



in cooperation with
Max Rubner-Institute
Institute of Safety and Quality of Cereal
and the
Institute for Food Technology
- Fermentation Technology- in Hohenheim

73rd Starch Convention
&
18th European
Bioethanol and Bioconversion
Technology Meeting

March 29th – 31st 2022
-online-

Tuesday, March 29th 2022

73rd Starch Convention

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14⁰⁰ 1.1 **Mervat Almuhammad**, Hohenheim (Germany)
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15⁰⁰ 3.1. **Simona Vackeova**, Brussels (Belgium)
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15³⁰ 3.2. **Nelli Elizarov**, Berlin (Germany)
Bioethanol in Germany under Europe's climate targets

16⁰⁰ 3.2. **Johan v. Groenestijn**, Wageningen (The Netherlands)
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Summaries

1. Starch based packagings

- 1.1. **Birgit Kießler**, Heidenau (Germany)
Starch-based hotmelt adhesives for paper and board packaging

The packaging market in Germany is a steadily growing market in which paper products play a central role. In many respects, these claim to be an ecologically sensible alternative to plastic packaging due to their renewable basis and the possibility of multiple recycling. They could underline this claim in the future if the adhesives that are usually indispensable in their production were also based on renewable raw materials. Since the development of thermoplastic starch, there have been efforts to use starch- and carbohydrate-based polymers for hotmelt adhesives. Such products are described in numerous publications and patents. However, they are not yet on the market.

Within the framework of a completed project "Glykopack", we were able to show that by varying thermoplastic starch and other biobased main components, it is possible to develop a hotmelt adhesive that is at least equal to synthetic hotmelt adhesives in almost all important product and processing parameters. The results will be presented in this lecture. In addition, the follow-up project "BioGlyk" will be presented, which mainly aims at solving the still open questions such as the thermostability of the hotmelt and efficient production routes.



***Dipl.-Ing. Birgit Kießler**, Project Manager Fibres & Composites, Papiertechnische Stiftung PTS, Heidenau, Birgit Kießler is a graduate engineer in process engineering and was able to gain experience in the fields of chemical analysis and the paper industry at various companies and research institutes. Birgit has been employed at the Papiertechnische Stiftung (PTS) in Heidenau since 2005. Her research focuses on the development of novel starch-based materials and the corresponding process technology*

2. Food / pharmaceutical applications

- 2.1. **D.C. Saxena**, Punjab (India)
Stabilization of Pickering emulsion using Octenyl succinic anhydride (OSA) modified Buckwheat starch with improved structure and morphology

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Abstract

Buckwheat esterified with OSA (0.5-3%) changed the morphology of starch with rough and irregular shape (SEM). Crystallinity decreased after modification (XRD). FTIR showed peak at 1750 cm^{-1} and NMR spectra at 0.80-0.89 and 1.2-2.7 ppm confirming OSA modification. Amphiphilic granules were utilized in stabilization of Pickering emulsion (ultrasonication with 30 and 40% amplitude for 2 and 4 min). The stable Pickering emulsion with droplet diameters was 3.43 to 86.86 μm to be formed with 3% OSA starch particle, treated with 40% amplitude for 4 min. The study confirms that buckwheat starch can be used for Pickering emulsion using ultrasonication.

Introduction

Fagopyrum esculentum (Common buckwheat) and *Fagopyrum tataricum* (Tartary buckwheat) are two mainly grown Buckwheat in India whose groats are principally used for human consumption as breakfast cereals, flour for making various bakery products like snacks, noodles and cookies. Total carbohydrates content present in buckwheat groats is about 67.8 -70.1%, from which starch contributes around 51-57% of total carbohydrates (Li & Zhang, 2001). The buckwheat starch granules have a polygonal or round shape having 2 to 12 μm diameter. Therefore, the smaller granules size can be utilized as particles-based stabilization of emulsion. However, the native starch shows poor functional properties that can be improved by physical, chemical, and enzymatic modification.

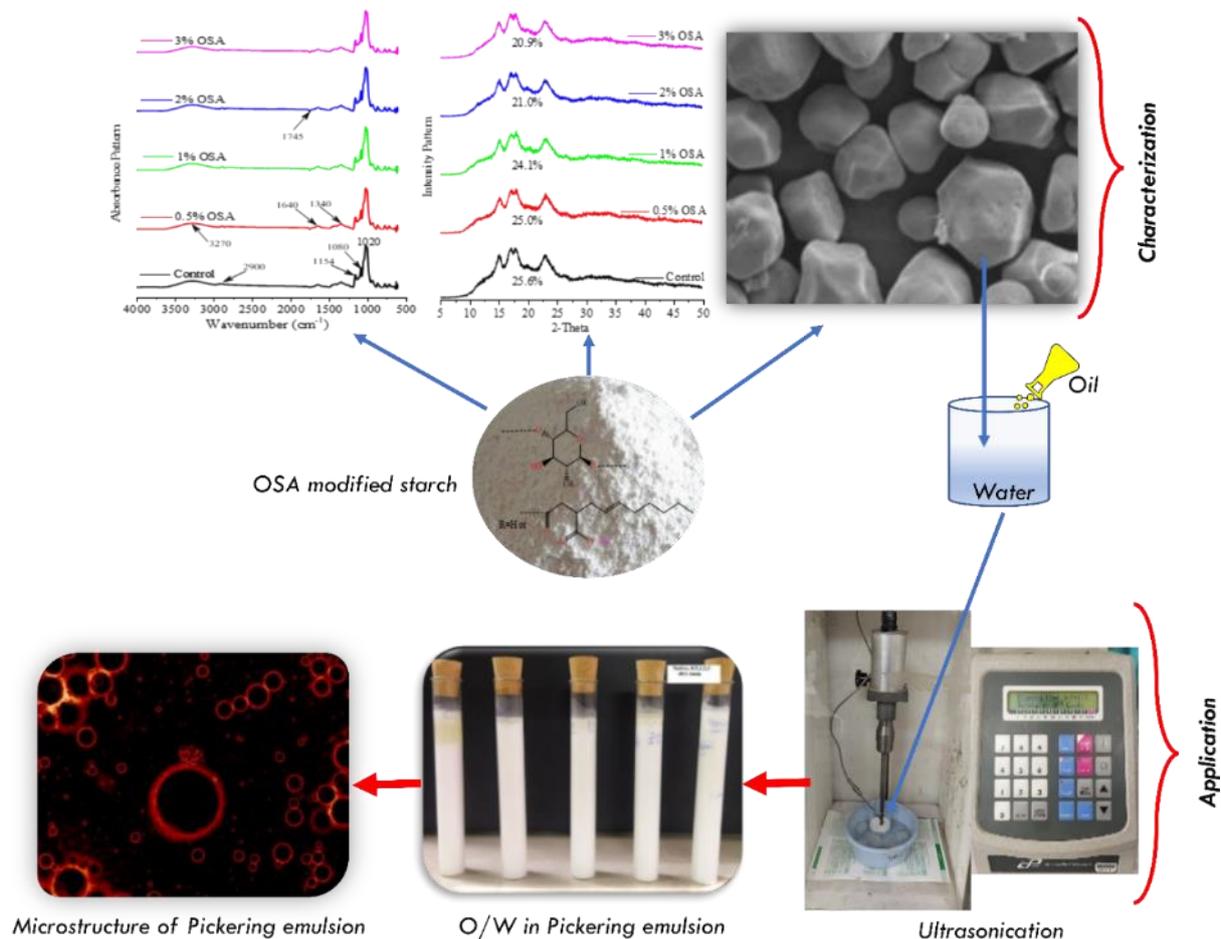
Chemical modification reaction like esterification with Octenyl Succinic Anhydride (OSA) (<3%) is used to produce highly functional groups of starch (Song, He, Ruan, & Chen, 2006). Native starches are generally hydrophilic, but hydrophobic sites from octenyl groups impart amphiphilic characteristics and interfacial properties to OSA Modified Starches (OSMS) (Sweedman, Tizzotti, Schäfer, & Gilbert, 2013). Different types of particles have been proposed to stabilize the emulsion; however, foods-based Pickering particles included protein, lipid, and carbohydrate particles (Marefati, Wiege, Haase, Matos, & Rayner, 2017). Different botanical sources of modified starch particles are used as a Pickering emulsion like sago starch (2.5% OSA) used by Saw, Shariffa, Ruri, & Uthumporn (2020), waxy maize, oats, potato, and quinoa (1.84 to 3.18% OSA) used by Saari, Heravifar, Rayner, Wahlgren, & Sjöo (2016), and rice, quinoa, and amaranth starch (0.6 to 3% OSA) by Marefati et al. (2017). However, due to limited studies on the implementation of ultrasonication in preparation of Pickering emulsion, the role of important ultrasonication parameters is still unclear. Moreover, synergetic effect OSA modified starch particles and ultrasonication amplitude and time is still not totally established. Therefore, the present work was designed to modify buckwheat starch by different OSA concentrations (0.5, 1, 2, and 3%) to study the structural and morphological properties of new solid particles. To test the ability of modified particles as an emulsifier, the particles were utilized to stabilize oil-in-water Pickering emulsion prepared by ultrasonication (30 and 40% amplitude for 2 and 4 min).

