



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

75th Starch Convention

April 09th – 10th 2024

program
supporting program
list of participants
summaries

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Evening Program

Monday, April 08th 2024

19⁰⁰ **Welcome Evening** at the **Convention Hall**, Detmold, Schuetzenberg 10

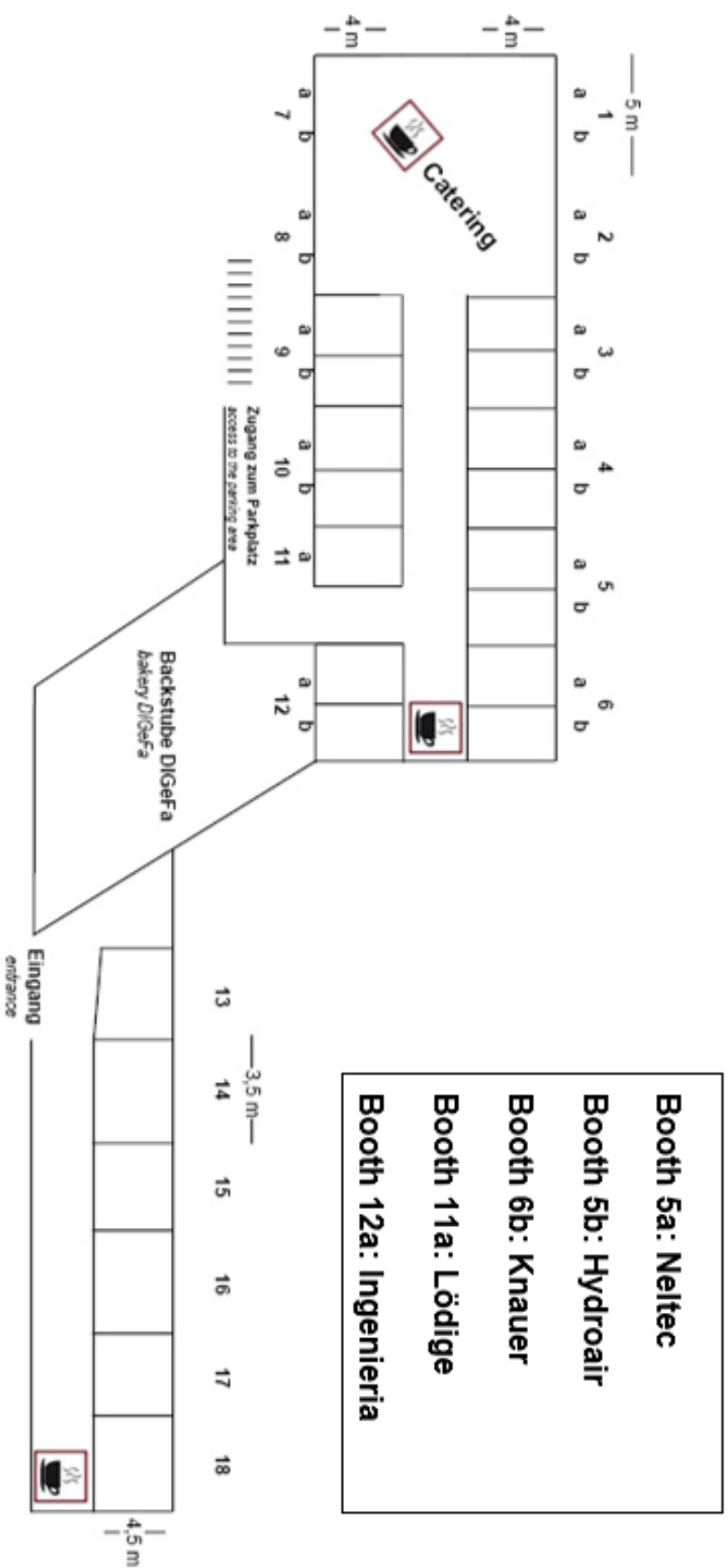
Tuesday, April 09th 2024

18³⁰ **“Stratosphäre Beer tasting”- Get-together** in the „Stratosphäre“, Detmold,
Privat-Brauerei Strate, Palaisstr. 1-13, 32756 Detmold

Exhibition Hall Association of Cereal Research

Stand allocation

75th Starch Convention from April 09th – 10th 2024



Exhibitors

Ingetecsa, Ingenieria y Tecnica del Segado, SA, Parets del valles, Barcelona, Spain

HydroAir Research Italia srl, Merlino, Italy

Neltec Denmark A/S, Bevtoft, Denmark

Knauer Wissenschaftliche Geräte GmbH, Berlin, Germany

Gebr. Lödige Maschinenbau GmbH, Paderborn, Germany

Exhibitor's Forum

1. **Bjarne Christian Nielsen**, Neltec Denmark A/S
“New instruments with new opportunities for process control and quality monitoring”
2. **Christian Schmidt**, Knauer Wissenschaftliche Geräte GmbH
“Introducing the AZURA® SEC - A new chromatography portfolio for the characterization and purification of biopolymers”

Lunch

Lunch will be served in the exhibition hall:

Tuesday, April 09th 2024

Malayan chick peas curry
cooked with eggplants, coconut milk, red lentils and mince

“Gumbo” is a type of stew from the southern United States
with turkey, sausage, scampi, vegetables and rice

The main dishes are flanked by mixed canapés (vegetables/fish/meat)

Dessert – Mousse au Chocolat and fruit salad

Beverages:

Mineral water

Coca-Cola

Orange juice

Apple juice

**Bon appétit
and interesting conversations!**

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Participants

Stand 04.04.2024, 16.40 Uhr

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Tuesday, April 09th 2024

08⁰⁰ – 08³⁰ Registration

08³⁰ **Opening Remarks** by the President of the Association of Cereal Research,
Götz Kröner, Ibbenbüren (Germany)

1. Structure

08⁴⁵ 1.1. **David Seung**, Norwich (England)
Understanding starch granule morphology in cereal endosperms: Insights from exploring natural variation

Despite the fundamental importance of starch to humankind as a major source of calories in our diets, we still do not fully understand how plants make starch. Starch is unique among carbohydrate polymers in that it forms insoluble, semi-crystalline structures called starch granules. While the synthesis of the starch polymers is relatively well understood, we are only beginning to understand how starch granule formation is initiated, and how granule size and shape are determined. Cereal grains such as wheat, rice, and maize contain starch granules that are remarkably different in morphology. We have discovered two major factors that underpin this variation. Firstly, we discovered specific proteins that control the initiation of starch granules, which are distinct from the proteins that synthesise the starch polymers. This includes the enzymes starch synthase 4 and plastidial glucan phosphorylase, and their numerous non-catalytic protein interaction partners. Differences in the expression patterns of granule initiation proteins and their protein interactions between species leads to distinct spatio-temporal patterns of granule initiation, which in turn leads to distinct granule morphologies. Secondly, interspecies variation in the morphology, structure and dynamics of the amyloplast, the organelles in which starch granules are produced, plays a major role in creating distinct granule shapes. Disruption of amyloplast structure by mutating components of plastid division can greatly alter the size and shape of starch granules. Our findings advance the understanding of starch granule formation in plants, and using this knowledge, we can now manipulate granule size and shape in wheat to resemble the morphologies found in other crops. This has major implications on improving the nutritional and functional qualities of starch.



David Seung is a Group Leader at the John Innes Centre, Norwich (UK). Research in his lab aims to understand starch biosynthesis at the molecular level, including the initiation process of starch granules and the control of granule size, shape and polymer composition. He received his B. Sc. from the University of Sydney, and M. Sc. and Ph.D. degrees from ETH Zurich. He joined the John Innes Centre after being awarded a BBSRC Future Leader Fellowship in 2017, and a Chris J. Leaver Fellowship in 2019.

Notizen:

[Zurück zum Anfang](#)

Starch is a polymer of glucose connected by α 1-4 glycosidic linkages. Each anhydro-glucose unit consists of six hydrocarbon groups in addition to three hydroxy groups. Consequently, a starch chain has a random coil conformation in an aqueous dispersion and amphiphilic properties. Because of the amphiphilic property, starch chains are not stable in the aqueous dispersion. When there are complexing agents, such as 1-butanol or fatty acids, which carry hydrophobic moiety, present in the aqueous dispersion, starch chains form single helical complexes with the complexing agents instantly. The single helical complex fold back and forth to form crystallites with ca. 10 nm in thickness (Jane and Robyt, 1984). In the absence of complexing agents, two starch chains would align with each other, having hydrocarbon groups facing each other, driven by hydrophobic interaction, to form double helix. This process is commonly known as retrogradation. Having hydrocarbon groups folded inside of the single or double helix and away from interacting with water molecules in the medium, the single helix and double helix are at a lower-energy state and are stable.

Starches of different botanical origins are known to have different structures, including different amylopectin branch chain-lengths, amylose contents, and crystalline structures, i.e., A-, B- or C-types polymorphism (Jane, et al., 1999). These different structures, in turn, lead to different thermal and pasting properties and enzyme digestibility. Starch that comprises more long-branch chains, e.g., B2, B3 and B4 chains, is known to display the B-type polymorph, whereas, that consists of more short-branch chains, i.e., A and B1 chains, displays the A-type. On the basis of different structures of their Naegeli dextrans, Jane, et al., (1997) propose that the A-type starch comprises branch linkages in the crystalline lamella in addition to the amorphous lamella. Whereas, the B-type starch comprises branch linkages mainly in the amorphous lamella. These structural features are confirmed by studies on biosynthesis of branching structures of starch using mutants of branching enzyme BEIIb (Nakamura, et al., 2020). All of those structures reported are in good agreements with the cluster model of amylopectin structure. Jane (2006) further proposes that the A-type starch comprising more short-branch chains (A and B1 chains), which extend through one cluster and possess free chain-ends. Those short-chain double helices have more freedom to rearrange into a thermodynamically more stable monoclinic packing of the A-type polymorph from kinetically favored hexagonal packing (the B-type) and result in a porous structure within the starch granule. The porous structure of the A-type starch is responsible for the starch being more susceptible to enzymatic hydrolysis and chemical penetration.

