of the iron-bearing components was possible with a degree of metallization of >80 % at a reduction temperature of 873 K. At elevated reaction temperature of 1023 K, the degree of metallization increased to 92 %. Precise adaption of the process parameters reduction temperature and feed gas composition allow for assembling a tailor-made process gas with specific CO₂:CO ratios. Further valorization of the process gas by catalytic CO₂ hydrogenation with bifunctional Ni/MgO and Cu/MgO catalysts showed high catalytic activity and selectivity for methane and methanol, respectively. The results offer a holistic technology concept for CO₂ emission-lean iron production and, based on green hydrogen, enables fossil-free production of methane and methanol.