Materials and Methods

Materials

A commercial variety of buckwheat groats (average size: $4.06\pm 0.19\times 3.29\pm 0.15\times 2.87\pm 0.11\text{ mm}^3$) (Kuttu Giri) under the brand name SWaBHIman33 (FSSAI License no: 10819005001011) was purchased from Objectiviwitty Agrotech Pvt. Ltd. Gurugram, Haryana, India. The proximate composition of buckwheat groat was determined using the AOAC (2005) methods.

Methodology



Results

Characterization of OSA modified starch

The degree of substitution (DS) of OSA modified starch was significantly increased ($0.0032 \pm 0.001 - 0.0229 \pm 0.003$) with the OSA concentration. The amylose content of OSA modified starch (0.5-3%) decreases ($21.7 \pm 0.11 - 16.2 \pm 0.86\%$) because alkali-treated starch disrupts the amorphous region that contains the amylose chain (Karim et al., 2008). The DS of octenyl succinylation activity of OSA modified starch is negatively correlated with amylose, as higher DS decreased in amylose content.

The mean diameter of native starch was $7.44 \pm 0.03 \mu\text{m}$, which increased after OSA modification ($7.60 \pm 0.03 \mu\text{m}$, 3% OSA). The increase in diameter might be due to aggregation effects that cause the changes in the amphiphilic nature of OSA modified starch. These intact and smaller granular size is expected to create more surface area to react with OSA, which provide better emulsifying properties and more stable Pickering emulsion (Rayner et al., 2012).

The diffraction of native and OSA modified starches in XRD pattern showed a conventional 'Type A' pattern like cereal starches with strong peaks intensity at 2θ of 15.5, 17.3, 18.3, and 23.03°. In FTIR, a broad peak of around 3270 cm^{-1} indicated the presence of intermolecular hydroxyl groups (O-H). The peak around 2900 cm^{-1} shows weak intramolecular stretching vibration of O-H and the peak at 1640 cm^{-1} showed C=C bond stretching. The fingerprint region ($800 - 1200 \text{ cm}^{-1}$) of the starch spectrum showed five peaks that represent the C-O bond stretching. The OSA modified starch spectrum showed a peak around 1750 cm^{-1} which could be ascribed to the IR stretching vibration of C=O of ester carbonyl groups (Chen et al., 2014).

The morphological properties of native and OSA modified starch was evaluated with SEM with the magnification of 1000x and 2000x. Native starch showed an irregular and polygonal shape with relatively smooth surfaces and sharp edges. After OSA modification, the surface of granules became rough and irregular-shaped while developing some pores. The changes in surface features were due to the attack of OSA on granules surface to form small pores on them (Sharma et al., 2016). The other possible reason may be surface gelatinization during the addition of NaOH

during esterification reaction to maintain the alkaline condition that causes rough appearance to granules surface. OSA modified starch did not show any visible crack on the surfaces.

NMR spectra were used to confirm the OSA substitution in modified starch. The chemical shifts of all starches in NMR arise from the glucose unit of starch. These chemical signals of all starches were present at 3.20-3.33, 3.19, 3.60-3.75, and 3.90 ppm showed hydrogen positions at 2,3,4, and 5 for the internal hydrogen bond of the glucose unit (Simsek et al., 2015). The OSA modified starches showed a resonance spectrum at a peak around 0.80-0.89, which was associated with three protons of the terminal methyl group of OSA. The other specific peak of octenyl succinic anhydride were assigned at 1.27-2.7 ppm, associated with the methylene group, but not shown in native starch.

Pickering emulsion

The emulsion samples stabilized by native and modified starch were initially visually analyzed at room temperature for 10 days to observe the effect of OSA concentration on emulsion stability. The phase separation, creaming and sedimentation were observed during this period. The increase in the concentration of OSA from 0.5% to 3% of modified starch increased the creaming due to an increase in the attractive forces between starch particles and oil droplets. It was found that after day 1, native and 0.5% OSA modified starch emulsion showed foaming and phase separation because of partial coalescence and clustering between the oil droplets. On days 5 and 10, three different layers were observed in all emulsion samples, except for sample sonicated at 40% amplitude for 4 min. Three layers were observed: the opaque white layer at the top, a turbid layer at the middle, and a transparent or white layer at the bottom. The opaque white layer could be primarily of flocculated droplets that creamed rapidly. The turbid layer at the middle due to non-flocculated droplets creamed more slowly. Based on phase separation, creaming, and sedimentation, the best emulsion samples (3% OSA modified starch) were analyzed for further analysis like microscopic images, droplet size distribution and stability of the emulsion.

Stability of emulsion

The stability of emulsions using 3% OSA modified starch was visualized for 30 days at room temperature. It was found that 30% amplitude for 2 and 4 min showed increased creaming values (0.061 ± 0.002 - 0.534 ± 0.014 ml/ml) and (0.053 ± 0.001 - 0.417 ± 0.004 ml/ml), respectively, because of partial- coalescence and clustering between the emulsion droplets. Moreover, emulsions prepared using lower amplitude showed higher creaming because of low collision of molecules due to less intense collapse and lower velocity of shock waves. In contrast, emulsion prepared using 40% amplitude for 2 min produced low creaming (0.061 ± 0.002 - 0.144 ± 0.003 ml/ml) and high sedimentation (0.051 ± 0.012 - 0.092 ± 0.003 ml/ml) due to higher dispersed phase density and more rupture of emulsion structure at a higher amplitude. However, emulsion prepared using 40% amplitude for 4 min showed stable creaming value and minimal change was observed in the creaming value (0.376 ± 0.006 to 0.380 ± 0.019 ml/ml) after 5 to 30 days of storage. The possible reason might be higher amplitude, higher intense collapse, and the very high velocity of shock waves which facilitated complete mixing of the mixture, resulting in uniform emulsion formation and stable creaming (Anandan et al., 2017).

Conclusion

The buckwheat starch was esterified with OSA at different concentration level have a significant effect on the morphological and structural properties. The OSA modification decreased the relative crystallinity of OSA modified starch. The FTIR and NMR spectra confirmed the formation of ester linkages between OSA and starch. The intact and smaller granular size of native buckwheat starch showed a smooth surface and sharp edge, which were effectively utilized to stabilize the Pickering emulsion. 3% OSA modified starch granules showed better satiation due to improved amphiphilic behavior. In addition, ultrasonic amplitude and time also had a considerable effect on the stability of the emulsion, where lower (30%) amplitude for a short time (2 min) resulted in more sedimentation. In contrast, the higher amplitude and time significantly enhanced the stability by reducing the sedimentation and improving the creaming. Hence, the better syneresis effect of both OSA modification and ultrasonication produces stable oil-in-water Pickering emulsion stable

till observed for 30 days. The limitation of Pickering emulsion is the theories that can explain the interaction of the solid particles of Pickering emulsion are still low, and more still in demand for better understanding. Therefore, it can be concluded that oil in water Pickering emulsion stabilized by OSA modified buckwheat starch has great potential to improve the dissolution rate of poorly water-soluble bioactive by enhancing their bioavailability, which could be explored in future studies.

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2.2. Fred Warren, Norwich (United Kingdom) Structural heterogeneities in starch hydrogels

Hydrogels have a complex, heterogeneous structure and organisation, making them promising candidates for advanced structural and cosmetics applications. Starch is an attractive material for producing hydrogels due to its low cost and biocompatibility, but the structural dynamics of polymer chains within starch hydrogels are not well understood, limiting their development and utilisation. We employed a range of NMR methodologies (CPSP/MAS, HR-MAS, HPDEC and WPT-CP) to probe the molecular mobility and water dynamics within starch hydrogels featuring a wide range of physical properties. The insights from these methods were related to bulk rheological, thermal (DSC) and crystalline (PXRD) properties. We have reported for the first time the presence of highly dynamic starch chains, behaving as solvated moieties existing in the liquid component of hydrogel systems. We have correlated the chains' degree of structural mobility with macroscopic properties of the bulk systems, providing new insights into the structure-function relationships governing hydrogel assemblies.