Amylose and amylopectin molecules are synthesized initiating at the hilum and are interspersed in starch granules, which have been proven by using cross-linking reactions (Jane, et al. 1992; Kasemsuwan and Jane, 1994). Results obtained from surface gelatinization of starch granules (Jane and Shen, 1993; Pan and Jane, 2000) and from starch developmental studies at different maturation stages of maize (Li, et al., 2007) demonstrate that amylose concentration increases toward the periphery of starch granules and amylopectin branch-chains are shorter at the periphery. Developments of elongated starch-granules of the high-amylose starch are results of amylose molecules of adjacent starch-nuclei in the amyloplast interact to form double helices and bind the nuclei together to develop into elongated granules (Jiang, et al., 2010).

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Pan, D. D. and J. Jane. Internal structure of normal maize starch granules revealed by chemical surface gelatinization. *Biomacromolecules* 1 (2000) 126-132.



Professor **Jay-lin Jane** is a Charles F. Curtiss Distinguished Professor, Emeritus, in the Department of Food Science and Human Nutrition at Iowa State University. She obtained her Ph.D. in Biochemistry and Biophysics at Iowa State University. Dr. Jane's research focuses on starch structures and properties and has published more than 225 peer-reviewed papers with an h-index 76.

Notizen:

[Zurück zum Anfang](#)

09⁴⁵ 1.3. **Birte Svensson**, Lynby (Denmark)
Structure and function of enzymes acting on starch granules

Yu Wang,¹ Silke Flindt Badino,¹ Yu Tian,² Yang Li,¹ Stefan Jarl Christensen,¹ Bernard Henrissat,^{1,3,4} Štefan Janeček,^{5,6} Georges Feller,⁷ Peter Westh,¹ Andreas Blennow,² Marie Sofie Møller¹ & [Birte Svensson](#)¹

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Biosynthesis, mobilization and application of starch granules involving specific enzymes present an exquisite crowd of fascinating molecular interactions and catalytic activities. We'll present a few founding and recent examples of enzyme structures and functions along with current results from heterogenous enzymatic catalysis on starch granules.

During barley seed germination or industrial malting α -amylase binds to starch granules and hydrolyses α -1,4 linkages. Crystal structures of α -amylase in complex with maltooligosaccharides and site-directed mutagenesis revealed two binding sites on the enzyme surface at a distance of the active site, which are essential for degradation of starch granules [1-3]. Next, barley limit dextrinase hydrolyses α -1,6 linkages as illustrated in the complex with an α -limit dextrin accommodated across the catalytic site [4]. The limit dextrinase activity moreover is regulated by a barley cereal type inhibitor of picomolar affinity [5,6]. Surprisingly, limit

dextrinase contains two starch binding domains (SBDs) but fails to bind to starch granules [7]. These non-catalytic SBDs are categorized into 16 Carbohydrate Binding Module (CBM) families in the Carbohydrate Active enZYme database based on sequence similarity [8]. SBDs exist in many starch degrading and biosynthetic multi-modular enzymes and proteins [9]. Here, green fluorescent protein (GFP) fusions of selected members of the large CBM48 family, comprising 64345 sequences (March 27, 2024; www.cazy.org/), were functionally characterized using pull-down, affinity electrophoresis (AE) and surface plasmon resonance (SPR) techniques. While CBM48 from branching and debranching enzymes [4,10] showed very poor or no binding, CBM48-GFPs of rice FLO6 (FLOURY ENDOSPERM6 [11]) and *Arabidopsis thaliana* PTST2 (PROTEIN TARGETING TO STARCH 2 [12]) bound with very different affinity (K_d : 0.08–9.3 mg/mL) to maize, potato, barley, wheat and rice starch granules, amylose and amylopectin (K_d : 0.006–0.02 mg/mL), and α -cyclodextrin (2.7–9.7 μ M). Two *A. thaliana* α -glucan phosphatases, SEX4 (containing CBM48) and LSF2 (having two SBSs), showed highest affinity for amylopectin ($K_{d,SEX4}$: 0.03, $K_{d,LSF2}$: 1.6 mg/mL), respectively, amylose ($K_{d,SEX4}$: 5.4, $K_{d,LSF2}$: 0.68 mg/mL) [13]. Being insoluble particles, starch granules are degraded by hydrolytic enzymes in heterogeneous catalysis [14,15]. We adopted an interfacial kinetics method, inspired by cellulases acting on cellulose [16], using the classical Michaelis-Menten equation with substrate in excess and the inverted approach with enzyme in excess combined with Langmuir adsorption isotherms. This, besides kinetic parameters (k_{cat} and K_m), informed the densities of attack sites ($^{kin}\square_{max}$) and binding sites ($^{ads}\square_{max}$) for α -amylase, glucoamylase and pullulanase hydrolyzing α -1,4 and α -1,6 linkages on waxy, normal, and high-amylose starch granules from maize and other plants [14–18]. Notably, the attack site density of resistant high-amylose starch granules was only 10 and 25%, respectively, of waxy and normal starch granules [19]. Fusion of SBDs of family CBM20 to a psychrophilic α -amylase from the Antarctic bacterium *Pseudoalteromonas haloplanktis* increased the density of attack sites ($^{kin}\square_{max}$) and activity on starch granules by up to 5- and 2-fold, respectively [15]. The N-terminal SBD of family CBM41 in pullulanase from the human gut bacterium *Lactobacillus acidophilus* similarly controlled activity and attack site density for starch granules [18]. Enzymatic modification of raw starches receives increasing attention as a sustainable and environmentally friendly methodology [20]. Debranching by *Bacillus licheniformis* pullulanase of starch granules after such green modification by *Rhodothermus obamensis* branching enzyme and *Thermoproteus uzoniensis* 4- α -glucanotransferase indicated important increase on the modified granular surface of short branches (A-chains) and reduction in long branches (B-chains) as well as significantly elevated attack density after branching enzyme modification [17]. This shaving procedure using pullulanase allows to monitor and quantify the extent of surface modification on starch granules [17]. We will also discuss the impact of the active site architecture of different transglucosylases; branching enzyme, cyclodextrin glucanotransferase, 4- α -glucanotransferase, 6- α -glucanotransferase and amylosucrase on their ability to modify starch granules.

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Birte Svensson is professor at Department of Biotechnology and Biomedicine, Technical University of Denmark. Her main research interests are structure/function relationships and applications of multidomain carbohydrate active enzymes acting on starch, alginate and other dietary polysaccharide fibers, proteins and hydrocolloids in food and dairy.

Notizen:

[Zurück zum Anfang](#)

10¹⁵ 1.4. **Ian Tetlow**, Ontario (Canada)
Manipulating Source Leaf Starch Metabolism to Increase Crop Yields

Storage starch is an important agricultural commodity harvested from major crops as a food source, a raw material for many industrial applications, and distinct from transitory starch formed in photosynthetic (source) tissues which is degraded at night. Starch synthesis and turnover occur inside plastids and are dependent on the coordination of over 30 (iso)enzymes [1]. Starch serves a critical biological role in the carbon budget of most land plants, mediating carbon fluxes during development and in response to environmental stress, and therefore underpins plant fitness. During the plant's life-cycle starch plays a dual role in carbon allocation, acting as both a source, releasing carbon reserves produced in leaves for growth and development, and as a sink, either as a dedicated starch store in its own right (e.g., in seeds and tubers), or as a temporary reserve of carbon contributing to sink strength, in organs such as flowers, fruits, and developing non-starchy seeds [2]. The presentation describes a long-term study involving manipulation of transitory leaf starch structure and turnover in the model oilseed plant *Arabidopsis thaliana* L. (*Arabidopsis*) and its close relative, the crop plant canola (*Brassica napus* L.). Replacement of endogenous starch branching enzymes (SBEs) with cereal endosperm-specific SBE isoforms from maize (*Zea mays* L.), led to an unexpected 400% increase in total seed weight in *Arabidopsis* [3] and provided proof of concept to increase oilseed yield and biomass in canola. The study in *Arabidopsis* highlights an indirect method of enhancing oilseed yield in plants through manipulation of leaf starch reserves, which in turn alters carbon availability to sink organs *via* as yet unknown signalling mechanisms.

Canola is one of the most important oilseed crops as global demand for edible oil and renewable industrial products are increasing with the growing worldwide population. Maximizing oil yield in crops such as canola is therefore of prime economic importance. Previous studies aimed at enhancing oil yield have largely been confined to exploiting mechanisms of lipid biosynthesis and turnover to achieve increased oil accumulation and/or altered quality in seeds [4]. We describe a novel method of increasing oilseed yield in canola based on previous work in *Arabidopsis* [3]. In *Arabidopsis*, approximately 30% of carbon assimilated through photosynthesis is stored as transitory starch in source tissues such as rosette leaves during daytime, and is degraded at night to provide a continuous supply of sugars for transport to other sink tissues *via* the phloem in order to meet plant developmental requirements [5]. Diurnal starch turnover is under concerted and intricate control, ensuring economical carbon partitioning throughout the life of the plant [2,5]. Two SBE isoforms are found in *Arabidopsis* belonging to the class 2 category, termed *AtSBE2.1*

and *AtSBE2.2* and have overlapping function. Loss of both SBEs (the *sbe2.1/sbe2.2* double mutant) leads to an inability to synthesize starch, impedes plant growth, and underlines the importance of transitory leaf starch for plant growth and development. Complementation of leaf starch synthesis in the *Arabidopsis sbe2.1/sbe2.2* double mutant *via* expression of a maize endosperm class 1 SBE gene (*ZmSBEI*) caused increased biomass, increased oilseed yield and no effect on oil quality [3]. Transgenic expression of the endosperm-specific *ZmSBEI* gene in *Arabidopsis* may have led to altered diurnal cycling of starch and changes in sugar signalling.