Fred Warren graduated from King's College London with B.Sc. in Biochemistry (2007) and Ph.D. in Nutritional Biochemistry (2011). His Ph.D. focussed on the relationship between starch granule architecture and the binding kinetics of pancreatic α -amylase. Following his Ph.D., Dr Warren spent a year working at PerkinElmer (Seer Green, UK) as an Applications Scientist where he developed infrared spectroscopy methods for the application in the food industry. He then moved to The University of Queensland (Brisbane, Australia) as a University Research Fellow. Here he developed an interest in linking carbohydrate structure and gut microbiome function. He returned to the UK in 2015 to join the Institute for Food Research (now Quadram) as a Career Track Group Leader. He was also a Marie Curie Fellow from 2017-2019. He now leads a multi-disciplinary research team spanning plant science, chemistry and biology of carbohydrate polymers and the human digestive system at the Quadram Institute.

2.3. **Roman Bleha**, Prague (Czech Republic)

Non-traditional sources of health beneficial polysaccharides

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Microalgae biomass contains bioactive polysaccharides that make it very useful as food supplements (Sui, 2012; Suárez, 2005; Champenois, 2015). Moreover, for example *Chlorella* cell wall polysaccharides have proven antitumor effects (Sheng, 2007).

Fungi are interesting sources of secondary metabolites and structural biopolymers such as polysaccharides and proteins, etc. (Zhong et al., 2009; Novák et al., 2010). Most of these compounds have several biological bioactivities and thus have a potential use in pharmacy or medicine (Yuan et al., 2009). Cell wall fungi β -glucans belong to natural immunomodulating polysaccharides. They are predominantly linear or branched homopolymers of glucose having (1 \rightarrow 4) or (1 \rightarrow 3), (1 \rightarrow 6) linkages (Synytsya and Novák, 2013).

Fruits and vegetable are important part of human nutrition. They obtain compounds such as carbohydrates, antioxidants, fibres, vitamins, etc. (Knai, 2006). Pectins from mulberry fruits (*Morus alba* L.) (Lee, 2013) or *Z. jujuba* (Zhao, 2006) have biological activities such as anti-obesity, etc.

All these natural polysaccharides are thus prepare to be used as food supplements or in pharmacy and medicine.

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- laboratory courses teacher – application of FTIR and Raman spectroscopy on food products
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Education:

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2.4. **Shiva Elayadeth**, Minneapolis (United States of America)
 Soluble Rice Flour Development

Maltodextrin is a common ingredient that is present in many common food and beverage products ranging from powdered drink mixes to reduced sugar bakery items. However, consumers – ever wary of unfamiliar ingredients- are less keen on the starch derivative with the complex name. A challenge that food developers and manufactures currently face is that maltodextrins can be difficult to replace in food formulations due to their excellent viscosity, sensory, taste, and bulking agent functionality.

To address this challenge, Cargill has recently developed SimPure™ RF 92260, which may be declared on ingredient statements as Soluble Rice Flour. The ingredient is GRAS, non-GMO, and consumer research has confirmed that soluble rice flour is viewed positively by consumers, and also has a positive net purchase impact.

Compositionally soluble rice flour primarily contains carbohydrates, but also contains protein (7%), and lipid, dietary fiber in minor levels. From a functionality perspective, the soluble rice flour is highly soluble in water (85-90%), maintains similar viscosity to 10 DE maltodextrin in water when measured at 10% and 25% dry solids, and displays similar bulking agent functionality as maltodextrin. In addition, when soluble rice flour is incorporated into food applications like powdered chocolate milk beverages, soluble rice flour may offer an added advantage of conferring a creamier mouthfeel. Soluble rice flour has displayed excellent bulking agent functionality in several food application categories including salad dressings, powdered beverage formulations, instant sauce mixes, prepared sauces, and seasonings.



Shiva Elayadeth, Senior technical manager for texturizers and specialties business, Cargill

Shiva has been in the food R&D and Innovation space for over twenty years, within the US and globally. Today Shiva leads the company's development efforts in starches and other texturizers, working closely with customer partners and industry experts. Prior to joining Cargill, Shiva worked for General Mills Inc. in R&D across various product categories and brands. He has supported product development across multiple food categories in the US and international markets, including Latin America, Europe, and Australia. Shiva holds a bachelors degree in

Dairy Technology from the National Dairy Research Institute, India and a Master's degree in Food Science & Technology from the Ohio State University.

2.5. **Kathleen Hooyberghs**, Leuven (Belgium)
Impact of postharvest storage of potatoes on the molecular architecture and
viscosifying potential of their starch

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Although the industry requires potatoes for processing throughout the entire year, harvesting of potatoes mainly occurs in August and September. Consequently, postharvest storage of potatoes, preferably under (strictly) controlled conditions, is essential to ensure a continuous supply of potatoes and to avoid significant quality loss. Changes in potato starch characteristics during storage may impact how potatoes can be processed. Here, the characteristics of starch isolated from two potato cultivars, Challenger and Fontane, were mapped to gain insight into (i) the changes in starch structure during postharvest potato storage and (ii) how these changes impact starch functionality. The potatoes were cultivated and stored under the same conditions and characterized after harvest (September 2020), after 2 (November 2020), 4 (January 2021) and 6 months of storage (March 2021).

The composition of potato tissue was analyzed for 5 potatoes of each cultivar for each storage time. The starch and protein contents did not change during storage and were respectively 78% and 8% on dry matter (dm) for Challenger, and 85% and 8% on dm for Fontane potatoes. Furthermore, starch was isolated from 5 potatoes of each cultivar for each storage time. The molecular structure, gelatinization characteristics and pasting properties of the isolated starches were then determined with High Performance Size Exclusion Chromatography, Differential Scanning Calorimetry and Rapid Visco Analysis (5.0% dm; total sample size 25.0 g), respectively. The molecular size of starch decreased as a function of storage time. By way of example, for starches isolated from Fontane potatoes, the hydrodynamic radius corresponding to the peak maximum of the molecular size distribution decreased from 106 to 70 nm as a result of 4 months of postharvest storage. This was hypothesized to be due to β -amylase activity during cold storage. Besides, for both cultivars, the gelatinization temperatures and enthalpies seemed to decrease during storage. By way of example, the onset temperature of gelatinization decreased from 60 to 58 °C and the gelatinization enthalpy decreased from 21.5 to 19.5 J/g starch dm for starches isolated from Fontane potatoes. The changes in starch molecular architecture and thermal properties went hand in hand with increases in average peak viscosity values by 51 and 49% for starch isolated from Challenger and Fontane potatoes, respectively. In addition, the cold paste viscosities of the isolated starches increased as a function of potato storage time for both cultivars, while their setback viscosities remained unchanged.

We here conclude that postharvest storage of potatoes impacts the molecular structure and viscosifying ability of their starch. This may well affect the functionality of potato (starch) constituents during manufacturing of potato-based food products throughout the year. Therefore, this study could be of great interest to the potato-based food manufacturers.



Kathleen Hooyberghs obtained a Masters in Bio-Science Engineering at KU Leuven, Belgium in 2020. Afterwards she joined the Laboratory of Food Chemistry and Biochemistry as a Ph.D. candidate under supervision of Professor Jan A. Delcour. In her Ph.D. work, she aims at unravelling the impact of raw material properties, with a focus on starch, and processing parameters on the visco-elastic properties of mashed potato products.

2.6. **Gueba Agoda-Tandjawa**, Caen (France)

Starch-carrageenan interactions in aqueous media: role of each polysaccharide chemical and macromolecular characteristics

G. Agoda-Tandjawa*, C. Lascombes, P. Boulenguer, C. Le Garnec, M. Gilles, S. Mauduit, P. Barey, V. Langendorff

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Starch - carrageenan combinations have been used in processed dairy products since about 1950 for a wide range of texturizing and sensorial properties as described elsewhere (BeMiller, 2011). However, due to the increasing demand of dairy materials, it is becoming important to optimize the use of both ingredients through rational designing of innovative and cost-effective dairy desserts. Indeed, as the aforementioned features mainly depend on the physico-chemical interactions that could be established between both biopolymers and other components, many industrial professionals wish to better understand the nature and type of these interactions. In fact, although a great interest has been given to starch-carrageenan mixed systems in order to better understand the physico-chemical interactions between both polysaccharides, the type and nature of these interactions are still unclear. Additionally, the interaction mechanisms (“exclusion”, adsorption on and/or penetration in the granules) are still a matter of debate (Matignon, et al., 2014). Therefore, this study focuses on a better understanding the physico-chemical interactions between carrageenan and starch granules, taking into account the intrinsic characteristics of each ingredient. For that purpose, various proportions of κ -, ι - or λ - carrageenan and different types of starches (native, chemically modified, deproteinated or not) were used. Following some existing approaches on spectrophotometric methods using methylene blue properties of absorption (Garnier, et al., 2003; Michon, Konaté, Cuvelier, & Launay, 2002; Rohart, Jouan-Rimbaud Bouveresse, Rutledge, & Michon, 2015), we explored the possibility to quantify or characterize these interactions, in terms of the quantity of carrageenan trapped by starch granules. As starch granule swelling can strongly impact the evaluations of trapped carrageenan quantity, the exact swelling ratio of each starch sample was determined through another original spectrophotometric method using blue dextran as described elsewhere (BeMiller and Pratt, 1981; Tester and Morrison, 1989). Finally, rheological characterizations of starch-carrageenan mixed systems were performed to support the methylene blue spectrophotometry approach.