We have conducted a similar experiment in canola by transgenic expression of *ZmSBEI* in a background in which endogenous canola SBEs have been gene-edited. Canola is an allopolyploid ($2n = 38$, AACCC) derived from crosses of the progenitors *Brassica rapa* ($2n = 20$, AA) and *Brassica oleracea* ($2n = 18$, CC). We generated mutants for all endogenous SBEs in canola (*BnaSBE2.1* and *BnaSBE2.2*) by utilizing the clustered regularly inter-spaced short palindromic repeats–CRISPR associated protein 9 (CRISPR–Cas9)-mediated gene-editing system driven by a highly-expressed embryo-sac promoter termed YAO (*At4G05410*) previously used in *Arabidopsis* [6] in order to increase the incidence of heritable mutations [7]. Multiple single-guide RNA (sgRNA) expression cassettes were assembled into a binary vector and two rounds of transformation were employed to edit all six *BnaSBE* genes. All mutations were heterozygous monoallelic or biallelic, and no chimeric mutations were detected from a total of 216 editing events. Previously unannotated gene duplication events associated with two *BnaSBE* genes were characterized through analysis of DNA sequencing chromatograms, reflecting the complexity of genetic information in canola. Five Cas9-free homozygous mutant lines carrying two to six mutations of *BnaSBE* were obtained, allowing us to compare the effect of editing different *BnaSBE* isoforms [7]. We also found that in the sextuple *sbe* mutant, although indels were introduced at the genomic DNA level, an alternate transcript of one *BnaSBE2.1* gene bypassed the indel-induced frame shift and was translated to a modified full-length protein. These sextuple mutants were used as the basis for creating transgenic canola expressing *ZmSBEI*. Analysis of the sextuple mutants showed reduced SBE enzyme activity and starch branching frequency, higher starch-bound phosphate content, and altered pattern of amylopectin chain length distribution relative to wild-type (WT) plants. Rosette leaf starch granules in the sextuple mutant showed irregular morphology and a slower rate of degradation during the night compared to WT plants. At maturity (pod-filling stage) the sextuple mutant was distinguishable from WT plants by a thicker main stem. The thick stem phenotype of the sextuple mutant confers increased resistance to drought and high temperature compared to control (WT) lines.

Transgenic expression of the cereal endosperm-specific *ZmSBEI* gene in gene-edited canola was driven under a cauliflower mosaic virus (CaM) 35S promoter. Transgenic canola were generated through *Agrobacterium*-mediated transformation, constitutively expressing the *ZmSBEI* gene. Transgenic canola expressing *ZmSBEI* consistently showed increased biomass and increased total seed yield in greenhouse conditions. Specifically, transgenic plants showed increased (60%) numbers of stems leading to a 40% increase in siliques (pods) and a 35% increase in total seed yield per plant. Significantly, the thick stem trait observed in the sextuple mutant is retained in the transgenic plants expressing *ZmSBEI* which offers potential advantages, for example in terms of abiotic stress/lodging resistance. This biotechnological approach offers a one-step strategy of manipulating source leaf starch metabolism to achieve a double benefit of increased oilseed yield and abiotic stress tolerance and is the subject of a current patent [8]. We are currently conducting field trials with the transgenic canola line and attempting to elucidate the mechanism(s) underlying these physiological traits.

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plant biomass and triples oilseed production. *Plant Biotechnology Journal* 14: 976-985. doi: 10.1111/pbi.12453

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[8] Methods of Increasing Plant Biomass and Oilseed Production (US patent 11,028,403, issued June 2021).



Professor Ian Tetlow studied Plant Science at the University of Newcastle-upon-Tyne, U.K. in 1986 and did his Ph.D. at University College of North Wales (Bangor), U.K., under the supervision of Professor J.F. Farrar. The focus of his Ph.D. work was understanding the regulation carbon partitioning in plants which has been an underpinning theme of his subsequent research. In 1990 he began post-doctoral work at the University of Manchester, U.K. studying the regulation of starch biosynthesis and aspects of carbon metabolism in heterotrophic plastids (amyloplasts). Following post-doctoral studies Dr. Tetlow continued to work in the area of non-photosynthetic carbon metabolism and was awarded a Leverhulme Special Research Fellowship, followed by an Industrial Fellowship at the University of Manchester. In 2002 Dr. Tetlow moved to the University of Guelph (Ontario, Canada) and is currently a Professor in the Department of Molecular and Cellular Biology. Current research interests involve understanding the role of protein-protein interactions and protein phosphorylation in regulating starch metabolism in plants, and the regulation of yeast and mammalian glycogen metabolism. Applications of his research involve improving yield of crop plants and modifying the physicochemical characteristics, and therefore functionality, of starches for food and non-food applications.

Notizen:

[Zurück zum Anfang](#)

10⁴⁵ Communication Break

2. Modification

- 11¹⁵ 2.1. **Victoria Goetjes**, Kassel (Germany) **-online-**
Characterization of the degradation processes of PLA starch composites in hydrothermal environment

An increasing demand for plastics and the simultaneous requirement for more sustainability and CO₂ savings leads to a steadily growing need for bioplastics. Poly (lactic acid) (PLA) is one of the most important bioplastics. However, the synthesis of PLA is associated with CO₂ emissions, which, comparable to petro-based plastics, are around 2 kg CO₂ equivalents per kg. To counteract this, PLA can be filled with natural fillers. Native starch is especially suitable for this purpose, as it has a significantly lower CO₂ footprint than PLA, which first must be synthesized from this starch. The addition of starch can also reduce the price of PLA.

PLA has so far only been used in a small number of applications, with a focus on the packaging industry. The reason for this so far limited use in application is, as a previous study showed, the lack of information regarding the long-term behavior of PLA and filled PLA under the influence of environmental conditions such as temperature and humidity. In order to expand the application areas of PLA, it is essential to understand the material behavior and aging as well as degradation processes. In hydrothermal environments, PLA can be aged or completely degraded not only by hydrolytic degradation but also by thermo-oxidative processes. The processes differ not only in terms of the degradation products formed but also in terms of the cleaved bonds within the PLA macromolecules.

In order to address the lack of information on the long-term behavior of PLA on the one hand and to optimize the carbon footprint on the other hand and thus advance the use of PLA and starch-filled PLA, this study investigated the behavior of neat PLA and PLA filled with 50% potato starch under the influence of elevated temperatures and humidities. For this purpose, composites of PLA and starch were first prepared using a twin-screw extruder, and test specimens were manufactured from them by injection molding. The test specimens were then subjected to accelerated aging in climate chambers at varying temperatures and humidities for 48, 96, and 168 hours, following their use in technical components and outdoor applications.

The aged specimens were then first characterized mechanically. The results indicate, that with increasing humidity and temperature, both the composites and pure PLA show a significant reduction in tensile strength and elongation at break up to a complete failure in this regard, the starch-containing samples show slightly improved resistance to the aging processes at elevated temperatures but exhibit greater sensitivity to increased humidity. To identify the degradation of PLA due to the hydrothermal effects, GPC measurements were performed. For PLA, there is a clear correlation between decreasing mechanical properties and reduced molecular weights. In contrast, the starch-containing samples show lower degradation processes in the form of chain scission due to increased temperature and humidity. However, physical aging processes occur due to the increased moisture influence, which also leads to a reduction in the mechanical properties.

FTIR measurements of the aged samples show that degradation is due to both hydrolytic and thermo-oxidative processes. Accordingly, changes in the FTIR spectra occur only for the test specimens that show a significant change in molar mass. In order to further characterize the degradation processes and to differentiate the degradation mechanisms, additional NMR measurements were taken, which revealed changes in the chemical structure of the aged samples.

Overall, the study allows a comprehensive understanding of the behavior of PLA and PLA starch composites in hydrothermal environments, as well as the effects of aging on the structure and properties of the materials. This study adds value to research on bio-based composites, as there is an urgent need to expand the applications of PLA beyond the packaging sector to advance the use of established bioplastics.



Victoria Goetjes studied mechanical engineering in Kassel, Germany with a focus on materials and construction. Since 2021 she has been working as a research associate at the institute of Materials Engineering and coordinates the research network beBio2 which deals with the resistance of bioplastics and biocomposites.

Foto: Sonja Rode/Lichtfang.net

Notizen:

[Zurück zum Anfang](#)

11⁴⁵ 2.2. **Christina Gabriel-Liebs**, Potsdam (Germany) Future perspectives of starch esters

In times when alternatives to synthetic polymers are being sought, the raw material starch, as a worldwide available raw material with high purity and a favorable price, is increasingly becoming the focus of interest. At the same time, however, starch as a raw material also presents a challenge, as native starches don't often meet the requirements for specific applications. This is mainly due to their high molecular weight on the one hand and their hydrophilicity on the other. These disadvantages can be adapted through (chemical) modifications. When testing various starch derivatives for different fields of application, the starch ester class has repeatedly proved to be successful. Thereby, the application-specific requirements for their application in e.g. technical coatings, adhesives and cosmetic products were primarily met by adjusting the molar mass, the ester chain length and the degree of substitution.

For the synthesis of the degraded starch esters, first of all, different native starches were degraded by enzymatic or oxidative processes. Afterwards the degraded starches were esterified using carboxylic acid anhydrides in water or carboxylic acid vinyl esters in DMSO to reach low up to high degree of substitutions (DS 0.01-1.8).

For the application in technical coatings and adhesives the potential of the degraded starch esters were tested as film formers / binders. As adhesives and coatings are linked in many requirement aspects, the basic requirements for both were the following:

- a) High solids content ($\geq 30\%$)
- b) Processable rheological behavior (flow behavior, viscosity)
- c) Improved water resistance
- d) Adhesion to specific substrates, e. g. metal and wood substrates
- e) Good film formation (for coatings)

All these requirements were fulfilled by medium substituted starches esters (water soluble/dispersable) as well as by high substituted starch esters (organic soluble or hot melt adhesive), whereby the latter, with their high DS, have significantly better water resistance. These starch esters show also excellent barrier properties as film formers (coatings), even comparable to automotive clear lacquers.

For cosmetic products degraded starch esters (laurates) with low to medium DS (0.01-0.45) were primary tested as emulsifiers in O/W emulsions, but also as thickener. Both functions were fulfilled by a starch ester with a medium M_w (10^5 g/mol) and a low DS (0.06). This ester emulsifies O/W emulsions successfully with an oil part up to 60% while having commercially suitable viscosities and pH stabilities (6-9). Basic creme formulations were also successful. The test formulation formed a nice and pleasant film on the skin and was absorbed so that no residue remained on the skin.

In summary, degraded starch esters are very flexible and have the potential to be used in very different application fields to replace the corresponding synthetic polymers in the future.



Dr. Christina Gabriel-Liebs studied chemistry at the University of Ulm. She completed her master thesis and her PhD (2019, University of Potsdam) in polymer chemistry at the Fraunhofer Institute for Applied Polymer Research IAP in Potsdam, where she subsequently remained as a scientist. As a project leader and manager in public funded and industrial projects, she is engaged in the development and characterization of modified starches for various technical applications. Her focus is on coating and adhesive applications, but also on new green synthesis routes.

Notizen:

[Zurück zum Anfang](#)

12¹⁵ 2.3. **Johanna Thomann**, Groningen (Netherlands)

Magnesium-containing potato starch (derivatives) and their applications in food –
OR: Effects of mineral elements and annealing on the physicochemical properties of native potato starch

Native potato starch is an excellent carrier of minerals due to its inherent ion exchange capacity, owing to the presence of negatively charged phosphate esters. Mineral enrichment not only changes the nutritional value of starch but also influences pasting and swelling properties. Furthermore, hydrothermal treatments like annealing, heat-moisture treatment and dry heat treatment constitute a straightforward way to tune functional properties without producing problematic chemical waste. Here, novel combinations of mineral enrichment and annealing were studied in detail. Ion exchange was readily achieved by suspending starch in salt solution at room temperature over 3 h and enrichment was confirmed by inductively coupled plasma optical emission spectroscopy (ICP-OES). Annealing was performed at 50 °C for 24 h using demineralized water or aqueous solutions of NaCl, KCl, CaCl₂ or MgCl₂ · 6 H₂O.

Pasting, thermal and swelling properties were strongly influenced by annealing. Differential scanning calorimetry (DSC) results support a more ordered structure after annealing, however, only a minor increase in relative crystallinity was observed in XRD. Solid-state NMR spectra showed similar spectral features before and after annealing, with no detectable changes. The amount of total digestible starch decreased after annealing. In both ion exchange at room temperature and during annealing, monovalent cations promoted swelling and peak viscosity in rheological studies (RVA) and divalent cations suppressed peak viscosity, most likely through ionic crosslinking.

Annealing in the presence of mono- and di-valent cations allows fine-tuning of the pasting behavior of native potato starch thereby offering an alternative to chemically modified starches for respective food applications. Furthermore, these findings can be used for the development of healthy and environmentally benign starch-based food ingredients high in target essential minerals like magnesium, and probiotics through increased resistant starch content.

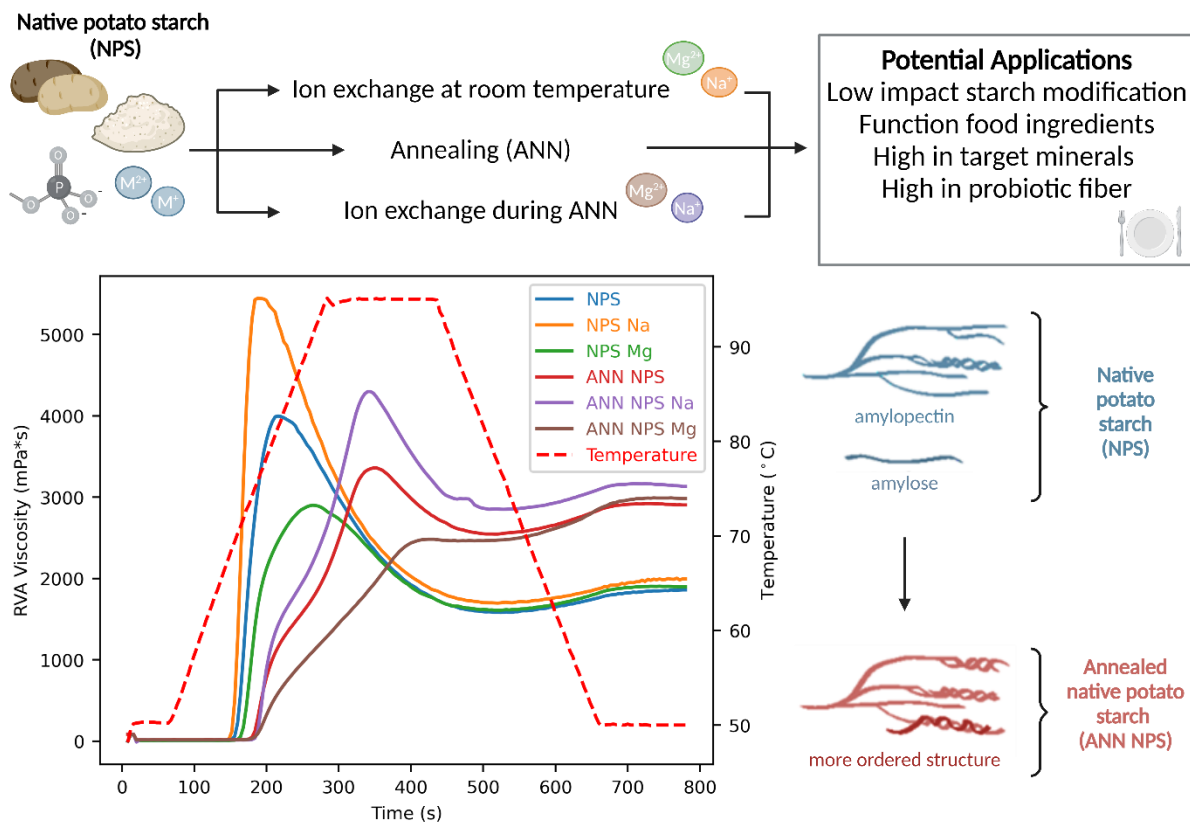


Image created with BioRender

Publications:

Broekman, J.O.P.; Dijkhuis, B.W.; Thomann, J.A.; Heeres, A.; Heeres, H.J.; Deuss, P.J. Dibasic Magnesium Hypochlorite as an Oxidant to Tune Pasting Properties of Potato Starch in One Step. *ChemEngineering*, **2023**, 7, 24. <https://doi.org/10.3390/chemengineering7020024>
 Thomann, J.A.; Heeres, A.; Heeres; Bekkering, E. The Northern Netherlands: Transformation of a gas-producing region into a forerunner in the biobased circular transition. *Journal of Business Chemistry*, **2023**, 20, 1, DOI: 10.17879/11059720490.



Johanna Thomann is a PhD candidate at the Research Center Biobased Economy in Groningen (NL), where she studies environmentally-benign alternatives for common chemically-modified starch products by combining regional feedstocks. During her studies in Germany and the Netherlands, she worked in the field of organic photochemistry in the groups of Prof. dr. Anna McConnell and Nobel laureate Prof. dr. Ben Feringa.

Notizen:

[Zurück zum Anfang](#)

12⁴⁵ Lunch Break

13⁴⁵ 2.4. **Michael Polhuis** and **Frederico Perondi**, Groningen (Netherlands)

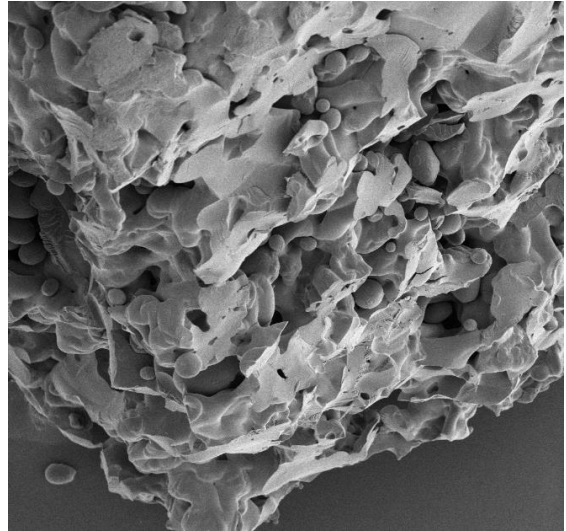
RVA profiling and microscopic appraisal of native potato starch treated in an scCO₂-capable reactor

The utility of treating granular native potato starch in a supercritical carbon dioxide-capable reactor at various temperatures and pressures, and different initial moisture contents, was investigated. The rationale behind this study is based on the observations (a) that previously reported results show contrasting effects of reactor pressure on treated starch properties, (b) complete gelatinisation of a 1:1 wheat:potato starch blend after 30 minutes has been observed, whereas potato starch alone did not gelatinise after 60 minutes under the same conditions, and (c) reported ranges of temperatures and pressures to date do not fully encompass the actual operational ranges of current scCO₂-enabled reactors.

For the investigation, native potato starch samples having an initial moisture content of either 15 or 40% were subjected to varying pressures and temperatures for a period of 4 hours. Pressures were dictated by the operational safety limits and capabilities of the peripheral equipment, e.g., pump, and were fixed at 60, 100, 200 300 and 550 bar. Temperatures encompassed a range commencing at 20°C and ending at 100°C, with special attention being paid to the range approaching and then exceeding potato starch gelatinisation temperature (approximately 69°C for native potato starch), viz., 60, 65, 70, and 75°C. These combinations of pressure and temperature enabled CO₂ in the gas, liquid and supercritical phases to be employed.

All treated starches were analysed by RVA profiling and optical microscopy to gauge the effect of treatment on starch granule pasting and morphology. A limited selection of modified starches were also analysed using X-ray diffractometry (XRD), further augmented by scanning electron microscopy (SEM).

The results show that native potato starch after treatment in an scCO₂-capable reactor afford starch pasting profiles that in some cases are highly reminiscent of those that have been reported previously for heat-moisture treated (HMT) and annealed (ANN) starches.



A MiniTab Response Surface statistical analysis of the pasting data reveals that potato starch initial moisture is the primary factor, although temperature plays a role, together with pressure, starch batch and CO₂ phase also having some contributions. At the highest temperatures and pressures used, and for starch with an initial moisture content of 40%, almost complete gelatinisation of starch is observed. This is exemplified by a SEM image of a starch sample having an initial moisture content of 40% that was treated at 100°C and 550 bar for 4 hours, affording a mostly amorphous product in which small, intact starch granules remain embedded.



Michael Polhuis, Ph.D. working for Avebe since October 2004, I at present occupy the role of Principal Scientist – Starch: Clean Label and Fibres. My primary focus is on the investigation of more sustainable starch processing and valorisation of associated side-streams, such as starch fibres, as well as the potential applicability of various physical processes for the preparation of Clean Label modified starches.