Through this study, it was demonstrated that the interactions between starch granules and carrageenan chains are not strongly impacted by starch chemical modifications (Lascombes, et al., 2017). However, experiments with “deproteinated” starch showed that starch endogenous proteins can play a part in starch-carrageenan interactions. Three coexisting phenomena were found to occur when starch is pasted together with carrageenan. That consisted in: (i) partial penetration of carrageenan in starch granules, (ii) partial “exclusion” of carrageenan by starch granules and (iii) a predominant adsorption of carrageenan on starch granules due mainly to osmotic pressure effect. Moreover, the electrostatic interactions between starch endogenous proteins and carrageenan chains played a minor role. As expected, the interaction level appeared to be strongly depending from the carrageenan charge density: the lower the charge, the higher the interaction between starch and carrageenan, independently from starch chemical characteristics. Furthermore, it seemed that the rheological properties of the mixed systems are preferentially driven by the partial “exclusion” phenomenon as well as the role of starch granules as “filler” (Lascombes, et al., 2017).

Keywords: Carrageenan; Starch; Interactions; Spectrophotometry; Rheology.

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*After receiving his Engineer's and Master's degrees in Chemical Engineering, **Dr. Guéba Agoda-Tandjawa** obtained a PhD in Physical-Chemistry of polymers from the University of Nantes (France) in 2009. He held three post-doctoral positions in french academic research (working on soft matter physics for 3 years) and an Associate Professor position in Physical-chemistry of polymers for 1 year (From Sorbonne Université, Paris), and then joined Cargill R&D job family in July 2013 as a senior scientist. His R&D activities within Cargill focus on better understanding structure-function relationships of hydrocolloids.*

2.7. **Florian Hess**, Greifswald (Germany)

3D printing - The path from powdered polymers like starch to medicines customised for each patient

Providing medication that can be customised for the individual needs of patients is one of the challenges that the pharmaceutical industry is currently facing. One possibility to enable the concept of personalised medicine is additive manufacturing.

3D printing is gaining high interest within nearly all industrial sectors. Also pharmaceutical industry is currently evaluating this promising technology concept. The approval of the first 3D printed drug by the FDA in 2015 really got the ball rolling and now many companies in the pharmaceutical and life science sectors are performing research activities in this field.

There are many different types of 3D printing technologies which differ fundamentally in their manufacturing process. In addition to the most widespread method, the filament-based FDM method (Fused Deposition Modeling), there are several other approaches such as stereolithography and laser sintering. They differ fundamentally in their process. The stereolithography process has been around the longest. The principle behind the process is the curing of liquid polymers with light. A printing plate is placed in a bath of liquid polymer, certain areas are exposed to a laser beam and the polymer hardens. The printing plate sinks further and further and the polymer can harden layer by layer. If you now want to add active pharmaceutical ingredients, these are either dissolved in the polymer or finely dispersed beforehand in a liquid state. The selective laser sintering process uses the same energy source, a laser. However, the laser beams are used here to melt or sinter heat-sensitive polymers in powder form. Here, too, the printing plate is lowered, a new layer of powder is applied over the first, already hardened layer and the procedure is repeated until the desired shape is reached. The most common method is Fused Deposition Modeling. Here a previously produced filament is melted again in a freely movable print head and pressed through a fine nozzle onto a printing plate. Possible active ingredients are directly incorporated into the filament. Due to the almost infinite structure possibilities, there is a wide field of application in pharmacy and medicine. From prostheses to implants to dosage forms individually adapted for patients, everything is possible with additive manufacturing. Especially the ease of dose adjustment makes 3D printing an interesting opportunity for improvement in geriatric and paediatric treatment.

A bottleneck that large parts of the pharmaceutical industry are currently facing is the development and approval of suitable excipients that meet the high pharmaceutical requirements. As part of her diploma thesis, Michelle Schneider, from Professor Weitschies' working group, investigated various polymers with regard to their behaviour in FDM 3D printing. Among other materials, she also investigated starch. The use of starch in melt extrusion is already well known, but as a carrier material in 3D printed tablets it is rather a novelty. Of course, the first tests with starch still show purely optical disadvantages compared to the polymers specially optimised for HME. But the tablets give reason to hope that with further optimisation of the process, starch is suitable as a carrier material for 3D printing. Its good release kinetic and biodegradability are particularly interesting aspects in the field of tablet production by 3D printing, especially at a time when biopolymers are becoming more and more popular.

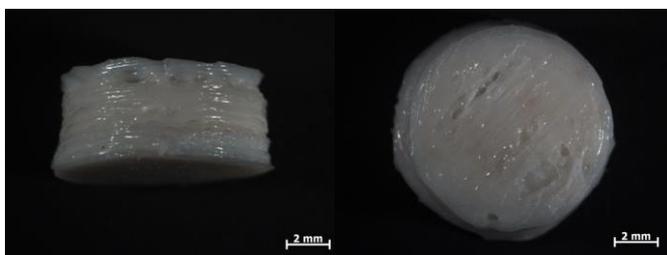
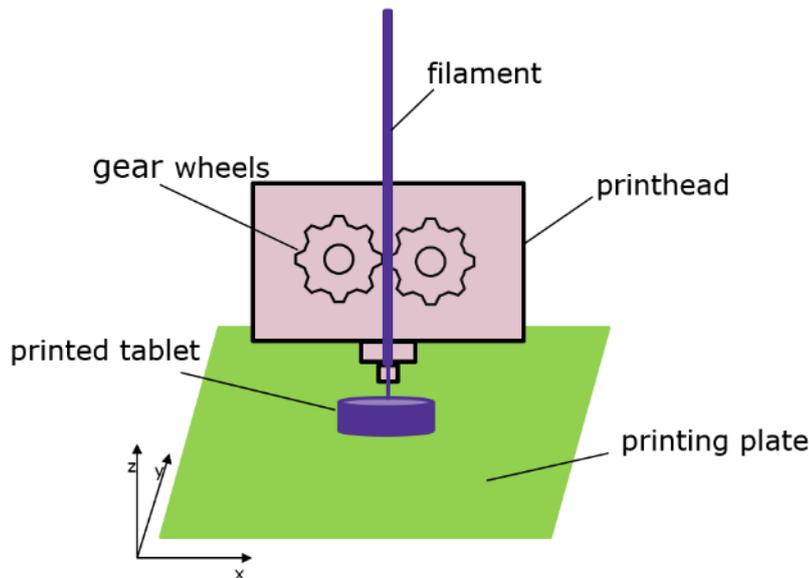


Image of the waxy maize starch printed tablet



Graphic: Schematic illustration of an FDM based 3D printer.



My name is **Florian Hess**, I am 26 years old. I studied pharmacy at the Technical University of Braunschweig. I graduated in December 2020 and received my "Approbation". For almost a year now, I have been working on my PHD in collaboration with Prof. Weitschies from the University of Greifswald and the company Merck KGaA. The topic of my work is "Additive manufacturing of oral pharmaceutical dosage forms using melt-based 3D printing processes".

2.8. **Tim Terstegen**, Berlin (Germany)

Organic pea starches – Comprehensive investigation of molecular, physicochemical and technofunctional properties

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The cultivation of peas increased significantly in recent years, mainly due to a higher demand of leguminous protein. Therefore, higher quantities of pea starch are also available. Pea starch shows a variety of interesting product properties such as gel strength or viscosity.

Here, a large variety of pea starches (smooth pea winter varieties, harvested in 2020 and 2021) were analyzed. The granular structure of the starch granules was studied using microscopy and image-based particle size determination. The thermal pasting properties were investigated using differential scanning calorimetry (DSC). The amylose content was determined using a modified wet chemical analysis based on the blue value determination using iodine. The molecular properties were analyzed using a size exclusion chromatography coupled with a multi angle laser light scattering detector (SEC-MALLS).