Federico Perondi hailing from Italy, I am currently conducting research towards my Ph.D at the University of Groningen in The Netherlands, and expect to graduate later this year. My focus is on supercritical CO₂ drying of potato starch and how this treatment affects the pasting and morphology of the treated starch granules.

Notizen:

[Zurück zum Anfang](#)

3. Application

14¹⁵ 3.1. **Jens Buller**, Potsdam (Germany) Enzyme-catalyzed selective oxidation of starch

Oxidised starches are widely used in food, paper, pharmaceuticals and beyond. Their good solubility, customised viscosity and stability due to molar mass reduction and the introduction of various functional groups such as carbonyl and carboxyl groups make them valuable materials for numerous applications. In an expansion of the concept the regioselective C6-OH oxidation of starch using TEMPO (2,2,6,6-Tetramethyl-1-piperidinyloxy radical) as Mediator and NaBr as Cocatalyst has also gained attention due to that it can obtain high carboxyl content while maintaining higher molecular weight of starch. However, the use of the unsustainable oxidizing agent NaOCl and the generation of poisonous chlorinated byproducts poses serious environmental and corrosion risks, making also sodium bromide's use as a co-catalyst highly undesirable.

Laccases are natural copper containing enzymes capable of oxidizing a variety of substrates. They represent a promising green alternative for the oxidation of alcohols and thus also of polysaccharides, as they only use O₂ as a oxidising agent and thus make additional chemical oxidising agents such as sodium hypochlorite unnecessary. To date however, little is known about the use of laccases with polysaccharides and only a few papers have been published on this subject, although most of them didn't investigated the structural changes in starch during oxidation.

In this study, we therefore analysed the oxidation of potato starch and potato starch dextrin with laccase from *Trametes versicolor* and mediator TEMPO in more detail. We varied parameters such as time, TEMPO co-catalyst concentration and laccase dosage in a suitable buffer solution. Both homogeneous and slurry conditions were used in the oxidation reaction. The products were analysed by FTIR, ¹³C NMR and titration methods to determine the proportions of CHO and COOH groups. High performance anion exchange chromatography (HPAE-PAD) was additionally utilized for the analysis of the selective C6 oxidation. With the conditions used, we achieved a maximum degree of oxidation of 0.2 by using laccase. Notably, under both air and oxygen atmospheres, similar results were observed. In contrast, blind tests without laccase or mediator showed little or no reaction.

Microscopic investigations revealed preserved starch granule morphology, albeit with minor damage.

Our findings also indicate significant molecular weight degradation attributed to β-elimination reactions due to the formation of aldehyde groups at C6, highlighting the efficacy of Laccase-Mediator Systems in starch oxidation.



Dr. Jens Buller is a chemist and received his PhD in polymer chemistry from the University of Potsdam in 2013. He is particularly interested in linking classical polymer and materials research with the world of starch. He is head of the Starch Modification / Molecular Properties department at the Fraunhofer Institute for Applied Polymer Research. His goal is to expand the possibilities of starch as a raw material and to find new applications or modifications of starch. His research therefore focuses on the modification and molecular and physical characterization of starch, especially for technical applications such as paper, adhesives and plastics. In his function he leads several industrial, but also publicly funded national and international research projects.

Notizen:

Zurück zum Anfang

14⁴⁵ 3.2. **Tatiana Budtova**, Sophia Antipolis (France)
Starch aerogels and aerogel-like xerogels for drug delivery

Fangxin Zou, Tatiana Budtova
Mines Paris, PSL University

Starch is one of the most abundant and biodegradable natural polysaccharides; it has been widely used in pharmaceuticals in tablets, as disintegrants or as porous carriers. Various chemical, physical and enzymatic methods are used to make porous starches resulting in materials with different morphology, porosity and pore dimensions /1/. Among physical methods (i.e. no chemical modification, no crosslinking) are extrusion of starch-water blend at high temperatures, microwave heating, freeze-drying or supercritical fluid extrusion. These porous starches are usually of low density and with large pores, resulting in materials with low specific surface area. In the past two decades, drying of starch gels with supercritical CO₂, resulting in aerogels of density 0.1 – 0.3 g/cm³ and specific surface area up to 350 m²/g, was reported /2/. The properties and morphology of starch aerogels can be tailored by starch concentration /3/, amylose/amylopectin ratio /4/ and gelatinization temperature /5/.

In this work, we demonstrate a pathway of making aerogel-like starches without any chemical modification and without using high- or low-pressure technology /6/. As an example of application, we used these materials for the release of a model drug, theophylline (Figure 1) /6/.

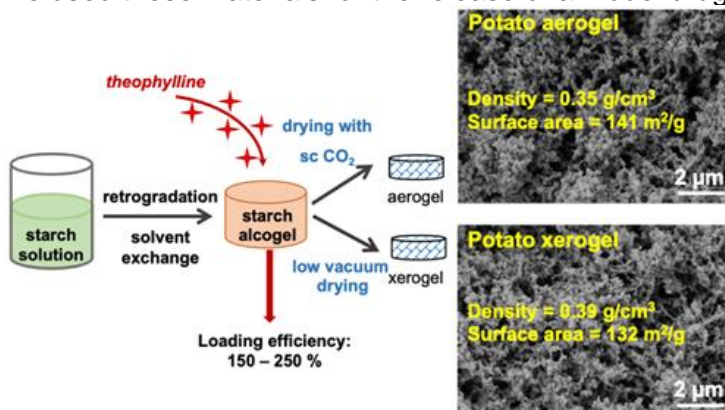


Figure 1. Illustration of the preparation of starch aerogel and xerogels and their loading with theophylline /6/

We made retrograded starches (named hydrogels) by dissolving different starches, followed by exchanging water to ethanol (resulting in *alcogels*) and performing drying under low-vacuum, simply using water aspirator (named *xerogels*). For comparison, we also used supercritical CO₂ to obtain *aerogels* and freeze-drying to obtain materials named *cryogels*. The influence of starch type (amylose/amylopectin ratio), concentration in solution, retrogradation time and drying method (supercritical CO₂ drying and low-vacuum drying) on dry porous starch density, specific surface area and morphology was investigated.

Two main achievements will be demonstrated: 1) several starch xerogels (i.e. materials obtained via low-vacuum drying) turned out to be aerogel-like, i.e. with low density and high specific surface area (above 100 m²/g), demonstrating the possibility to prepare aerogel-like starches avoiding high-pressure technology. 2) Starch alcogel adsorbs theophylline from ethanol loading bath resulting in loading efficiency as high as 250 – 300%. Theophylline was not bound to starch as the drug was completely released in aqueous simulated gastric and intestinal media, suggesting that drug adsorption from ethanol is due to the better affinity of theophylline to starch as compared to ethanol, confirmed by the values of solubility parameters. The materials were characterized by morphology, density, specific surface area, theophylline loading efficiency and release kinetics.

Acknowledgements

The authors are grateful to Roquette for sponsoring the work and providing starches, to Pierre Ilbizian (PERSEE, MINES Paris) for the supercritical CO₂ drying, and to Suzanne Jacomet (CEMEF, MINES Paris) for the help with SEM imaging

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Tatiana Budtova is expert in polymer chemical physics: polymer solutions, gels and aerogels with the focus on bio-mass based polymers. She holds “research director” position in the Center for Materials Forming of Mines Paris, France. She is one of the editors-in-chief of Carbohydrate Polymers journal and chair-elect of Cellulose and Renewable Materials Division of American Chemical Society. She published more than 170 articles, and in 2020 she was awarded a “Silver Medal” by CNRS (France) for the outstanding research on bio-aerogels.

Notizen:

[Zurück zum Anfang](#)

4. Process technology

15¹⁵ 4.1. **Harry Bitter**, Wageningen (Netherlands)

(Poly)saccharide conversion: from thermal-catalysis to electro-catalysis & from monomer to polymer

In this presentation, I will provide an overview of our efforts towards the development of (primarily) heterogeneous catalysts for the oxidation of glucose to gluconic and glucaric acids, as well as our initial attempts at the electrocatalytic oxidation of starch to anionic starch.

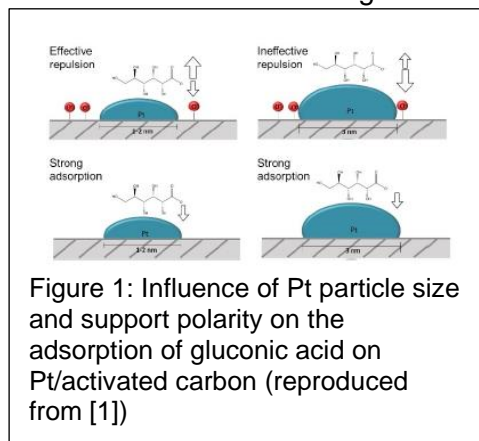
Platinum on carbon is a versatile catalyst for the conversion of polysaccharide feedstocks, whether through thermal catalysis or through potentially more sustainable energy inputs such as electricity.

In the thermal oxidation of glucose using Pt on carbon as a catalyst, it will be demonstrated that

both the support polarity and the Pt particle size determine the catalyst's performance. For catalysts with small Pt particles (2-3 nm), a polar support repels the oxidized product more effectively, while for larger Pt particles, the influence of the support is less significant (see Figure 1).

A support effect was also observed for the electrocatalytic oxidation of glucose over Pt on carbon nanofibers (CNF). The activity of Pt/CNF for the electrocatalytic oxidation of glucose increased with Pt particle size and with an increasing content of oxygen groups on the CNF surface. We concluded that support oxygen groups improve the adsorption of reactants in the vicinity of the Pt particle, thereby enhancing catalyst activity [2]. Additionally, the selectivity of the catalyst depends significantly on the

oxidation state of the Pt. PtOx increases the selectivity towards the electrocatalytic dehydrogenation/oxidation of the anomeric carbon at the expense of primary alcohol dehydrogenation. Despite the high selectivity of PtOx towards gluconate, it also promotes the successive dehydrogenation of secondary alcohol groups, leading to complex reaction mixtures. Pt0, on the other hand, directs the selectivity towards the dehydrogenation of primary alcohol groups at the expense of aldehyde oxidation reactions and anomeric carbon dehydrogenation/oxidation [3].



Initial attempts were also made to electrocatalytically oxidize starch to anionic starch. Figure 2 shows a comparison of linear sweep voltammetry of glucose and starch over Pt/CNF. For glucose, oxidative current was observed at 0.6 and 1.0V resulting from the oxidation of glucose to gluconolactone and the subsequent oxidation to carboxylic acids. For starch, only one oxidation peak was observed. Though this indicates that starch could be oxidized in this manner, the selectivity of that reaction requires further investigation.

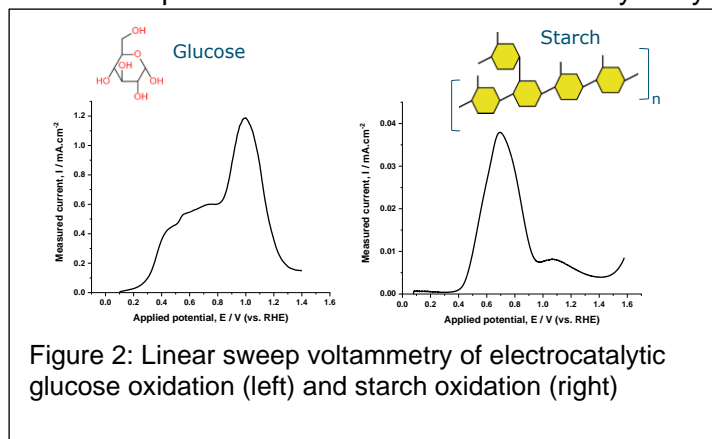


Figure 2: Linear sweep voltammetry of electrocatalytic glucose oxidation (left) and starch oxidation (right)

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Improved electrocatalytic activity of Pt on carbon nanofibers for glucose oxidation mediated by support oxygen groups in Pt perimeter. Appl.Catal. B. Environmental. 338 (2023) 123046.
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Steering the selectivity of electrocatalytic glucose oxidation by the Pt oxidation state. Angewandte chemie 62.33 (2023): e202306701.



Prof. Dr. Harry Bitter holds an MSc (1993) in organic chemistry and PhD (1997) in heterogeneous catalysis.

Since 2013 Harry is full professor at Wageningen University and Research in the Netherlands where he holds the chair Biobased Chemistry and Technology and focuses on catalytic biomass-based conversion (and extraction) processes on multiple length scales using both experimental and computational approaches.

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15⁴⁵ Communication Break

Starch biosynthesis is a complex machinery involving a multitude of enzymes including soluble starch synthases (SSS), starch branching enzymes (SBE) and debranching enzymes (DBE) and glucan water dikinase, (GWD) for amylopectin biosynthesis and mainly granule bound starch synthase (GBSS) for amylose biosynthesis. With the purpose of understanding starch bioengineering strategies in a wider sense, to generate high-amylose (HAS) starch crops mainly aiming at providing high resistant starch, by engineering the starch biosynthesis metabolic pathways. Hence, we recognise the content of low glycemic slowly digestible starch (SDS) and prebiotic non-digestible starch (RS) with the prospect of generating health-promoting starch. The application of new breeding technologies (NBT), especially CRISPR, in the breeding of HAS provides a novel route for this purpose. We identify three main engineering strategies and the generated products evaluated based on the building block backbone model for amylopectin (Zhong et al., 2022): 1. Suppression of SSSs or GWD for reduction of amylopectin side-chains or GWD for reduction of amylopectin biosynthesis through reduced phosphorylation; 2. Suppression of starch SBEs for production of amylose-like materials; and 3. Suppression of DBEs to restrain the transformation from over-branched pre-amylopectin to more ordered amylopectin. We conclude that amylose generated by the second strategy can be classified into two types: i) normal amylose synthesized mainly by regular expression of granule-bound starch synthases, and ii) modified linear amylopectin chains i.e. amylose-like material synthesized by starch synthases due to the suppression of starch branching enzymes, the latter material very similar to normal amylose.

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Zhong Y, Qu JZ, Liu X, Ding LY Liu, Bertoft E, Petersen BL, Hamaker BR, Hebelstrup KH, Blennow A (2022) Different genetic strategies to generate high amylose starch mutants by engineering the starch biosynthetic pathways. *Carbohydrate Polymers*, 287, 119327,



Andreas Blennow, received his MSc in 1987 and PhD in 1992, at Lund University, Sweden with research on starch metabolism and structure. Postdoctoral studies were carried out at Plant Science Centre, RSBS, ANU, Canberra, Australia on starch enzymology and crop bioengineering. He continued the research activities at the Royal Veterinary and Agricultural University, now University of Copenhagen, on research involving bioengineering of starch to generate functional and health-promoting starch. Blennow has since headed a research group active within plant-based foods and human nutrition, starch, and glycan bioengineering, polysaccharide-based biocomposites and fundamentals of starch structure and metabolism. AB has close cooperation with industry and is cofounder of the spinoff company plantCarb ApS for the design of amylose-only health-promoting starch crops. He has published around 200 scientific articles and book chapters and contributed to more than 200 conferences.

Notizen:

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Clean label is a new food trend. Consumers want a short list of common ingredients, which they are familiar with and they think “safe” for consumption. They do not want chemicals or chemically derived products in their foods. Clean label has been associated with “natural,” “organic,” “non-GMO,” “minimally processed,” “no chemicals,” “non-chemically modified,” “no additives,” etc. However, they are not the same things.

Although clean label is not regulated and is subjected to the interpretation of the consumers, there are regulations for food ingredients and food additives, which has become the basis of the clean label development. Food ingredients are normally produced by physical and enzymatic methods, and there is no limit for their usages in food products. On the other hand, there is a limit for the usage of food additives. They can be man-made products or natural products that are produced or modified by chemical methods. Thus, they are considered as not clean label, and can be easily recognized by their E-numbers in certain countries. However, there are no clear guidelines why some compounds are food ingredients or food additives. Some natural products, such as vitamins and natural extracts, have been regulated as food additives.

Because of the customer demands, food industry has been adopting this new food trend, creating more and more products with simple and less ingredients and replacing chemical modifications with physical and enzymatic modifications. This practice reduces the use of dangerous chemicals and decreases the chemical waste, which complies with the local laws and is more environmentally friendly. With less usage of dangerous chemicals, physical and enzymatic modifications are also safer for the production operators than chemical modifications.

Native starch is considered as clean label, but it has many limitations for its usage in food industry. One of such is the low resistance to heat, shear, and acid, causing the loss of its viscosity during process and storage, especially at low (acidic) pH. In addition, the molecules in starch paste or gel can recrystallize during storage, which is also known as retrogradation, causing undesirable phenomena, such as bread stalling and gel syneresis.

Chemical modifications have been traditionally employed to improve the functionalities of native starch. Stabilization, such as by acetylation and hydroxypropylation, can improve the stability of starch paste and gel against retrogradation during storage. Phosphate and adipate crosslinking can increase the resistance of starch to heat, shear, and acid. Dual modification of stabilization and crosslinking is a common practice to combine the desirable characteristics of both stabilization and crosslinking modifications. However, these chemically modified starches are food additives with E-numbers, and hence they have been considered as not clean label although they have been used in food industry since the 19th century without showing any adverse effects. Thermal inhibition is a physical modification recently developed to obtain a starch product with functionalities similar to chemically modified starches. The starch is subjected to thermal treatment after pH adjustment. The resulting thermally inhibited starch showed the resistance to heat, shear, and acid similar to crosslinked starch. However, the thermal treatment might impact the appearance of the starch, resulting in a darker color than chemically modified starch that needs to be improved. Furthermore, there is still a need to develop a clean label starch with the same stabilization effect (higher viscosity and more stable against retrogradation) as the chemically modified starch. This is a challenging task as there are not many factors that can be changed in the physical modification.

In conclusion, to satisfy the demand of consumers for clean label foods, the industry has developed new products by physical and enzymatic modifications in order to replace the chemically derived products. One of those new developments is thermally inhibited starch, which has similar functionalities as chemically crosslinked starch. However, there are still rooms for improvement, including the color, the viscosity, and the stability of physically modified starch. Thus, further developments are needed in this area.



Dr. Jovin Hasjim is a starch expert in Roquette R&D Functionalization team. His responsibilities include the development of new ranges of clean-label starches and starch derivatives to replace conventional chemically modified starches and maltodextrin. Dr. Hasjim acquired his Bachelor of Science in Chemistry from Iowa State University, USA, in 2003, and Ph.D. in Food Science and Technology from the same university in 2009. Before joining Roquette, he worked for the University of Queensland as a Senior Research Officer from 2009 to 2013. He has authored 15 patent applications, 41 peer-reviewed scientific articles, and one book chapter.

Notizen:

[Zurück zum Anfang](#)

17¹⁵ **Exhibitor's Forum** – short term presentations

Wednesday, April 10th 2024

4. Process technology

08³⁰ 4.4. **Ibrahim Yalgin**, Seyhan/Adana (Turkey)
Yield Improvement and Cost Saving Opportunities in Corn Wet Milling and Sweetener Refinery Processes

Corn Wet Milling process undergone by many improvements over the years and now is extremely efficient. On the other hand engineering and operational knowhow have significant effect on efficiency. The best in class designed plants with the help of Best Practises can achieve maximum possible yields. In today's economical situation it is utmost importance to maximize the yields to stay more competitive.

In steeping process, corn is softened, moisture increased, solubles leached out predominantly from the germ. It is very critical to have certain germ texture after steeping. Otherwise there will be excessive germ breakage in process step. It is possible to have +7.5% germ yield with the best in class design while some operations have around 7% germ yield only. 0.5% germ yield difference is not resulting only economical loss for high value of germ but also some other difficulties and losses arisen. High oil mill stream increases fouling of centrifuges, not clear separations and some some more extent of protein in overflow streams. Gluten becomes more sticky with higher oil content and it creates gluten filtration problems. When there is low germ yield issue there are some hidden problems in the plants due to higher oil and higher protein content in starch slurry. Native Starch quality, Syrup smell, color problems, problems in starch modification, chemical and water consumption in Ion Exchange resin regeneration systems, more waste water generation are just some examples.