The pea starches showed very similar values regarding granular sizes ($24.0 \mu\text{m} \pm 1.2 \mu\text{m}$ [winter 2020] and $23.6 \mu\text{m} \pm 1.7 \mu\text{m}$ [winter 2021]) and calculated amylose content ($32.5 \% \pm 1.9 \%$ [winter 2020] and $32.4 \% \pm 1.9 \%$ [winter 2021]). Even though, large differences in other molecular properties (total molar mass M_w and molar mass of the amylose fraction M_{wAM}) were observed. The M_w ranged from $23.1 \cdot 10^6$ to $54.8 \cdot 10^6$ g/mol while M_{wAM} ranged from $0.53 \cdot 10^6$ to $2.23 \cdot 10^6$ g/mol.

The comparison of the thermal properties (onset-, peak-, conclusion temperature and enthalpy) with a principal component analysis showed that all pea starches could be distinguished from regular potato and maize starch, however, major differences between the pea starch varieties were also visible. Related observations were made during comparison of the technofunctional properties (gel strength and hot paste viscosity). As no remarkable difference was observed regarding the amylose content of the pea starch varieties, most probably the composition itself might impact the thermal and technofunctional properties and not the content.



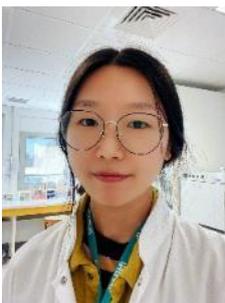
Tim Terstegen, Ph.D candidate at the department of Food Process Engineering at Technical University Berlin, is part of a project which is focusing on the optimization of organic produced pea starches for usage in different food products. He is a member of the Food Process Engineering Team since 2016 where he started as student assistant. Additionally, he wrote his bachelor thesis focusing on the molecular investigation of starch gel structures, and his master thesis about fat fractionation and analytical tools for process evaluation at the department.

3. Starch structure and functionality

3.1. **Yuzi Wang**, Paris (France)

Screening and investigating starch structure and enzymatic degradability in a wheat magic population

A major challenge faced when studying the “structure-degradability” interaction of native starch is deciphering the interdependency between different structural levels, especially when experimental conditions limit the number of samples. To tackle this challenge, 224 wheat starches from a 4-way multiparent advanced generation inter-cross population were screened for structural features and degradation profiles by porcine pancreatic α -amylase. A hierarchical clustering on principal components (HCPC) were used as multifactorial analysis to explore the data structure. In this presentation, the degradation procedure to screen a large collection of starches will be presented. Also, the HCPC results which highlighted the combined effects of granule size distribution, amylopectin chain length distribution, amylose content and endogenous α -amylase activity on degradation kinetics will be discussed.



Yuzi Wang, I'm a PhD student in food science at INRAE – Biopolymers Interaction Assemblies (BIA) in Nantes. I'm working on a joined project between INRAE and CSIRO in investigating starch “structure-degradation” relationship. I spent eight months working at CSIRO Agriculture and Food in Canberra during thesis.

4. Future developments

4.1. **Magdalena Zdanowicz**, Szczecin (Poland)

Starch treatment with deep eutectic solvents, ionic liquids and glycerol. A comparative study

In the studies changes in potato starch during thermal processing (dissolution as well plasticization via thermocompression and extrusion) in the presence of three types of media: deep eutectic solvents - DES (with different anions in choline salts, components types and their molar ratios), molecular ionic liquids (IL) and conventional plasticizer - glycerol (G) were investigated

and compared. DES are novel, widely developed media using in green chemistry, especially in biomass and biopolymer processing (i.e. dissolution, extraction, bioconversion, plasticization)¹. Functional properties of DES are highly depended on DES components. Urea-based DES and IL exhibited dissolving capacity towards starch (confirmed with microscopic observations and DSC), however for some of the media dissolution can be occurred with gelatinization. Plasticizing ability was studied using e.g. rheological characteristics of starch/media premixtures as well as mechanical tests, dynamical mechanical analysis (DMA), X-ray diffractometry (XRD) and IR-spectroscopic (FTIR) analysis of thermoplastic starch (TPS). Additionally, influence of concentration of two types of DES: dissolving and non-dissolving on final properties was investigated. Results revealed that viscosity of premixtures with DES and IL was lower that with G. Dissolving DES and IL led to highly amorphous materials with lower tensile strength and much higher elongation at break^{2,3} than TPS with non-dissolving DES and G. Moreover, DES are tailorable media lead to different properties and applications of final TPS materials dependently of the DES compositions.

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Magdalena Zdanowicz: My research areas focus on polymers (especially natural materials and their derivatives), composites, active packaging (plastics modification for e.g. antimicrobial activity) and deep eutectic solvents (DES). Currently, I serve as the manager of the project financed by the National Centre for Science and Development, Poland, related to materials aimed for agriculture.

4.2. **Bruna M. Miranda**, Goiás (Brasil)

A novel starch: Characterization of starch extracted from Jaboticaba (PLINIA CAULIFLORA) seeds

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Jaboticaba (*Plinia Cauliflora*) is a fruit from the southern center of Brazil, which has the shape of a spherical berry with a diameter of 2 to 4 cm, black peels and white pulp adhered to the seed. During Jaboticaba processing, the peels and seeds are generally discarded, and may represent up to 50% of the fresh fruit. A good alternative for using this residue is the recovery of its substances for technological purposes. The objective of this work is to extract and characterize starch from Jaboticaba seeds. An aqueous/acidic extraction was carried out and the starch obtained was characterized in relation to the content of proteins, phenolics compounds, light microscopy, scanning electron microscopy (SEM), determination of particle size distribution, diffraction analysis X-ray (XRD), thermal analysis (DSC) and molecular characterization using high-performance anion-exchange chromatography (HPAEC-PAD) and size exclusion chromatography (SEC). The extraction procedure presented a yield of $18.12 \pm 0.51\%$, and $1.19 \pm 0.11\%$ protein content and 0.58 ± 0.02 (mg gallic acid/ g starch) of phenolic compounds were found in the starch. The microscopy showed small granules with smooth and irregular shape. The particle size was between $6.1 \mu\text{m}$ to $9.6 \mu\text{m}$ and the X-ray diffractogram indicates that this C_c-type of starch. Analyzing the endothermal curve, it was obtained onset temperature (T_o) at 66.4 ± 0.46 °C, a peak temperature (T_p) at 73.5 ± 0.5 °C and a conclusion temperature (T_c) at 80.5 ± 0.76 °C. The molecular characterization by HPAEC-PAD showed intermediary chain length, similar to that of the crystalline C-type, and characterization by means of SEC-techniques proved

a comparatively low M_w ($5.3 \cdot 10^6 \text{ g} \cdot \text{mol}^{-1}$) of the extracted starch. The Jaboticaba starch proved to be a promising material for future studies and applications.

Keywords: Jaboticaba seed starch, granular properties, granule size, molecular characterization



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4.3. **Christina Gabriel-Liebs**, Potsdam (Germany)

New applications for starch esters – Film former in metal coatings

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In all industries, concepts of sustainability and environmental friendliness playing an increasing role, just as in the coating industry. Nevertheless, at most 10% of the coatings are semi bio-based and less than 3% are completely bio-based. Reasons are chiefly the hydrophilicity, but also high prices and unsecured availability. Thus, the demand for new bio-based solutions is high.