In the same way it is possible to have +5% gluten yield depending on corn composition while there are quite many plants having 3.5-4.5% gluten yield. For a wet miller increasing germ and gluten yield is vital as their price is much more higher than starch and syrups. By increasing Starch Yield, Germ Yield, Gluten Yield and Total Yield a few million Euros/year more income is possible for wet millers. In this lecture speaker will explain wet mill process briefly, will mention

critical parameters in each process steps focusing especially on hidden losses which will lead maximum possible yields.

Any improvement in Wet Mill process results some other improvements. For instance, decreasing starch content of fiber will increase not only starch yield but also Gluten Yield.

Speaker will mention from Cost saving opportunities in both wet mill and sweeteners process focusing Energy Saving showing importance of PINCH technology as there is 10-20% thermal energy consumption reduction opportunity.

Halil Ibrahim YALGIN, Founder & General Manager of GPPE, graduated from Gazi University as Chemical Engineer. Started working at Tate & Lyle – ADM Joint Venture in Adana-Turkey as Production Engineer in 2005. Promoted Corn Wet Mill Area Manager position in 2008 and then to European Plants Corn Wet Mill Coordinator position in 2010. Between 2010 and 2019 worked as Plant Manager in Turkish companies with production management and Project Management roles. Installed, commissioned and started up Turkey's First Potato starch plant in 2013. Managed extension and modernisation projects for corn wet milling, starches and sweeteners. In 2021 Founded GPPE Global Process & Project Engineering company to serve global starch and sweeteners industry. GPPE offers Engineering and Consultancy and has many clients in different continents.

Notizen:

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09⁰⁰ 4.5. **Bruno Stengel**, Brussels (Belgium)

Process intensification for the Production of Monosaccharides

In the food industry the sugar alcohol Mannitol is used in confectionary application, e.g. moisture absorbent in chewing gum. Mannitol is produced by hydrogenation of the carbohydrate monomer Mannose. Mannose itself is obtained from glucose via an epimerization process using molybdenum oxides derivatives as catalyst at elevated temperatures. We reported earlier a laboratory process yielding up to 32% mannose using a lab module with micro channel structure. Based on these findings a pilot scale process was developed for a continuous throughput of 25 kg/h. Since commercialization is envisaged pressure drop and operability were major focal points in the design of the pilot set-up. By switching to the recently developed FGE's (fluid guiding elements) the same temperature profile was aimed with a significantly lower pressure drop. Experimental results in the pilot unit showed that this upscaling was successful, confirming that the process had been understood well enough in the lab to provide an optimized solution for the pilot scale.



Dr. Bruno Stengel graduated from University Louis Pasteur, Strasbourg, France with a PhD in Organic Synthesis focusing on the development of chiral catalyst systems used in the Diels-Alder reaction and di-Arsenium ylides for the selective production of trans olefins used in the synthesis of natural compounds. Then moved to the UK to pursue a post-doctoral position at UMIST in Manchester working on chiral intermediates based around sugars. He then moved to Belgium to work with ICI in the field of polyurethanes. He returned to the UK in 2000 with ICI then Johnson Matthey to pursue his interests in catalysis based around Titanium and Zirconium to be used amidation, esterification of natural compounds as well as in polyurethane applications. He returned to Belgium where he joined Cargill as

Principal Scientist working on the developments of novel fibres.

Notizen:

[Zurück zum Anfang](#)

09³⁰ Communication Break

5. Side streams

10⁰⁰ 5.1. **Robin Spelbrink**, Groningen (Netherlands)

Starch industry side-stream valorisation: the case of potato protein

The production of potato starch requires processing the protein-rich liquid side stream. While the potato contains only a modest concentration of protein, modern high-yield potato varieties produce a surprisingly high amount of protein per hectare. Potato protein is a high-quality protein that is well-suited to combinations with other proteins to obtain a balanced amino acid profile. The production method determines the properties the protein, with coagulates being best suited for nutritional usage and native proteins best suited for food texturing purposes. Food use of potato requires adequate control of glycoalkaloid contents as well as trypsin inhibitor activity. Potato protein is increasingly used by the food industry in a variety of applications.

Dr. Robin Spelbrink PhD, a seasoned biochemist, brings a wealth of expertise to the field of food science. With 17 years of dedicated work in the potato industry, he has been instrumental in developing sustainable food ingredients with improved flavour and nutrition.

Based near the city of Groningen, Robin resides with his supportive family—a wife and two children. Beyond the lab, Robin cultivates a passion for gardening, nurturing both plants and ideas.

Notizen:

[Zurück zum Anfang](#)

10³⁰ 5.2. **Florian Geyer**, Leipzig (Germany)

Valorization of by-products: upscaling the mono-fermentation of wheat pulp

Keywords:

Wheat pulp, biogas, high load fermentation

Introduction:

Wheat pulp is a carbohydrate and energy rich byproduct from industrial starch production from wheat. With its relatively low and fluctuating dry matter content of 10-15 % it is currently mostly used as feed in pig farming. The aim of the present joint project “Pülpegas” (pulp gas), led by Infra-Zeitz Servicegesellschaft mbH, in cooperation with DBFZ Deutsches Biomasseforschungszentrum gemeinnützige GmbH (DBFZ) and the Max Rubner-Institut (MRI), is the elaboration of a fully integrated reference plant for the complete material and energetic utilization of the wheat pulp stream on site. The task of the DBFZ was the determination of underlying process parameters for later system design, which was performed in semi-continuous experiments first in laboratory scale and subsequently in industrial scale at the DBFZ’s research biogas plant (FBGA).

Material and methods:

For the determination of the basic parameters for the mono-fermentation of wheat pulp, like biogas yield, biogas quality and minimal necessary urea supplementation, **laboratory scale** experiments were conducted. Two continuously stirred tank reactor (CSTR) with $V = 10 \text{ L}$ ($V_{\text{effective}} = 8 \text{ L}$) were fed once a day with the complete $\approx 70 \text{ g}_{\text{VS}} \text{ d}^{-1}$, and supplemented with urea solution (50 wt%) and trace element solution. The targeted organic loading rate (OLR) of $\text{OLR} = 7 \text{ g}_{\text{VS}} \text{ L}^{-1} \text{ d}^{-1}$ was reached after an initial starting phase of 60 days and held for another 60

days, equaling 3 hydraulic retention times (HRT) of the system. Unavoidable fluctuations in the contents of dry matter (DM) and volatile organic substances (VS) in different pulp batches from different deliveries were offset by recalculating and altering the daily feed. The evaluation of the measured data is performed for the period of the last 19 days.

For the purpose of the upscaling of the process the experiments were subsequently carried on to the **research biogas plant** (FBGA). After determination of the underlying parameters of the process the transferability to industrial scale was examined to investigate any adverse effects and identify possible problems. Therefore, the experiments were carried out in a CSTR with $V = 80 \text{ m}^3$ ($V_{\text{effective}} = 60,5 \text{ m}^3$). The necessary amounts of wheat pulp were delivered by and stored in a continuously stirred tank trailer ($V = 60 \text{ m}^3$). Following the receipt an incoming goods analysis was performed for determination of dry matter content and subsequently diluted with water to the target concentration of 13 – 15 %.

After an initial start-up phase of 50 days targeted OLR of $7 \text{ g}_{\text{VS}} \text{ L}^{-1} \text{ d}^{-1}$ was reached. Whilst the trial phase of 74 days (equaling 3,9 HRT), daily feed of $\approx 423 \text{ kg}_{\text{VS}} \text{ d}^{-1}$ was distributed to hourly substrate feeds. The in the preceding laboratory experiments determined optimal amounts of solutions of urea and trace elements were supplemented at daily basis. For binding of emerging H_2S in the biogas, FeCl_3 solution was dispensed on demand when hydrogensulfide (H_2S) contents exceeded the threshold of 500 ppm. The evaluation of the obtained data was performed for 21 days within the last retention time.

Results:

In the course of the **laboratory scale** experiments a biogas yield of $667 \text{ mL g}_{\text{VS}}^{-1}$ was obtained, with a methane content of 55 %, equaling $366 \text{ mL g}_{\text{VS}}^{-1} \text{ CH}_4$. The H_2S concentration was on average at $\approx 200 \text{ ppm}$. Despite the low pH value of $\approx 3,5$ upon receipt the pH value in both reactors equilibrated at $7,54 \pm 0,09$; whereas the pH value lowered whilst and directly after feeding to below 7 because of the low pH value of the substrate, but stabilized in the course of the day. The urea supplementation was adjusted to an average $\text{NH}_4\text{-N}$ threshold of $\approx 1,4 \text{ g L}^{-1}$. Because of observed strong deposition of suspended material it was necessary to stir the reactor at an unusual high stirrer speed of 120 rpm.

The experiments at the **research biogas plant** yielded a 27 % higher specific biogas yield of $850 \text{ mL g}_{\text{VS}}^{-1}$, with a methane fraction of 53 %, compared to the laboratory scale experiments. This equals a CH_4 yield of $460 \text{ mL g}_{\text{VS}}^{-1}$. The H_2S concentration increased in short terms above 900 ppm, whereupon FeCl_3 solution was dispensed to lower the concentration, whereupon the concentration could be lowered to $\approx 380 \text{ ppm}$ over the observation period. Through daily supplementation of urea solution, the $\text{NH}_4\text{-N}$ concentration could be adjusted to $\approx 1,9 \text{ g L}^{-1}$. Because of the distribution of the daily feed to hourly intervals greater fluctuations of the pH value, as seen in the laboratory scale experiments, could be prevented. Over the course of the experiment, the pH value was determined to be 7,66 on average.

As a reason of the, in comparison to the laboratory scale experiments, higher specific biogas and methane yields, as well as the higher H_2S concentration in the raw gas, the sedimentation of insoluble components of the wheat pulp and hence the enrichment of VS at the bottom of the fermenter is suspected. This assumption is supported by the observation of sedimentation of the wheat pulp in the laboratory scale experiments. Furthermore, whilst emptying and cleaning of the reactor after the experimental period, the deposition of a surprisingly high amount of crystals on all surfaces all over the reactor was observed. These crystals were in the aftermath identified as Struvite or magnesium ammonium phosphate (MAP).