With starch as a principally good film-forming substance, there is a cheap and worldwide available raw material. Nevertheless, starch is also strongly hydrophilic. This would lead to inclusion of water and finally delamination of the coating film from the substrate. Additionally, native starch shows high viscosities at low solids content, a high glass transition temperature, forms brittle films, and has a low mechanical strength as film. Consequently, the target was the modification of starch to overcome the aforementioned disadvantages. This included solids content of at least 30% at room temperature with suitable viscosities ($0.1\text{-}1 \text{ Pa} \cdot \text{s}$ at 100 s^{-1}) and Newtonian or shear thinning flow behavior, as well as sound results in test methods which show an improved water resistance. For the coating application, a high amylose starch was chosen. First, the starch was desintegrated and afterwards degraded by α -amylase ($0.2\text{-}1.0 \text{ U/g}$) for reaching higher solids content in the later binder dispersion. The weight average molar masses M_w of the products were determined by GPC-MALS and were $0.2\text{-}0.7 \cdot 10^6 \text{ g/mol}$. The degraded starches were further modified by transesterification with vinyl esters ($C_6\text{-}C_{12}$, 2 mol/mol AGU). The obtained degrees of substitution (DS) were determined by elemental analysis and were $1.47\text{-}1.90$. For the preparation of starch ester dispersions, the bio-based solvent Methyl-Tetrahydrofuran (Me-THF) was suitable for all samples. In the next step, the maximal suitable solids content of the different samples (varying M_w and DS) were identified starting with 30% solids content by investigation of the rheological properties mainly the viscosity. As long as the viscosity was $\leq 1 \text{ Pa} \cdot \text{s}$ and the dispersion visually clear the solids content was increased. The final dispersions had solids content of 30-40% and viscosities of $0.1\text{-}1.8 \text{ Pa} \cdot \text{s}$ with Newtonian or shear-thinning flow behavior. The film forming and adhesion properties were tested on aluminum substrates. For this purpose, the dispersions were applied with a doctor blade (gap: $400 \mu\text{m}$) and a cross cut test were performed. All samples (dry film thickness $\approx 30 \mu\text{m}$) formed transparent coating films and obtained the best classification (class: 0) in the cross cut test which means excellent adhesion properties on the substrate. The only exception were one laurate sample that had the classification 1. The Martens hardness testing showed that the hardness can be adjusted by the substituents as it is also known from the glass transition temperature^[1]. Using the example of the octanoate and laurate samples, it means that the laurate samples were softer and more elastic than the octanoate samples in the Martens hardness testing. As hydrophilicity and associated swelling of natural film formers is one of the biggest challenge in coating applications, as mentioned above, the so called "water uptake

reversibility (WUR)” was investigated. For this, the coated substrate was exposed to 0.05 KNO₃ solution and short thermocycles (15-45 °C in 30 min, 4 h). Continuously the capacitance was detected by using high frequency (HF) electrochemical impedance spectroscopy (EIS) data. The capacitance is a (relative) measure for the water uptake (reversibility) of a matrix (e.g. coating system). The results showed the correlation that the softest and most elastic starch ester coatings (laurates) have the lowest thermocyclically induced loss of WUR. Low frequency EIS measurements (8 h) gave additionally information about barrier properties. These results showed the same correlation as the HF-EIS tests.

The research results show that with starch by combination of different degrees of degradation, substitution and ester substituents coating relevant solids content and viscosities, as well as hardness can be reached and systematic adjusted. Furthermore, the modification lead to excellent adhesion on aluminum substrates and good results in WUR and barrier tests. In conclusion, the synthesized starch esters show the potential for film formers in metal coatings.

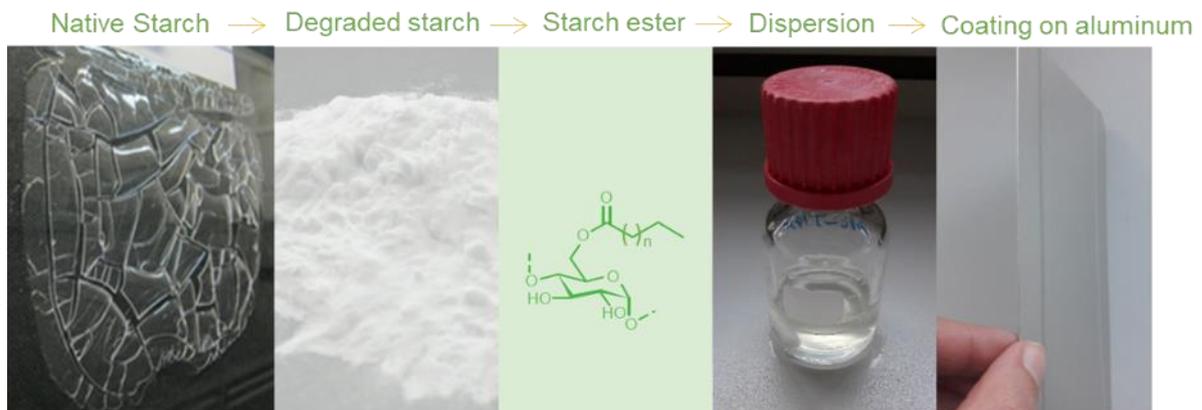


Figure 1: Path of development – from native starch to starch ester coatings on aluminum.

[1] Winkler, H.; Vorwerg, W.; Rihm, R.; *Carbohydrate Polymers*, **2014**, 102, 941-949.



Dr. Christina Gabriel-Liebs was born in 1988 in Kassel (Germany) and studied chemistry at the University of Ulm. She completed her master and doctoral thesis at the Fraunhofer Institute for Applied Polymer Research IAP in Potsdam, where she subsequently remained as a scientist. During the last 9 years, she has been working at the IAP on the development of modified starches for various technical applications. Her focus was and still is on coating and adhesive applications, but also on new green synthesis routes.

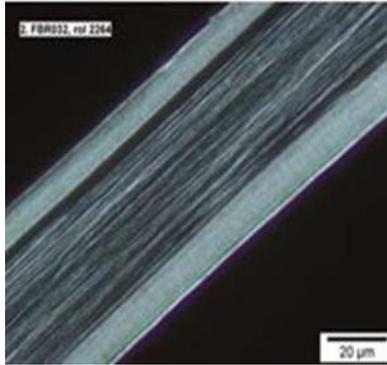
5. Technology

5.1. Gerald Schennink, Wageningen (The Netherlands)

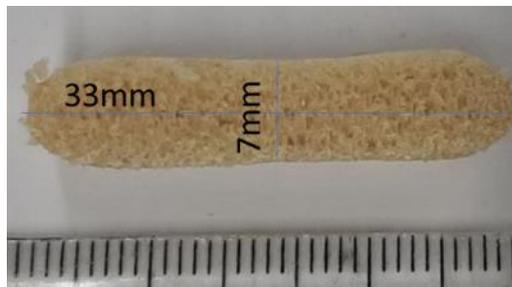
Latest achievements in thermoplastic starch processing: films and foams

Starch is a renewable, cheap raw material that can be obtained from a large number of plant sources (Potatoes, Corn, Wheat, Tapioca). Dry starch has a melting point far above its thermal degradation temperature. To make starch thermoplastically processable a plasticiser system is essential. Thermoplastic starch combines a low ecological footprint (especially when starches from (food/feed) side streams are used), with versatile material properties and biodegradability. Thermoplastic starch can be processed into products via several techniques including injection moulding, film blowing and foaming. In this lecture latest results of two projects will be highlighted:

- Development of a thermoplastic starch based multilayer film, based on side stream starches. With help of this film the Dynamic modified atmosphere packaging concept was demonstrated. Elongation of shelf life of fresh horticultural products and improvement of quality of these packed products could be proven.



- Development of an industrial feasible technology for the production of Thick walled, starch based, foamed packaging products with help of extrusion processes. Loose fill foams based on starch are commercially available for several decades already. However the production of foamed profiles with an exact defined shape and dimension is a bigger challenge. With advanced CFD techniques we tried to simulate the effect of die and composition modifications on the final shape of these type of products.



Gerald Schennink was born in The Netherlands in 1967. After his study at the University of Twente and the Eindhoven University of Technology (graduation work was performed on the field of Polymer Processing), he worked for some years within the Philips company on the field of injection moulding. In 1995 he accepted a position of project leader within the business unit Biobased Products of WFBR in Wageningen (NL). Key issue within these projects is development of biodegradable materials/polymers and/or product development with help of this type of materials. Main raw materials he worked with are biobased materials like starch, cellulosics, poly-lactic-acid and poly-hydroxy-alkanoates. Gerald has a lot of experience within a wide range of polymer processing methods, like injection moulding, sheet extrusion, film blowing, blow moulding and fibre spinning. Gerald is (co-)inventor of several patents.

5.2. **David Appelhaus**, Braunschweig (Germany)

Wetting situations and heat transfer performance of wiped film evaporators at different scales

*Stefan Jahnke, David Appelhaus, Katharina Jasch, Stephan Scholl
TU Braunschweig, Institut für Chemische und Thermische Verfahrenstechnik*

Wiped film evaporators enable the processing of temperature-sensitive, viscous and foaming-prone products. Due to the low hold-up and low operating pressures, comparatively short residence times and product-friendly boiling conditions are achieved. A rotating wiper system ensures uniform distribution of the feed on the inside of the evaporator surface and increases product-side heat transfer. To ensure an optimum operating point, knowledge of the wetting behavior and evaporator performance is required. For a subcooled feed, three zones of heat transfer can be distinguished. In the first zone, single-phase heat transfer takes place until the product boiling temperature is reached. This part of the surface is not available for evaporation. In the subsequent height segment, evaporation takes place. As long as the local circumferential

load of the liquid is higher than the minimum necessary circumferential load, evaporation occurs from a closed liquid film and the evaporator surface is completely wetted. If the circumferential load falls below this limit, the surface eventually locally and intermittently runs dry. This leads, among other things, to overheated spots and underutilization of the possible evaporation capacity. This presentation is about the qualitative and quantitative determination of the characteristic zones based on experimental data collected on a steam-operated stainless steel wiped film evaporator with block wipers. The heat transfer coefficients are calculated for each region along the evaporator and the wetting zones are determined. A key finding is that the minimum bottom perimeter load is not constant at increased heat fluxes, but increases with heat flux. This behavior may indicate earlier film breakup and possibly bubble formation on the inner evaporator surface. The effect of wiper type and speed on apparatus performance will also be evaluated, providing a more comprehensive view of wiped film evaporator operating behavior. The segmental approach is also applied to a steam-heated industrial apparatus with an evaporator surface of 2 m². For comparison, regular wetting and starting dewetting of the evaporator surface are shown for glass evaporator by high-speed recordings and will be discussed.



David Appelhaus studied chemical engineering at the TU Braunschweig from 2014 to 2020. Since April 2020, he has been working as a research assistant at the Institute of Chemical and Thermal Process Engineering in Braunschweig. Among other things, he is working there on the modeling and characterization of wiped film evaporators. His focus here is on the residence time behavior and heat transfer under evaporation conditions.

18th European Bioethanol and Bioconversion Technology Meeting

1. Utilization of side streams

1.1 Mervat Almuhammad, Hohenheim (Germany)

Leftover bakery goods for bioethanol production - potentials for medium-sized companies?

According to WWF Germany [1] (2018), every third food item is thrown away in Germany alone, which causes over 18 million tons of food waste every year. Bread is particularly affected as one of the most popular foods. It is one of the most thrown away foods in the world. Of the 4.5 million tons of baked goods in Germany, 1.7 million tons were disposed of in 2015, which corresponds to more than a third. Particular attention should be paid to the use of resources. Each piece of baked goods requires land and water for cultivation and production, as well as energy. This creates a significant environmental impact from baked goods that are later not used and disposed of. At first glance, an environmentally friendly alternative is the processing of old baked goods in so-called biogas plants. However, this incurs costs for the bakery, as well as an additional environmental impact due to the transport of the pastries to the exhaust system. It is therefore more practical and cost-effective for many companies to dispose of the leftover baked goods. In order to counteract this, an environmentally friendly, alternative use of the old baked goods is sought, in addition to other aspects such as the optimization of sales and production, as well as the return rate. A way of using it is to produce bioethanol by fermenting old baked goods. Bioethanol production from different old baked goods was examined in this research work, in order to investigate the potential of bioethanol production from each group and try to increase the glucose production using enzymatic hydrolysis and thus the yield of bioethanol. The old baked goods used in the experiment were provided by the "Webers Backstube" bakery in Germany. The baked products were sorted into 9 different groups; these were differentiated according to the type of dough and type of pastry (Brötchen/ Bread rolls, Laugengebäck/ Lye rolls, Plunder/ Plunder pastry, Mischbrot/ fine Rye Bread, Weißbrot/ White Bread, Spezial-Brot/ special bread, Zopf/ Butterzopf, gebackene Kuchen/ Cake and Sahne-cremetorten/ cream cake). The Liquefaction was conducted using EnerZyme® AMYL (90°C for 30 min) and the Saccharification with EnerZyme® HT (65°C, 30 min). The samples were fermented for 5 days by Ethanol Red at 30°C. The amount of the Glucose, Fructose, Ethanol, Lactic Acid, Acetic Acid and Glycerol was determined using HPLC, before and after Fermentation. The highest Ethanol yield in the fermented samples was investigated in Mischbrot/ fine Rye Bread 88.53 g/L, with conversion efficiency 82%. While the White bread has the lowest Ethanol yield 38 g/L with conversion efficiency 29,30%.

[1]: Ü. Brotkanten Und Wachsenden Brotbergen, unser tägliches Brot, n.d.



Mervat Almuhammad, in spring 2019 I successfully completed my master's degree in "Environmental Protection and Agricultural Food Production" at the University of Hohenheim. In September 2021 I started my PhD at The Institute of Food Science and Biotechnology- Department of Yeast Genetics and Fermentation Technology- University of Hohenheim and I am focusing in my research on Bioethanol production from Leftover bakery goods.

2. Non starch based feedstocks

- 2.1. **Luis Hoppert**, Hohenheim (Germany)
Overcoming factors limiting high-solids bioethanol production from lignocellulosic biomass

Economically feasible bioethanol production from lignocellulosic biomass requires solid loadings ≥ 30 % dry matter (DM). However, increased solid loadings can lead to process difficulties, which result in reduced ethanol yields. Enzymatic hydrolysis is mostly limited by physical factors, such as high apparent slurry viscosity, limited water availability and insufficient substrate mixing. The subsequent fermentation process is limited by biological factors like osmotic stress and high concentrations of lignocellulosic inhibitors. During my presentation I will go into detail on the mechanism behind these inhibitory effects and potential ways to overcome them. A main focus of my presentation will be on synergistic effects of lignocellulosic inhibitors and osmotic stress during high gravity fermentation of different lignocellulosic feedstock's.

Luis Hoppert, I have graduated at the University of Emden/Leer in Applied Life Sciences (M. Eng) in 2019. As part of my master's thesis I worked on the development of a new process for the fermentative production of medium-chain carboxylic acids at the German Biomass Research Centre (DBFZ). At the moment I'm in the last year of my PHD thesis at the University of Hohenheim, researching second-generation bioethanol production. Currently, my work is focused on overcoming inhibitory effects of lignocellulosic inhibitors during high gravity fermentation.

3. Future developments

- 3.1. **Simona Vackeova**, Brussels (Belgium)
How the future of the ethanol industry depends on various external developments (market, economy, policy)

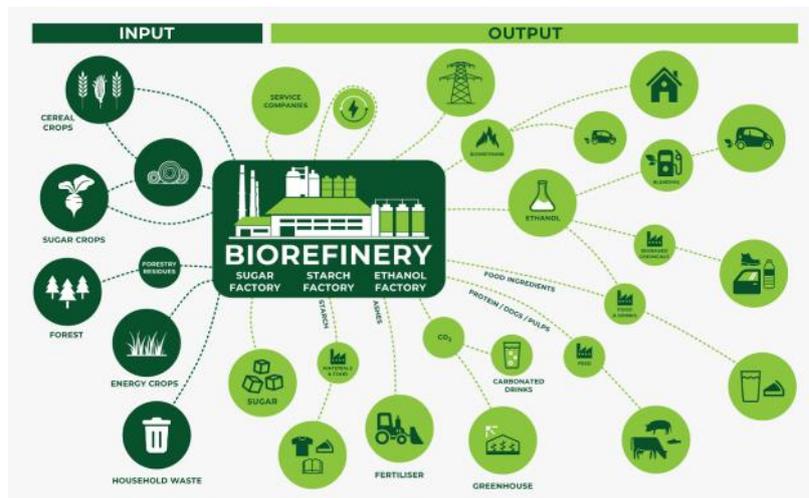
As a proven, domestically sourced technology for reducing emissions from road transport – renewable ethanol from ePURE members achieves more than 75% average emissions saving compared to fossil petrol and contributes to decarbonisation of road transport.

EU renewable ethanol can do even more in the drive to offset fossil fuel and reduce the need for imports of animal feed. The old 'food vs. fuel' and sustainability myths about EU ethanol have all been busted and it's clear that renewable ethanol is among the sustainable biofuels with the lowest carbon abatement costs that should be part of the EU Green Deal and 'Fit for 55' landscape.

The European Commission's 'Fit for 55' package sets important new goals for emissions reduction in transport. Giving a prominent role to renewable liquid fuels would create a solid foundation for reaching them. To that end, the EU should fully maximise the tools it has on hand for decarbonisation and de-fossilisation – starting with the Renewable Energy Directive while ensuring there is no discrimination among sustainable biofuels across the proposed policies incl. Energy Taxation Directive or FuelEU Maritime and ReFuelEU Aviation proposals.

With the 'Fit for 55' package, the EU promised big changes to its energy and climate laws in order to meet more ambitious decarbonisation targets for 2030 and beyond. But a lot depends on the coming months, as the European Parliament and EU Member States scrutinise the planned changes across the proposals which do not always give enough of a role to a proven decarbonisation solution: renewable liquid fuels such as European renewable ethanol.

But EU renewable ethanol production is about more than just renewable low-carbon fuel that helps Europe achieve its climate goals by displacing fossil petrol. As the EU aims to increase its energy independence and steps up its ambitions to fight climate change, it should not overlook the full potential of European renewable ethanol and the entire economic system, especially in the current political context.