Summary:

Despite the same OLR of $7 \text{ g}_{\text{VS}} \text{ L}^{-1} \text{ d}^{-1}$, the mono fermentation of wheat pulp in technical scale ($V_{\text{effective}} = 60,5 \text{ m}^3$) yielded a 27 % higher specific biogas- and methane yield in comparison to the laboratory scale experiments. Furthermore, a much higher threshold of H_2S , partly over 900 ppm, was observed in technical scale. To reduce the concentration to below 350 ppm the use of FeCl_3 solution was necessary. The reason for that observations is assumed to be the sedimentation of insoluble components or precipitated proteins of the wheat pulp in that larger scale. Additionally, the deposition of a technically relevant amount of Struvite on all surfaces of the reactor was observed.



Florian Geyer studied chemistry and food chemistry at the Martin-Luther University Halle-Wittenberg and graduated with his diploma thesis to Maillard-related alteration of wheat protein while baking. Since 2017 he worked in an environmental laboratory in Halle (Saale), most recently since 2020 as deputy laboratory manager, responsible for the areas of wastewater and waste. In 2023 he changed to the DBFZ Deutsches Biomasseforschungszentrum gGmbH in Leipzig and is since working as scientific coordinator of the research biogas plant. Beneath coordinating and managing scientific projects he contributes as researcher in projects regarding renewable energies and circular bioeconomy.

Notizen:

[Zurück zum Anfang](#)

11⁰⁰ 5.3. **Dirk Block**, Neuss (Germany)

Integrated use of by products from industrial starch manufacturing in biorefineries -
A route to decarbonized wheat based starch production?

Today in Europe more than 20MioT of agricultural raw materials are being use for an annual production of about 10 Miot of starch with wheat as a natural source representing the largest percentage followed by potatoes and corn. Its end use is divided about 1:1 between food and nonfood applications.

Depending on their natural source the respective processes to separate the individual components of the agricultural raw materials to isolate starch are leading to a range of different by-products that today utilized for example as substrates for the production of bio-ethanol or as nutrition in the intensive animal farming area.

They are themselves combinations of different liquid streams of variable composition with a wide range of active ingredients and content that are resulting from a series of purification steps during the isolation of starch. Their actual composition and concentration is predominately defined by the intended use and the corresponding requested end product specifications.

In the last years several studies have been conducted by companies to look at the economics of alternate use to the abovementioned options but acceptable returns on capital for their usage as source for biogas and biorefining could not be identified.

However, these studies might not have considered the use of individual streams in integrated bio refining processes. Depending on their physical properties and their actual composition each of these liquids could act as a source for biogas or biofuels and/or biotechnical production of basic chemicals.

Individually their application as substrates in integrated bio refining processes could provide significant additional economical potential that would result from reengineering of starch production processes on top of revenue from products of bio refining.

Additional regulatory changes initiated by existing RED or national legal requirements - including phosphorous recuperation in residuals - may increase the incentive to revisit the situation and to reconsider onsite integrated biorefining options.

This presentation will cover recent research activities looking at the use of wheat pulp from a starch producing factory. It will describe identified constraints in standard biogas units as well as potentials and will provide indications about options for process reengineering and opportunities related to the forward integration of the above described individual component streams.

Based on a schematic description for processing individual streams it will highlight the areas for additional process development in integrated bio refining and reflect first discussions with engineering companies. It will also describe the obvious gap concerning the availability of upscaling facilities that are essential to reach the technical readiness levels for commercial implementation of biorefining processes.

Thus, it has to be said that the ultimate approach for onsite bio-refining will depend on the individual existing customer and product portfolios at the respective sites.



Dr. Dirk Block, born in 1959, PhD in Chemistry University Hamburg and Cologne, MSc and MA in Psychology, 40 years work experience of which 30 years in the petrochemical industry and 10 years consulting, 30 years experience in designing and implementing restructuring projects with main focus on manufacturing and supply chain

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Marcus Schmidt, Elisabeth Scieurba

Max Rubner-Institut (MRI), Federal Research Institute for Nutrition and Food, Department of Safety and Quality of Cereals

Wheat pulp is a side stream that occurs in large quantities during the production of wheat starch. In 2020, 582,168 t of wheat starch were produced in Germany, which was associated with a quantity of at least 1,1 Mt of wheat pulp. To date, the pulp is mainly used as animal feed but it can be assumed that the dry matter of the pulp contains various wheat constituents which would be valuable for human nutrition. In particular, fibre and arabinoxylans should be considered to produce fibre-enriched foods, such as baked goods, and thus contribute to closing the "fibre gap" in the diet of developed countries. Wheat arabinoxylans are particularly noteworthy for their health-beneficial properties and can be advertised with a special "health claim". However, due to the low research interest in wheat pulp so far, little information is available on the composition of the pulp and the feasibility of its upcycling for human nutrition. Thus, the aim of this project is to investigate and assess the potential of wheat pulp for human nutrition. In addition to the fibre and arabinoxylan content, molecular properties, such as the molar mass of the arabinoxylans, also play an important role.

For this purpose, pulps were provided as sample material by two starch producers at regular intervals. Fresh pulp was provided by producer A, while the pulp from producer B was thermally treated to reduce the water content. The pulps were first subjected to a compositional analysis. Due to the low solubility of dietary fibres and the potential use of water-soluble components as a substrate for biogas production, the sediment was separated by centrifugation. These samples were analysed for dietary fibre, starch, protein and soluble mono- and disaccharides, as well as polyols. Due to the particular nutritional value of arabinoxylans, these were further characterised as water-soluble and water-insoluble, as well as for their arabinose/xylose ratio and the molar mass.

The dry matter contents of the analysed pulps from manufacturers A and B averaged 9% and 18%, respectively. The composition of the dry matter was comparable for both manufacturers with approximately 70% starch, 16% protein, 10% dietary fibre and 3% soluble mono- and disaccharides, as well as polyols. After centrifugation, the mass balance showed that 66% of the originally used dry matter was contained in the sediment, while the remaining 34% were present as dissolved substances in the supernatant. Analysis of the substances contained in the supernatant showed high levels of glucose and fructose, as well as water-soluble fibre and proteins, making it an interesting substrate for biogas production.

The composition of the sediments showed for starch, protein, soluble and insoluble fibre average contents of 70%, 10%, 2.5% and 15% for producer A, and 78%, 9%, 2.5% and 6% for producer B, respectively. The total fibre contents in the sediment from producer A (15 - 20%) were significantly higher than from producer B (7.5 - 9.0%), which is a value-adding aspect for use in human nutrition. The mass balance also showed that 97% and 23% of the originally present insoluble and soluble dietary fibres were found in the sediment. The characterisation of the arabinoxylans revealed significantly lower molar masses in the samples from producer B, but no differences with regard to the arabinose/xylose ratio. Accordingly, the samples from producer A have higher quality arabinoxylan in terms of nutritional value.

Thus, sediment from producer A was selected for baking trials. Model breads were prepared by replacing 0 (reference), 20, 30 and 40% of the wheat flour with wheat pulp sediment, using the appropriate amount of water (determined in accordance with ICC standard No.115/1) for each flour/sediment mixture. The resulting breads were evaluated based on their appearance, texture and crumb structure, as well as the dietary fibre and arabinoxylan content. The first results show a substantial quality deterioration with increasing levels of sediment, indicating the need for further optimisation.

In summary, the work has shown that wheat pulp is a side stream with insufficient utilisation of valuable ingredients, especially as it is produced in large quantities. By separating the liquid supernatant, a substrate with valuable ingredients for human nutrition can be obtained. In addition to starch and protein, the high fibre content is particularly noteworthy and can make a sustainable contribution to close the "fibre gap". However, before the utilisation of wheat pulp for human

nutrition can be implemented, further information needs to be generated regarding the factors influencing the composition of the pulp. In addition, extraction of the valuable ingredients from the sediment should also be investigated further to allow the production of sustainable, value-added, high quality products.



Dr. Marcus Schmidt, studierte an der Technischen Universität Dresden mit dem Abschluss zum Diplom-Lebensmittelchemiker. Die Forschung zur Biopräservierung von Getreide und Getreideprodukten mit Promotion in Food Science and Technology führte er am University College Cork (UCC), Irland unter Prof. Elke Arendt durch. Seit 2020 arbeitet er am Max Rubner-Institut als Leiter der Arbeitsgruppe Kohlenhydratanalytik. Seine aktuellen Forschungsschwerpunkte beinhalten Arbeiten zur Herstellung von FODMAP-armen (fermentierbare Oligo-, Di- und Monosaccharide, sowie Polyole) Backwaren, den gesundheitsfördernden Eigenschaften von Ballaststoffen der Ernährungssicherung durch Reststoffverwertung und die Kohlenhydratzusammensetzung von Kartoffeln.

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12⁰⁰ 5.5. **Nils Horstmann**, Oelde (Germany)
"New" starches as a by-product from plant protein recovery of legumes

Centrifugal separation technology plays a decisive role in the wet process for the extraction of plant proteins as well as starch and significantly influences the economic profitability as well as the quality and quantity of the end products. The presentation will go into the basics of centrifugal separation technology and the underlying physical parameters. In addition, two different types of centrifuges - separators and decanters - are explained, with the focus on the design features of different decanter types.

An overview of the processing of pulses into protein isolate powders is given, with a focus on wet separation. A key differentiation is made between starch decanters such as the GEA Starchmaster and protein decanters such as the GEA Proteinmaster. These decanters have customized designs that are optimally adapted to specific products and process steps.

In addition, the Varipond P system will be presented, which enables dynamic adjustment of the pond depth during operation. Relevant in protein-rich applications, this system increases yield, throughput and cleanability. The lecture will also present specific applications of both decanter types in the protein isolate extraction process, highlighting the process- and raw material-related influencing factors. Furthermore, an overview on the history of plant-based protein extraction processes and an introduction to the pulse market is presented.

Finally, the outstanding scalability of decanter and separator technology and the most important steps in process development will be the focus of attention.



Nils Horstmann, *Passionate bioprocess engineer specialized in downstream processing of plant-based and fermentation-based products. With a bachelor's from HSOS, a master's from DTU and work experience from industry leaders GEA, Novozymes & Danish Technological Institute, I bring a blend of academic rigor and industry experience. Currently developing & optimizing mechanical separation processes as a Process Specialist at GEA Westfalia.*

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12³⁰ **Closing remarks** by the Chairman of the Starch Experts Group, Christof Küsters

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