Source: ePURE - <https://www.epure.org/about-ethanol/how-is-ethanol-produced/>

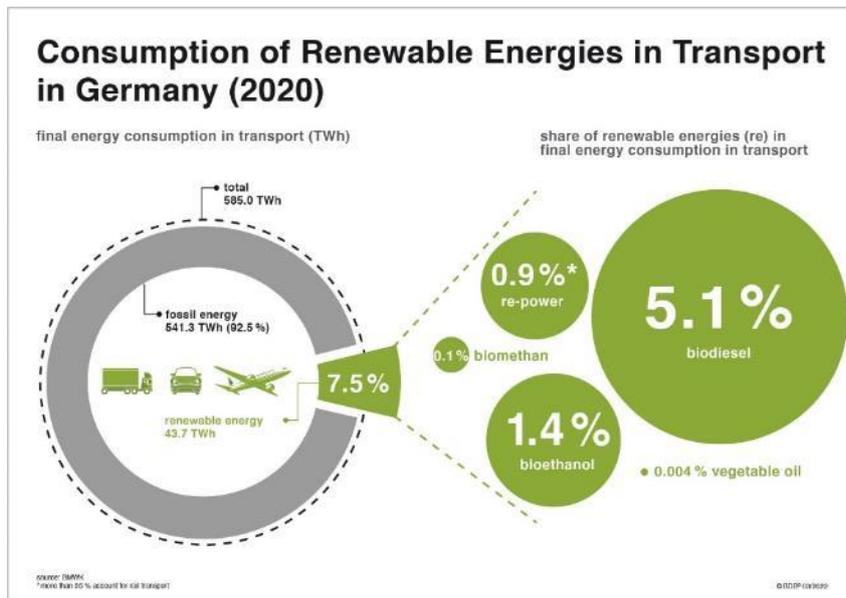


Simona Vackeová - Director of Government Affairs, ePURE
 Simona joined ePURE in October 2020 and is responsible for energy, environment and trade issues. She spent nearly a decade working on horizontal trade issues affecting the European ceramic industry. Simona holds a Master's degree in Politics & International Relations from the University of Aberdeen and in EU International Relations and Diplomacy Studies from the College of Europe.

3.2. **Nelli Elizarov**, Berlin (Germany) Bioethanol in Germany under Europe's climate targets

The European Commission recently presented revisions and initiatives under the Fit For 55 package. With the comprehensive legislative package, the climate and energy targets for 2030 will be completely revised. Following the vision of the Green Deal to make Europe the first climate neutral continent, the emission reduction target from current 40 % was increased to 55% compared to 1990 levels. An important element of the package is the revised Renewable Energy Directive setting targets and rules for the industry, cooling and heating sector as well as transport. For achieving the goals set for the transport sector and at the same time reducing air pollution, sustainable biofuels offer a high and already available potential.

One fifth of Germany's greenhouse gas emissions are caused by the transport sector. Due to increasing mobility, the transport sector has not seen a decline in emissions as other sectors. The reasons are the growing vehicle stock of passenger and freight transport as well as the tendency to purchase vehicles with more powerful engines. At the same time, current data suggest that the transport sector will continue to rely on liquid fuels. The majority of the Germany vehicle fleet consists of vehicles with combustion engines and is getting older every year (passenger cars: Ø 9.8 years). Therefore, vehicle emissions remain the main cause of air pollution in cities.



In the European Union and also in Germany the use of biofuels has the strongest impact on the reduction of greenhouse gas emissions in the transport sector. Another substantial benefit is that biofuels significantly reduce some exhaust emissions that are harmful to the environment and human health, e.g. particulate matter and polycyclic aromatic compounds.

Nevertheless, the use of biofuels is an often controversially discussed mean of achieving climate targets in transport sector. So what are the advantages of biofuels and which aspects can be critical? What are the prospects for biofuels in Germany under the recently passed legislative packages?

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Nelli Elizarov, Dr., German Bioethanol Industry Association, elizarov@bdbe.de, +49 (0)30 301 29 53 15, Since 2017: Advisor on Research and Statistics, German Bioethanol Industry Association, 2016-2017 Research Fellow, Université Côte d'Azur, Medicinal Chemistry (Total Synthesis), 2015-2016 Post-Doc, Université Côte d'Azur, Green Chemistry (Heterogeneous Bi- and Au-catalyzed reactions and Flow-Chemistry), 2010-2014 PhD, University of Potsdam, Organic Chemistry (Sequential oxidative and non-oxidative Pd-catalyzed reactions), 2005-2010

Chemistry, University of Potsdam

For publications please see [linkedin.com/in/nelizarov](https://www.linkedin.com/in/nelizarov)

3.2. **Johan v. Groenestijn**, Wageningen (The Netherlands) Production of acetic acid from waste activated sludge

J.W. van Groenestijn, T.M. Mubita and R.J.M. Bisselink; Wageningen Food and Biobased Research; correspondence: johan.vangroenestijn@wur.nl

The problem

Primary sludge (directly from sewage) and activated sludge (excess sludge and secondary sludge) are generated at wastewater treatment plants and are residual flows that are now not utilized effectively. Most of it is fermented into biogas (but with a very low yield), dried and sent to incinerators. This current sludge processing costs € 500 - 1000 per tonne of dry matter (DM) and, with an annual flow size of 350 ktonnes (DM) in the Netherlands, is a major cost item for Dutch water boards and industries. The policy of water boards and many companies with wastewater treatment is aimed, on the one hand, at reducing the costs of wastewater treatment and, on the other hand, at using sludge for higher value applications such as a raw material for the chemical industry. Next to sludge, verge grass was studied as a feedstock. Disposal of verge grass is also a cost item and road authorities in a similar way are looking for cost reduction and higher quality use of their residual flow.

Our solution

A laboratory scale process was developed in which low-grade sludge was converted in a cost-efficient manner into acetic acid (in concentrated solution). In a similar manner, the juice of pressed verge grass was as well converted into acetic acid. The poor digestibility of waste activated sludge is notorious, therefore, the sludge was first pretreated with alkali to open the cells and subsequently subjected to biological anaerobic acidification. In this bioconversion this same alkali served as a titrant to neutralize the organic acids produced. After a solid/liquid separation the liquid was concentrated using reverse osmosis. Further concentration of the organic carboxylates, while retaining neutral compounds, was carried out by electrodialysis. Subsequent electrodialysis with bipolar membranes enabled conversion of the organic carboxylates to the organic acids and alkali. The product is a mixture of highly concentrated organic acids with acetic acid being the main product, where the formed alkali can be reused in the process to neutralize formed organic acids during the biological acidification.

The innovation consists of the introduction of a process with a sequence of unit operations that has not previously been demonstrated in this combination: digestion of biomass with alkali, fermentative acidification with optimization of the acetic acid yield and electrodialysis. In addition to the acids, concentrated alkali is produced as well, which is used in the process for the digestion of biomass. The work is relevant as it utilizes large biomass streams which are now destroyed in incinerators or composted, to produce a biobased chemical which can be used by chemical industry. The work was carried out in collaboration with representatives of the whole value chain: water boards and potato starch company (owners of sludge), technology partner (with verge grass biorefinery and wastewater projects) and chemical companies (utilizers of acetic acid).

Preliminary results and conclusion

Activated sludge solubilization was reached by incubation at pH 12. Biological acidification was optimized in continuously fed reactors, some running 10 months. Conversion of volatile fatty acids (VFA) into biogas was avoided by using high pH in combination with short residence times in the reactor. Seventy percent of the sludge volatile suspended solids could be solubilized after pretreatment. More than 50% of the sludge organic matter could be converted into VFA of which 2/3 acetic acid. Verge grass juice contained 38 g organic matter/L and after acidification 18 g VFA/L was produced (11 g acetic acid/L). Down-stream processing by reverse osmosis and electrodialysis yielded solutions with concentrations near 60 g VFA/L. The solution is being tested for production of calcium magnesium acetate, an alternative environmental friendly deicer for removal of snow and ice from roads. In addition, it can be used to produce medium chain fatty acids. These two applications have been demonstrated by the two chemical companies in our research project. Preliminary design and costing studies indicated that the former sludge processing and disposal costs can be reduced by about 50% using this process.



Dr. Johan W. van Groenestijn is a bioprocess technologist with a MSc degree in environmental technology and a PhD degree in microbiology from Wageningen University (The Netherlands). Since January 2018 he is senior scientist and project manager at Wageningen Food & Biobased Research at which he develops production processes for the biobased economy.

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